

## Self-assembly of functionalized lipophilic guanosines into cation-free stacked G-quartets

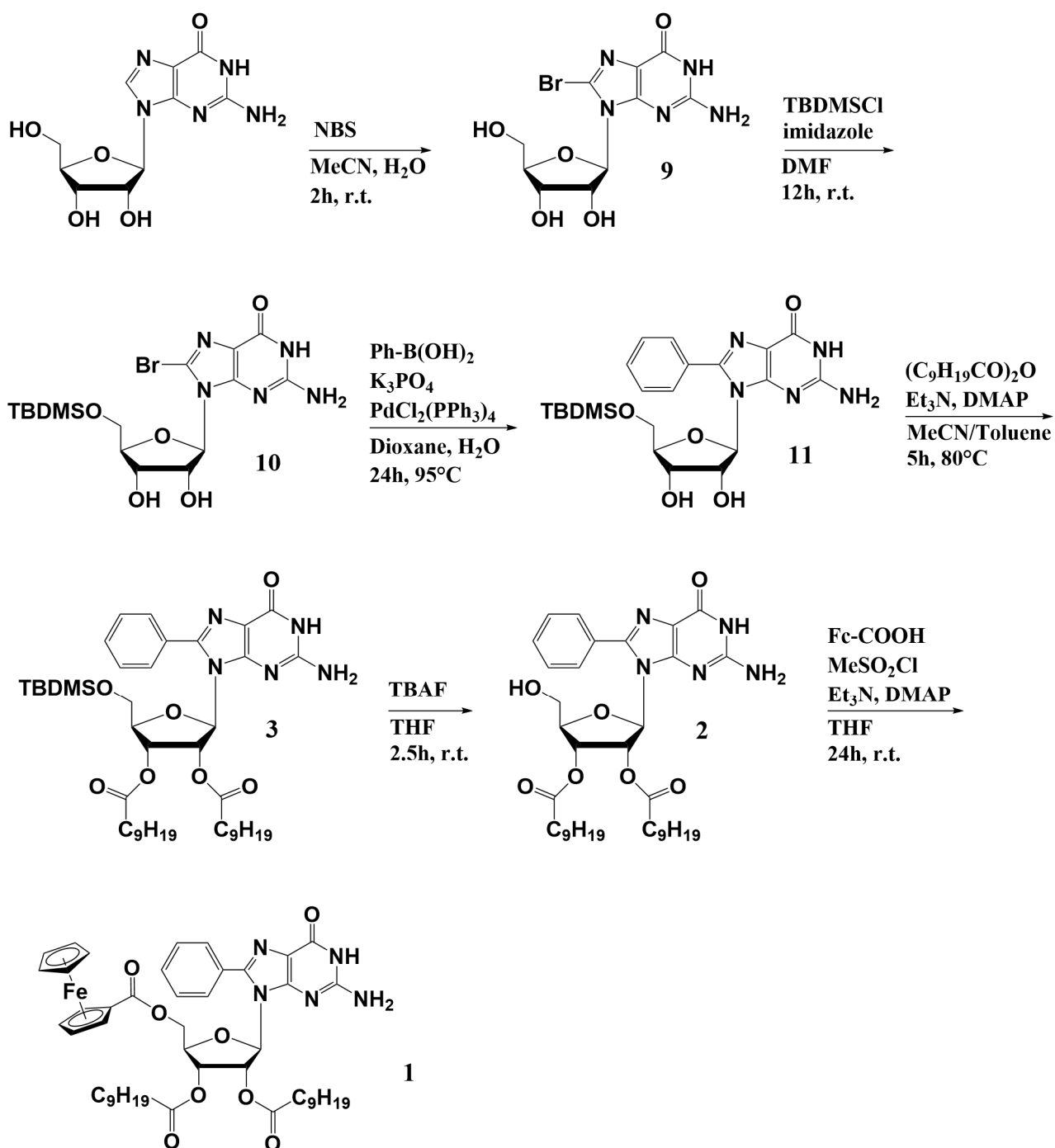
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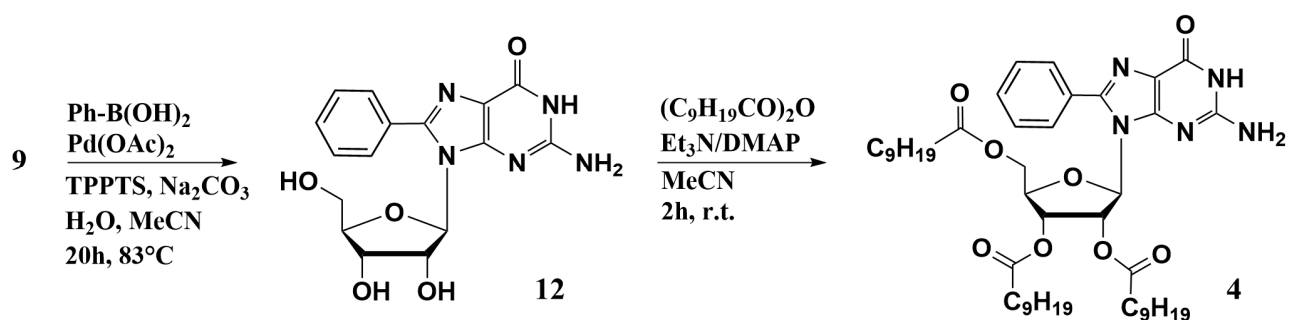
Synthetic schemes 1-4	S2
Selected CD spectra (Figure S1)	S4
VT <sup>1</sup> H-NMR of compounds <b>1-8</b> (Figure S2-S9)	S5
Table S1	S13
Selected 1D-NOESY spectra of compounds <b>1-8</b> (Figure S10-S16)	S14
Figure S17	S21
Figure S18	S22
Spectra of compounds <b>1-14</b>	S23

## Synthetic schemes

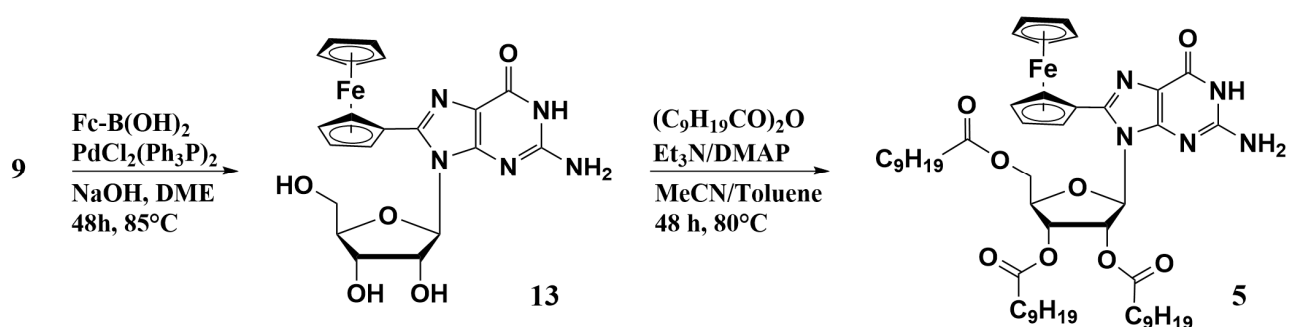


**Scheme S1.** Synthesis of guanosines **1-3** from commercial guanosine.

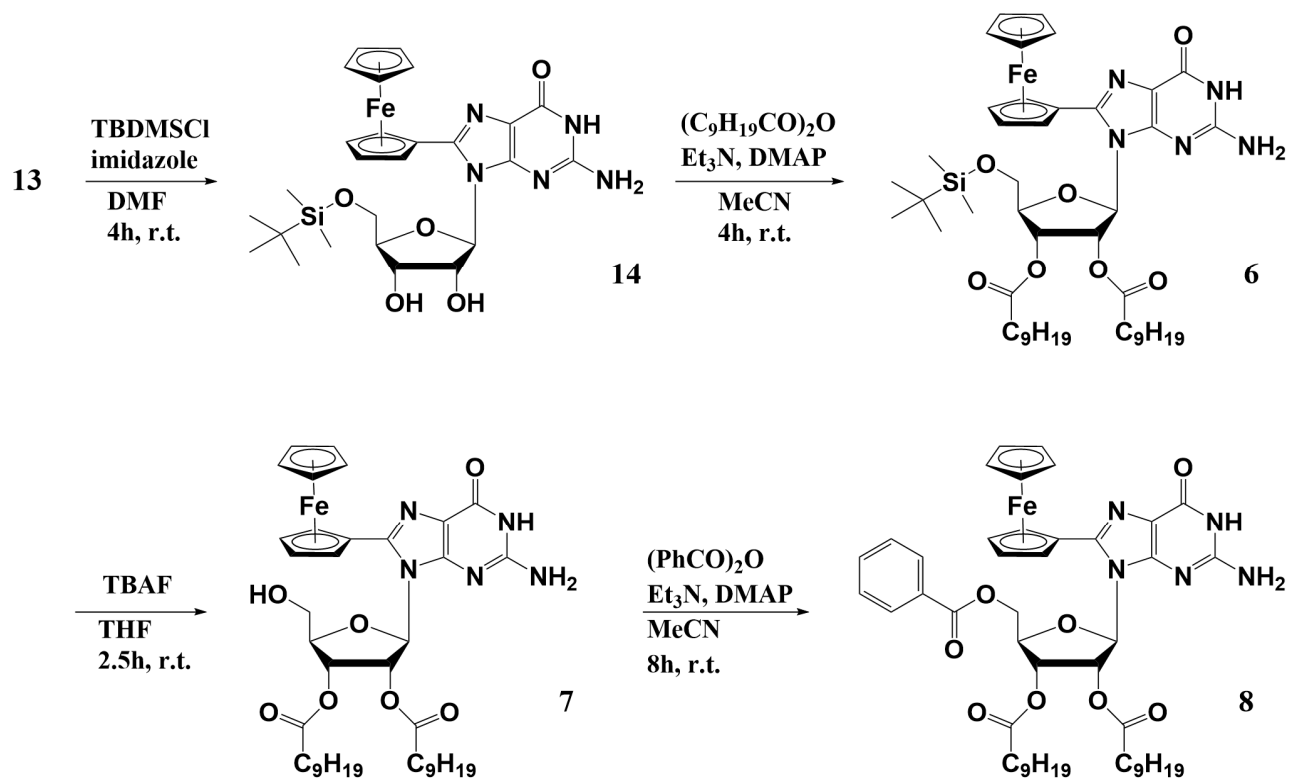




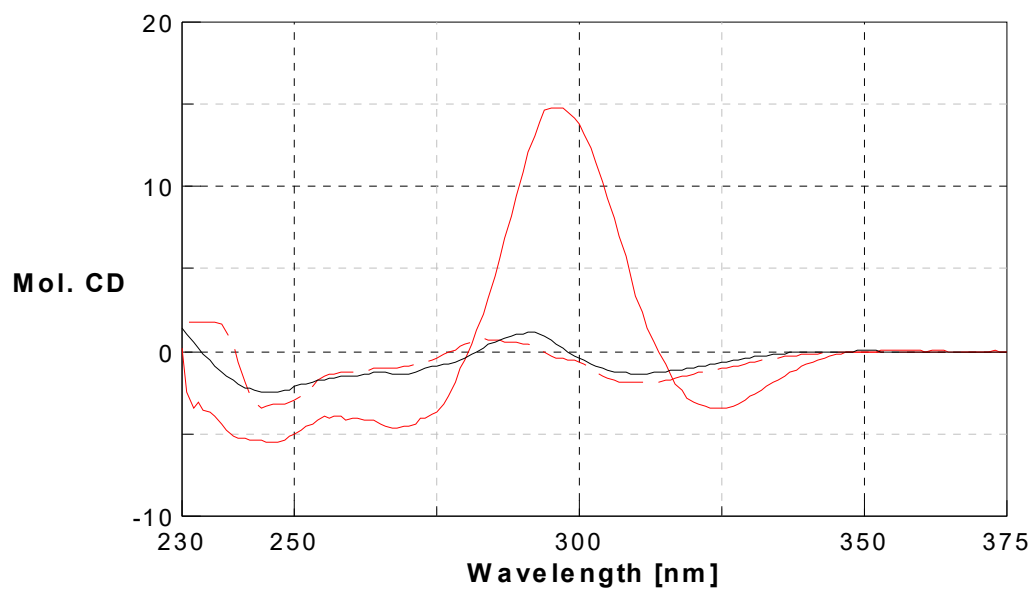
**Scheme S2.** Synthesis of guanosine **4** (8Ph5C10).



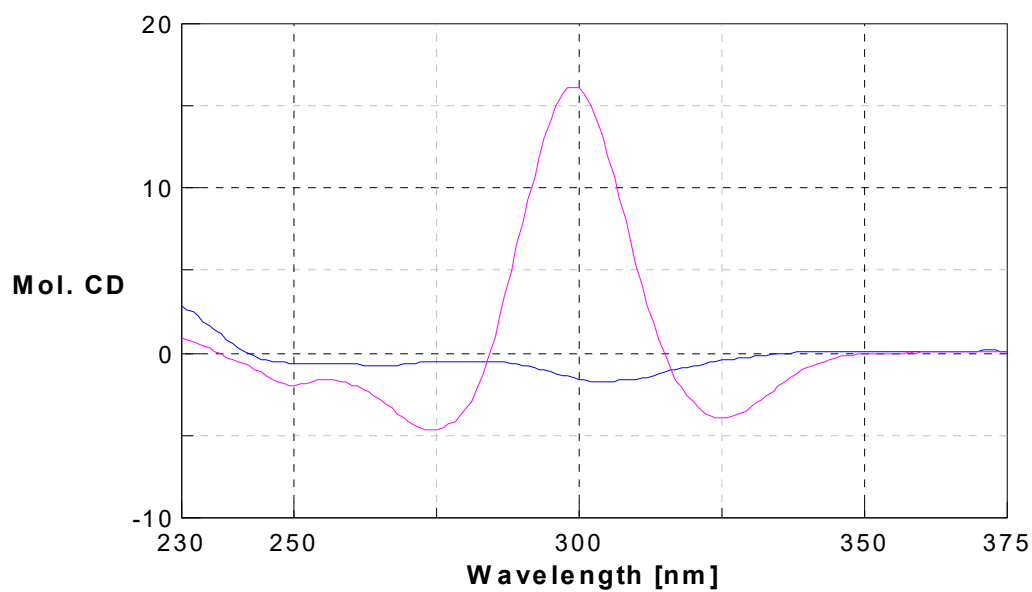
**Scheme S3.** Synthesis of guanosine **5** (8Fc5C10).



**Scheme S4.** Synthesis of guanosines **6-8**

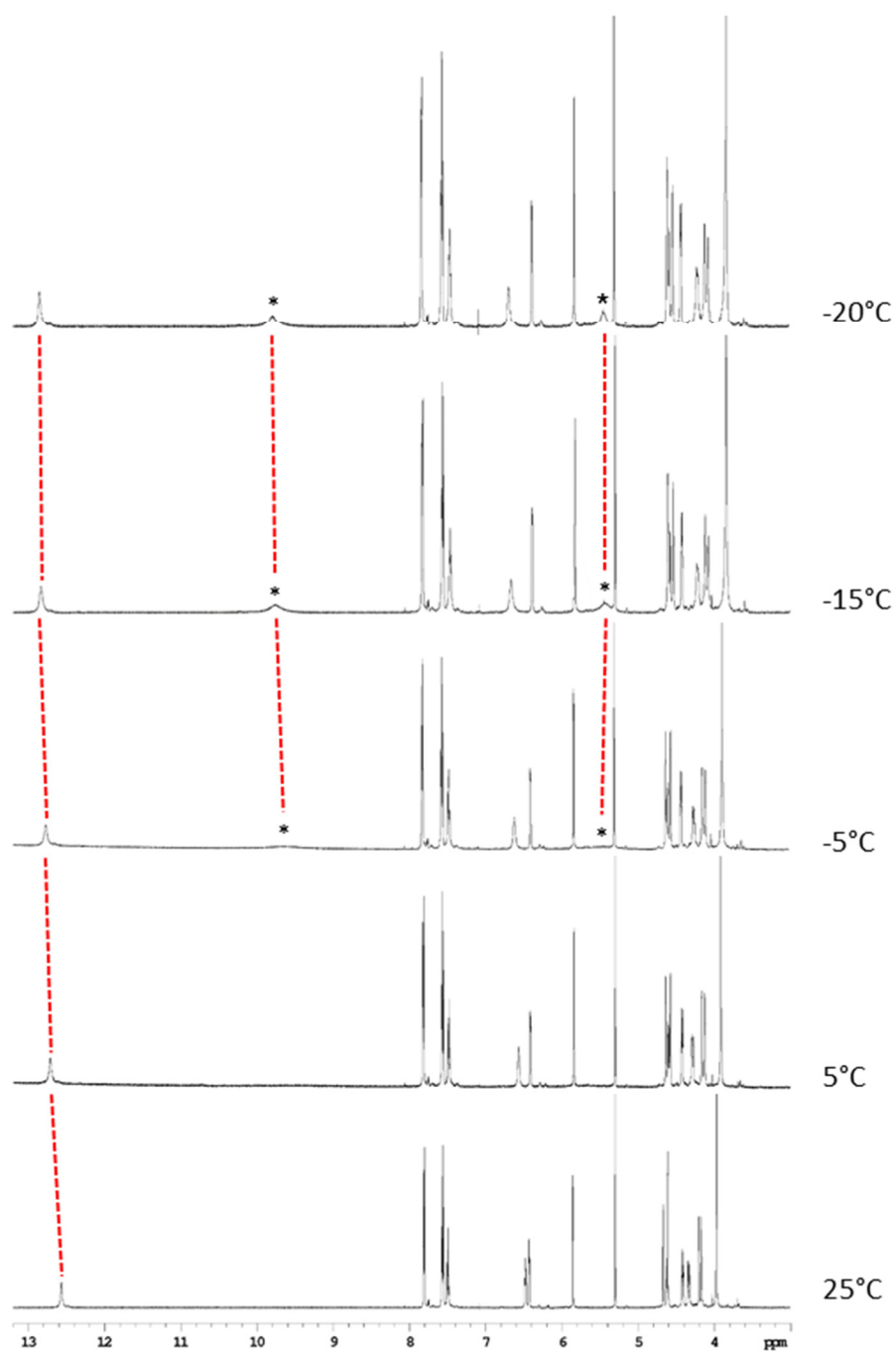


CD spectra recorded on 10 mM  $\text{CH}_2\text{Cl}_2$  solutions of **8Ph5Fc** before (black trace) and after addition of [2.2.2] cryptand (red dotted trace) or excess of KI (red continuous trace). Path length 0.01 cm.

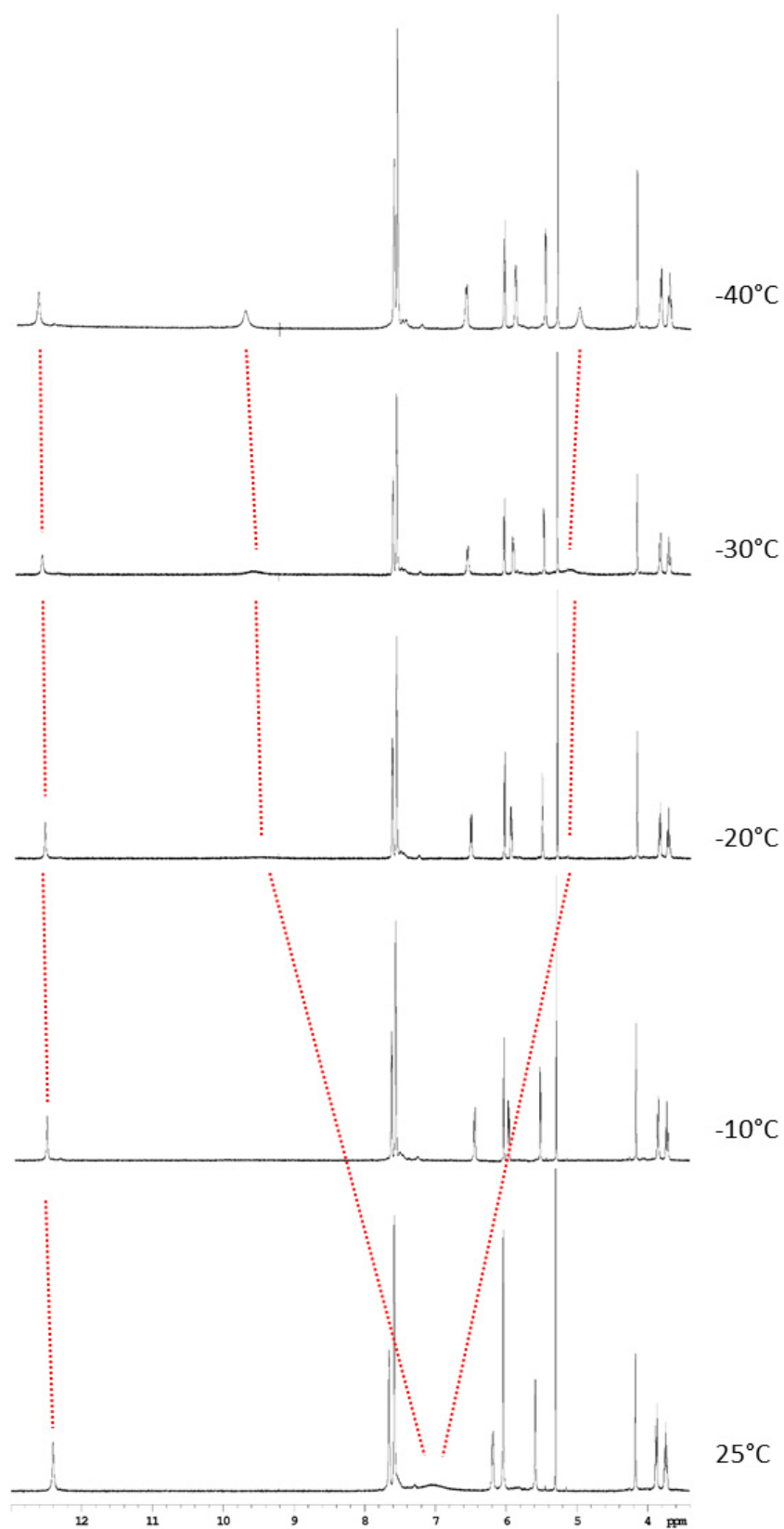


CD spectra recorded on 10 mM  $\text{CH}_2\text{Cl}_2$  solutions of **8Ph5Si** before (blue trace) and after addition of KI (pink trace). Path length 0.01 cm.

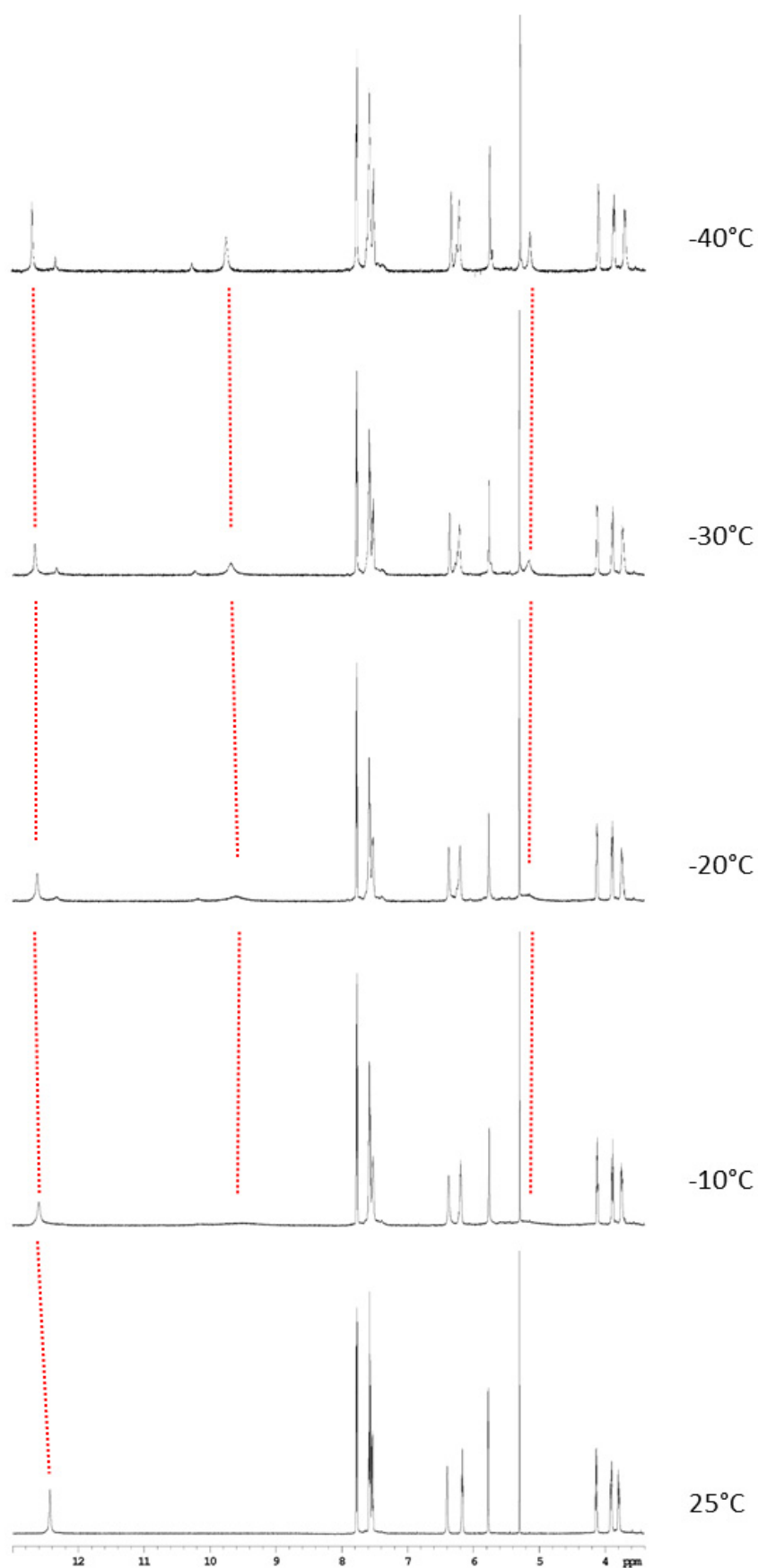
**Figure S1.** Selected CD spectra showing the behaviour of guanosines **1** and **3** upon addition of [2.2.2] cryptand or excess KI.



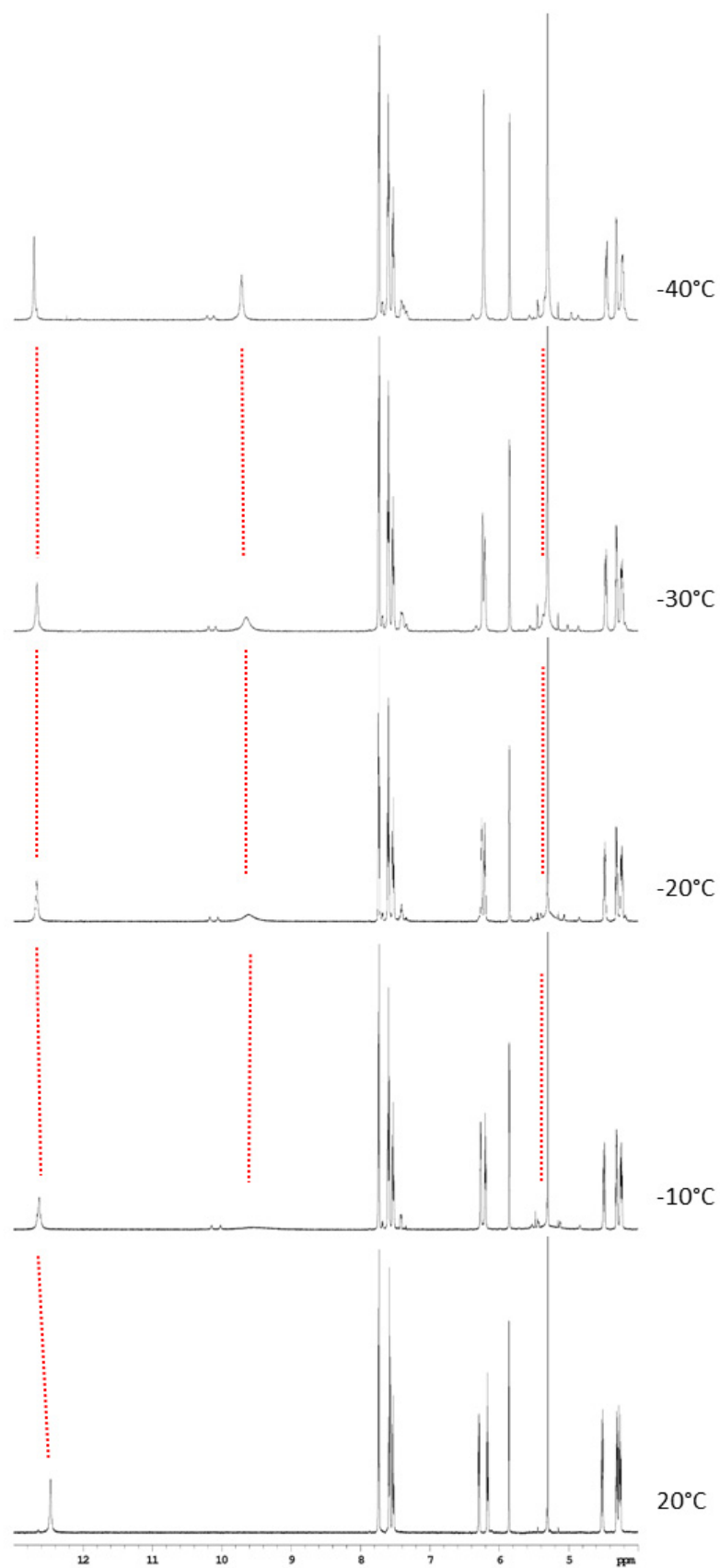
**Figure S2.** Downfield portion of the 600 MHz <sup>1</sup>H-NMR spectrum of **8Ph5Fc** (4.3 mM) at different temperatures in CD<sub>2</sub>Cl<sub>2</sub>. Amino protons are marked with asterisks.



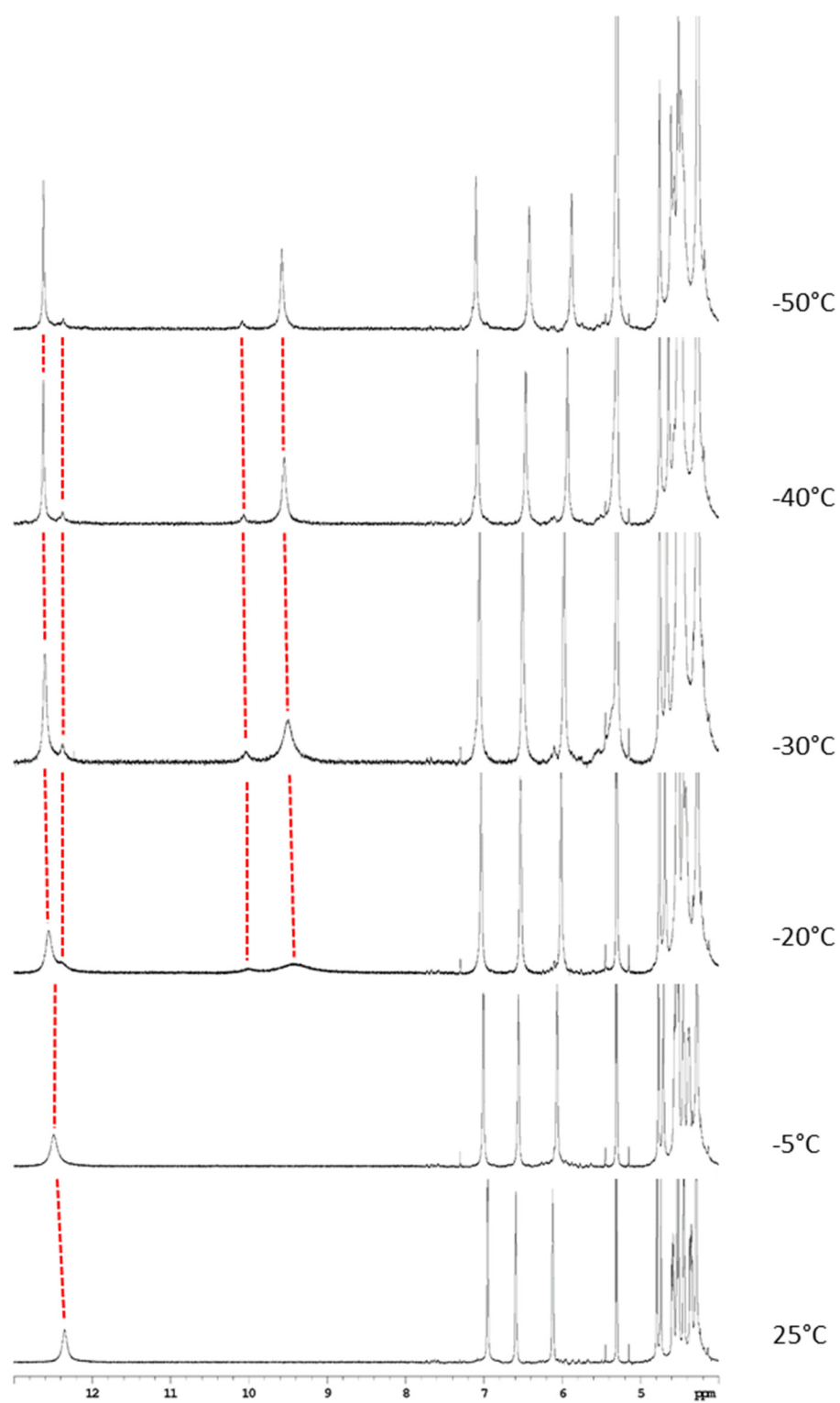
**Figure S3.** Downfield portion of the 600 MHz  $^1\text{H}$ -NMR spectrum of **8Ph5OH** (5 mM) at different temperatures in  $\text{CD}_2\text{Cl}_2$ .



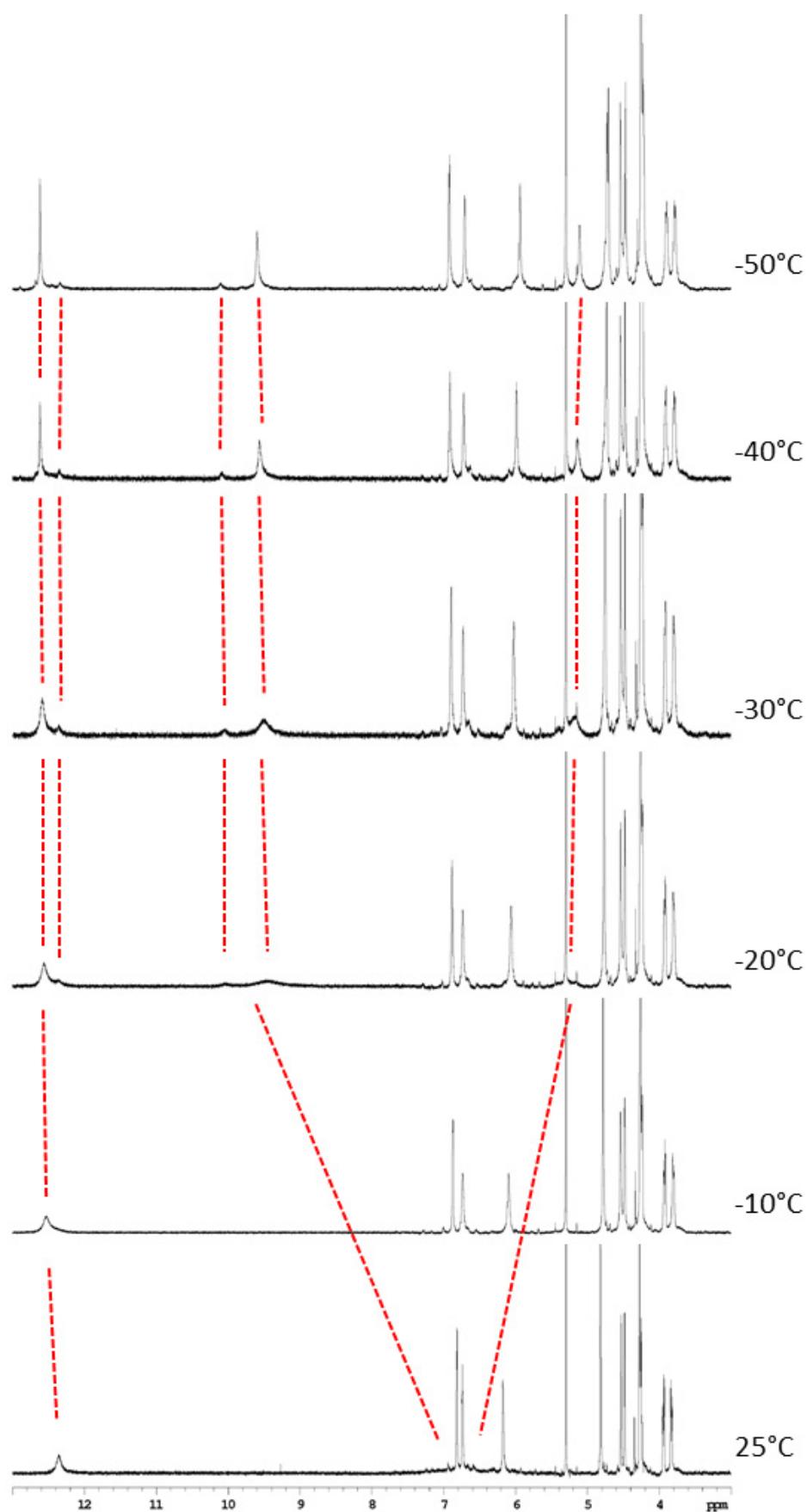
**Figure S4.** Downfield portion of the 600 MHz  $^1\text{H}$ -NMR spectrum of **8Ph5Si** (6 mM) at different temperatures in  $\text{CD}_2\text{Cl}_2$ .



**Figure S5.** Downfield portion of the 600 MHz <sup>1</sup>H-NMR spectrum of **8Ph5C10** (7 mM) at different temperatures in CD<sub>2</sub>Cl<sub>2</sub>.

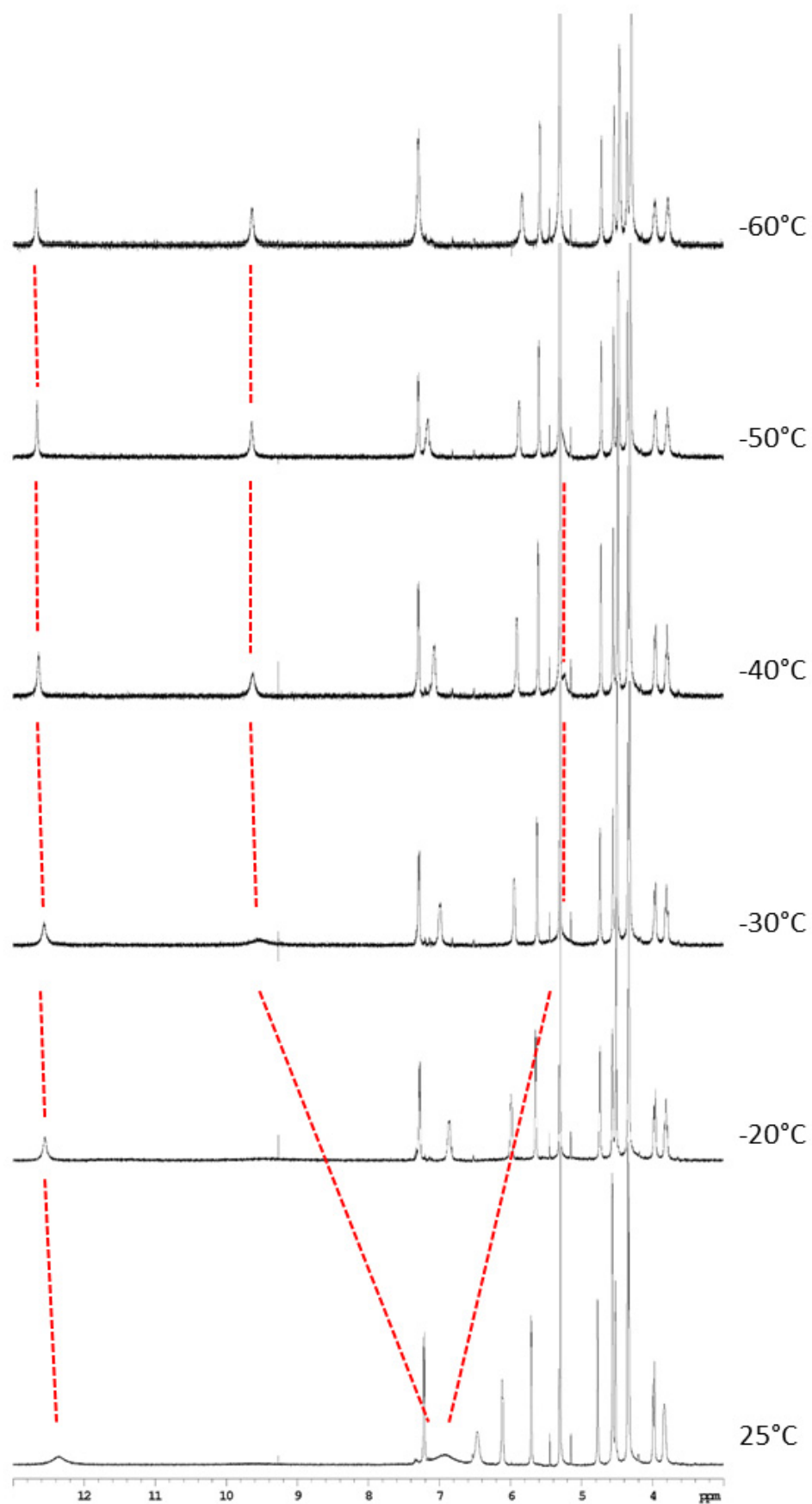


**Figure S6.** Downfield portion of the 600 MHz <sup>1</sup>H-NMR spectrum of **8Fc5C10** (4.5 mM) at different temperatures in CD<sub>2</sub>Cl<sub>2</sub>.

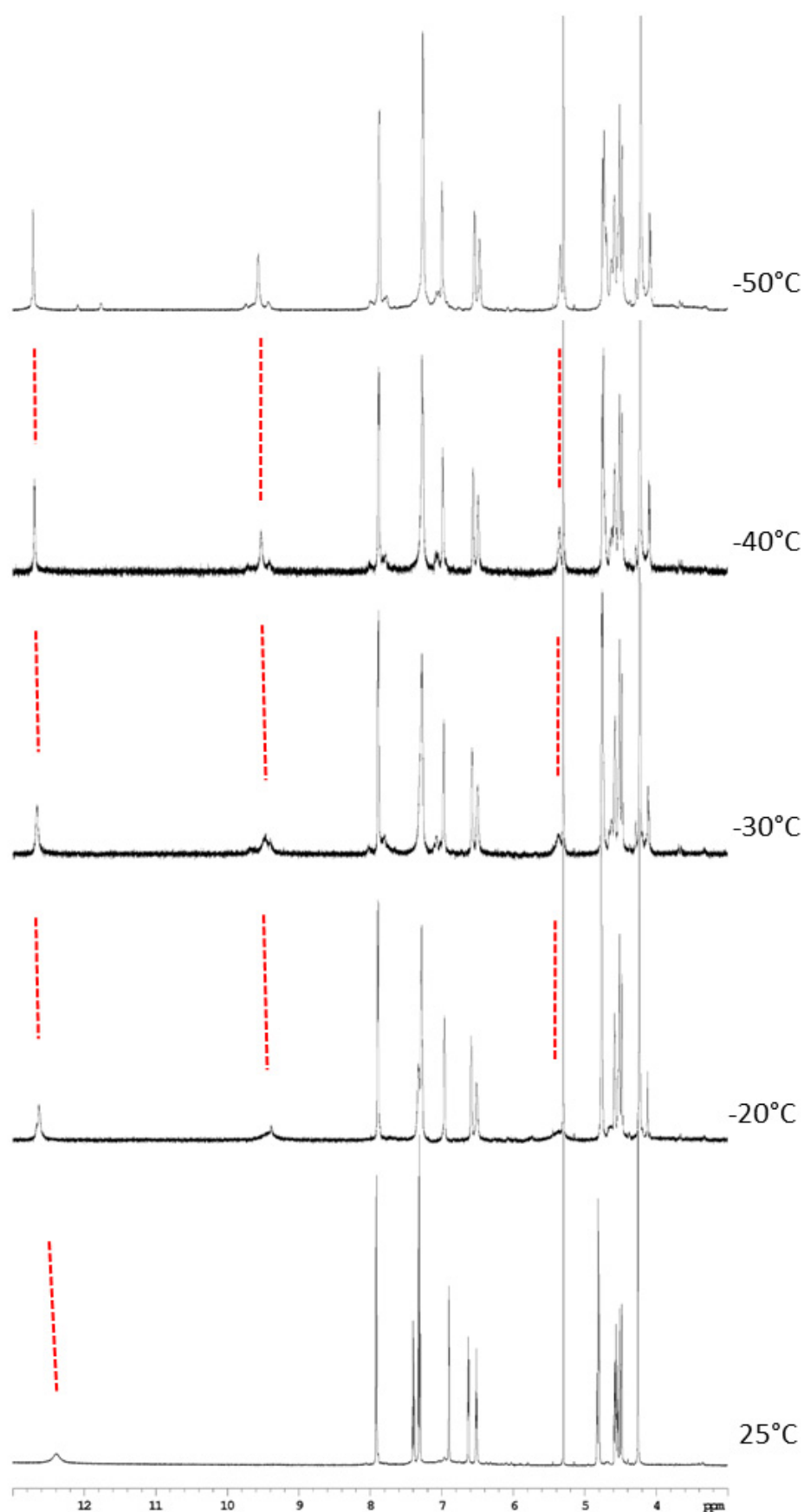


**Figure S7.** Downfield portion of the 600 MHz <sup>1</sup>H-NMR spectrum of **8Fc5Si** (6 mM) at different temperatures in CD<sub>2</sub>Cl<sub>2</sub>.





**Figure S8.** Downfield portion of the 600 MHz <sup>1</sup>H-NMR spectrum of **8Fc5OH** (7 mM) at different temperatures in CD<sub>2</sub>Cl<sub>2</sub>.



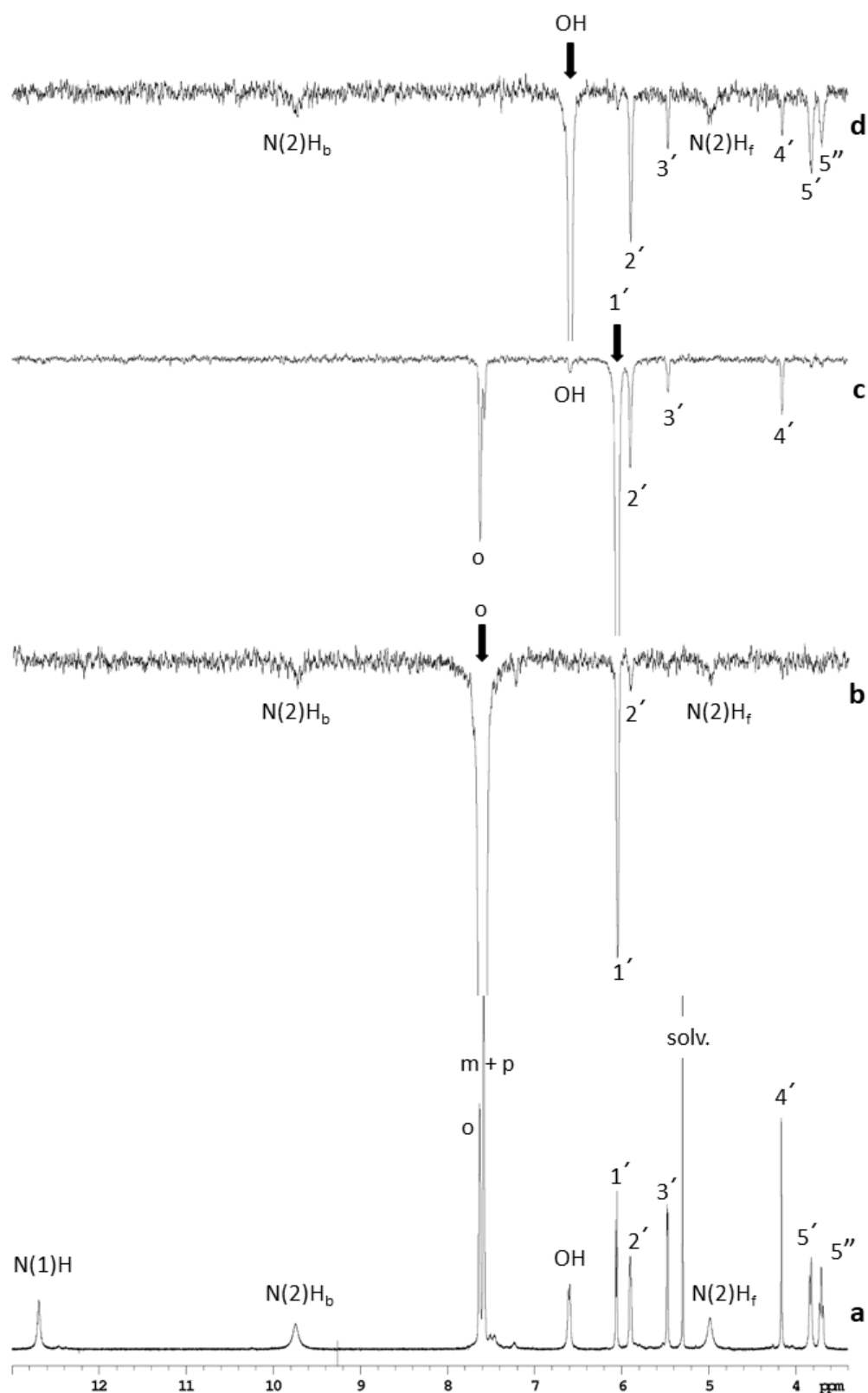
**Figure S9.** Downfield portion of the 600 MHz <sup>1</sup>H-NMR spectrum of **8Fc5Ph** (4 mM) at different temperatures in CD<sub>2</sub>Cl<sub>2</sub>.

**Table S1.** Room temp. chemical shifts\* for sugar protons.

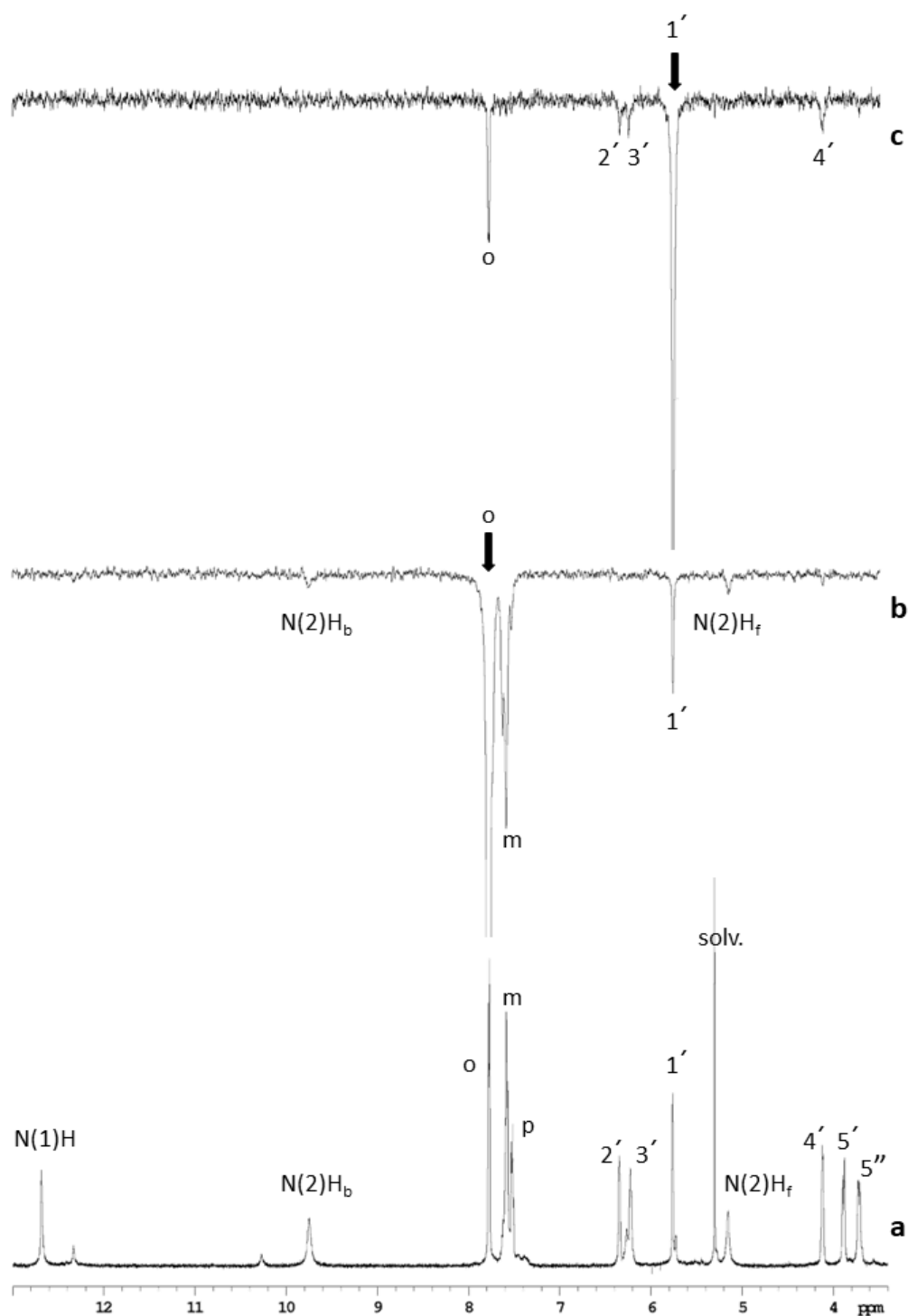
	Compound	solvent	$\delta(1')$	$\delta(2')$	$\delta(3')$	$\delta$ sequence
<b>a</b>	<b>8Ph5Fc</b>	dmsO	5.808	6.31	5.83	2'>3'>1'
		dcm	5.86	6.43	6.48	3'>2'>1'
<b>b</b>	<b>8Ph5OH</b>	dmsO	5.78	5.496	5.186	1'>2'>3'
		dcm	6.2	6.04	5.588	1'>2'>3'
<b>c</b>	<b>8Ph5Si</b>	dmsO	5.75	6.36	6.17	2'>3'>1'
		dcm	5.75	6.26	5.63	2'>1'>3'
<b>d</b>	<b>8Ph5C10</b>	dmsO	-	-	-	-
		dcm	5.86	6.29	6.16	2'>3'>1'
<b>i</b>	<b>8Fc5C10</b>	dmsO	6.75	6.58	5.74	1'>2'>3'
		dcm	6.95	6.59	6.12	1'>2'>3'
<b>e</b>	<b>8Fc5Si</b>	dmsO	6.747	6.69	5.71	1'>2'>3'
		dcm	6.82	6.74	6.18	1'>2'>3'
<b>f</b>	<b>8Fc5OH</b>	dmsO	6.86	5.61	5.3	1'>2'>3'
		dcm	7.22	6.12	5.71	1'>2'>3'
<b>g</b>	<b>8Fc5Ph</b>	dmsO	6.77	6.63	5.99	1'>2'>3'
		dcm	6.9	6.63	6.51	1'>2'>3'
<b>h</b>	8-Bromo-2,3,5-tri-O-decanoylguanosine <b>1</b>	dmsO	5.84	6.02	5.68	2'>1'>3'
<b>i</b>	2,3,5-tri-O-decanoylguanosine <b>2</b>	dmsO	5.96	5.81	5.52	1'>2'>3'
<b>l</b>	2,3,5-tri-O-acetylguanosine	dmsO <sup>3</sup>	6.007	5.809	5.515	1'>2'>3'
<b>m</b>	8-Bromoguanosine <b>9</b>	dmsO <sup>4</sup>		4.89		
		dmsO	5.68	5.01	4.14	1'>2'>3'
<b>n</b>	Guanosine	dmsO <sup>4</sup>		4.36		
		dmsO <sup>3</sup>	5.723	4.429	4.113	1'>2'>3'

\* signals are referenced to residual solvent peak.

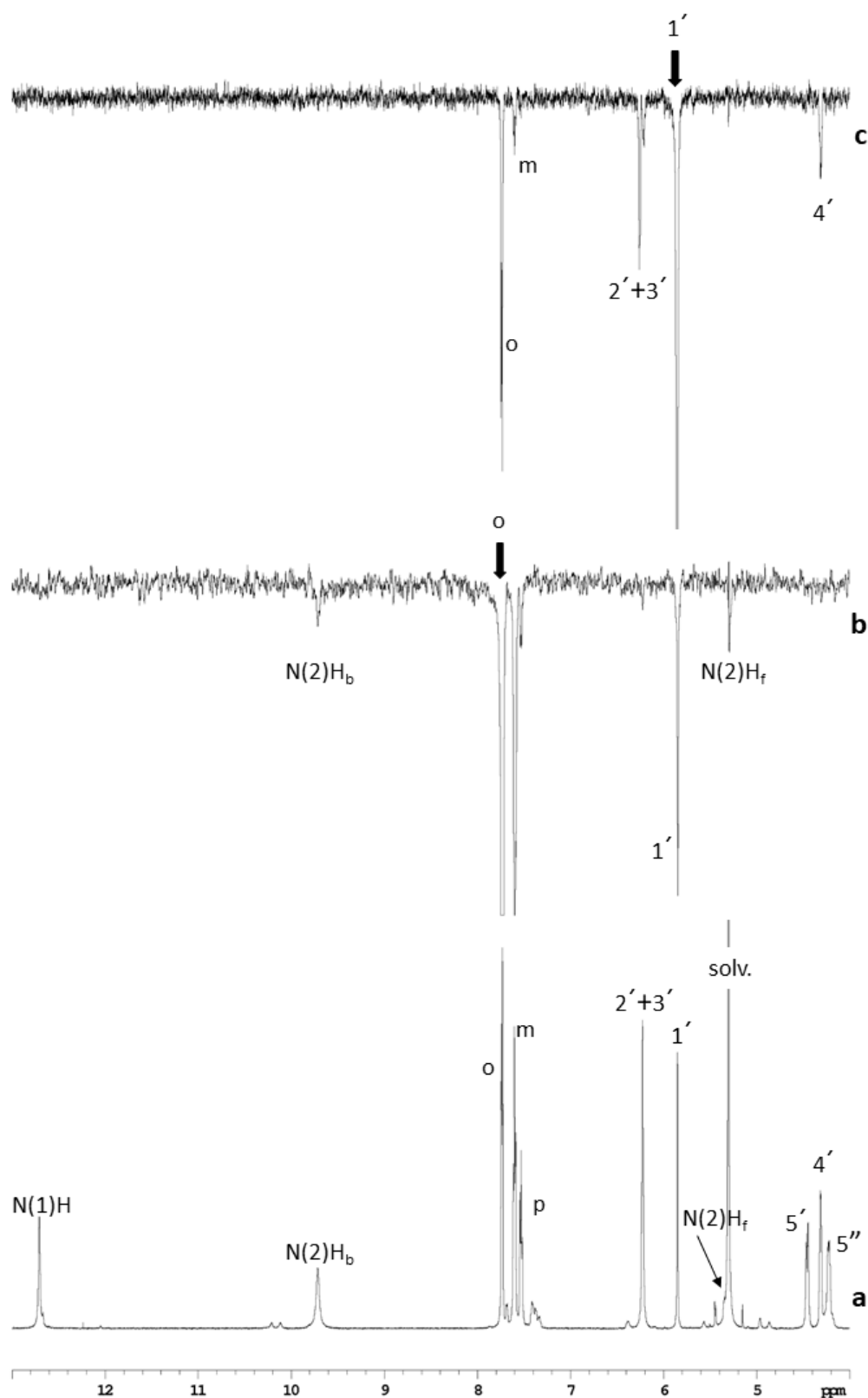
1. Prepared as described in Giorgi, T.; Lena, S.; Mariani, P.; Cremonini, M. A.; Masiero, S.; Pieraccini, S.; Rabe, J. P.; Samori, P.; Spada, G. P.; Gottarelli, G. *J. Am. Chem. Soc.* 2003, **125**, 14741
2. Prepared as described in Devetak, M.; Masiero, S.; Pieraccini, S.; Spada, G. P.; Copic, M.; Olenik, I. D. *Appl. Surf. Sci.* 2010, **256**, 2038
3. Spectral Database for Organic Compounds ([https://sdbs.db.aist.go.jp/sdbs/cgi-bin/cre\\_disclaimer.cgi?REQURL=/sdbs/cgi-bin/cre\\_index.cgi&REFURL=http://www.bing.com/search%3fq=spetra+database+japan&form=PRASU1&src=IE11TR&pc=ASTE](https://sdbs.db.aist.go.jp/sdbs/cgi-bin/cre_disclaimer.cgi?REQURL=/sdbs/cgi-bin/cre_index.cgi&REFURL=http://www.bing.com/search%3fq=spetra+database+japan&form=PRASU1&src=IE11TR&pc=ASTE))
4. L. E. Buerkle, H. A. von Recumab, S. J. Rowan, *Chem. Sci.*, 2012, **3**, 564 and references cited therein.



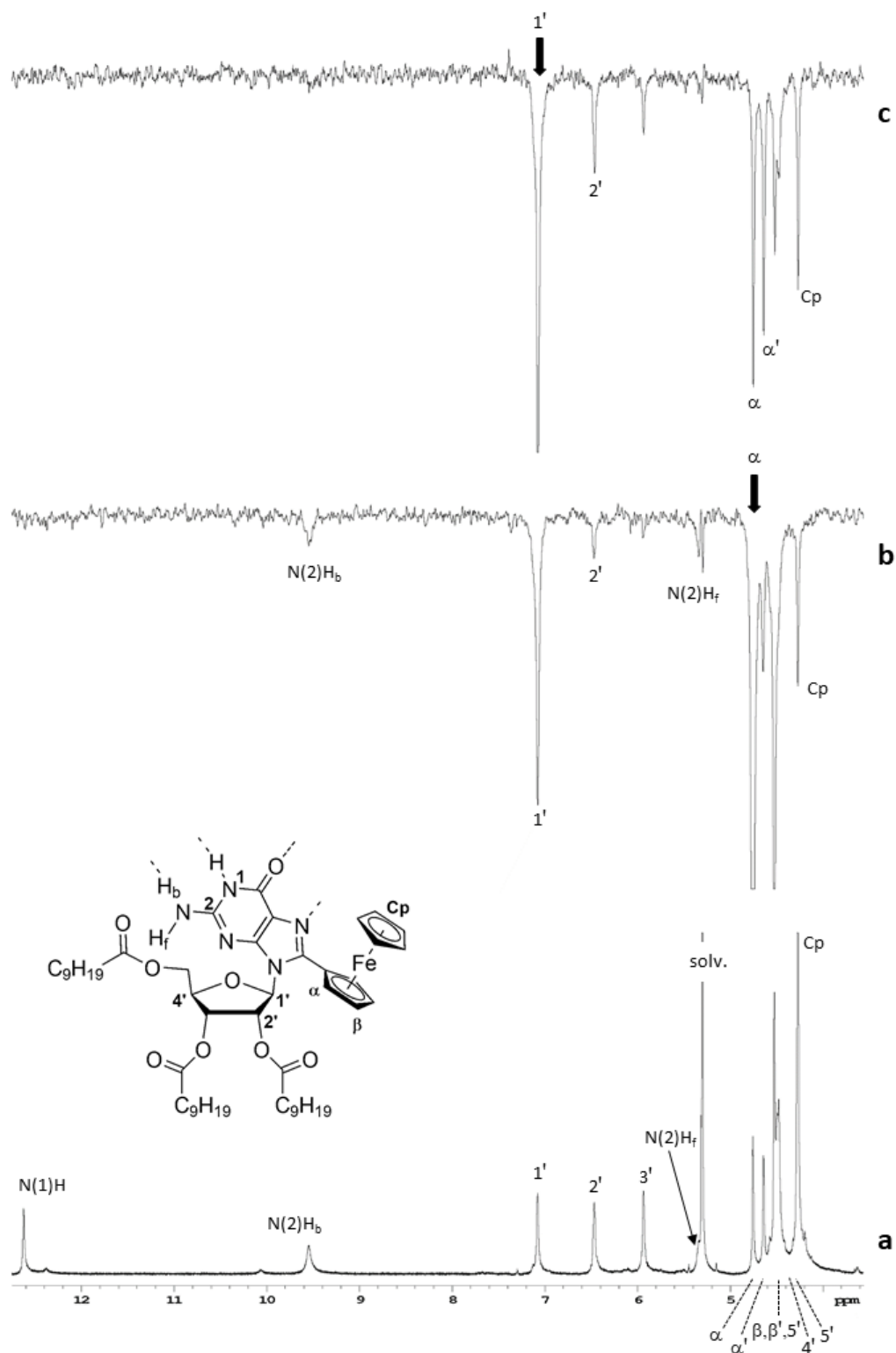
**Figure S10.** a) Downfield portion of the 600 MHz  $^1\text{H}$ -NMR spectrum of **8Ph5OH** in  $\text{CD}_2\text{Cl}_2$  (5 mM) and signals assignment (diastereotopic protons were not assigned); b), c), d): selected NOESY-1D spectra of the same sample. Irradiated signals are indicated by an arrow. In each NOE spectrum were used at least 1024 coadded transients, a recycle delay of 1 sec, a mixing time of 0.25 sec and a 20-50Hz shaped pulse. All spectra were recorded at  $-40^\circ\text{C}$ .



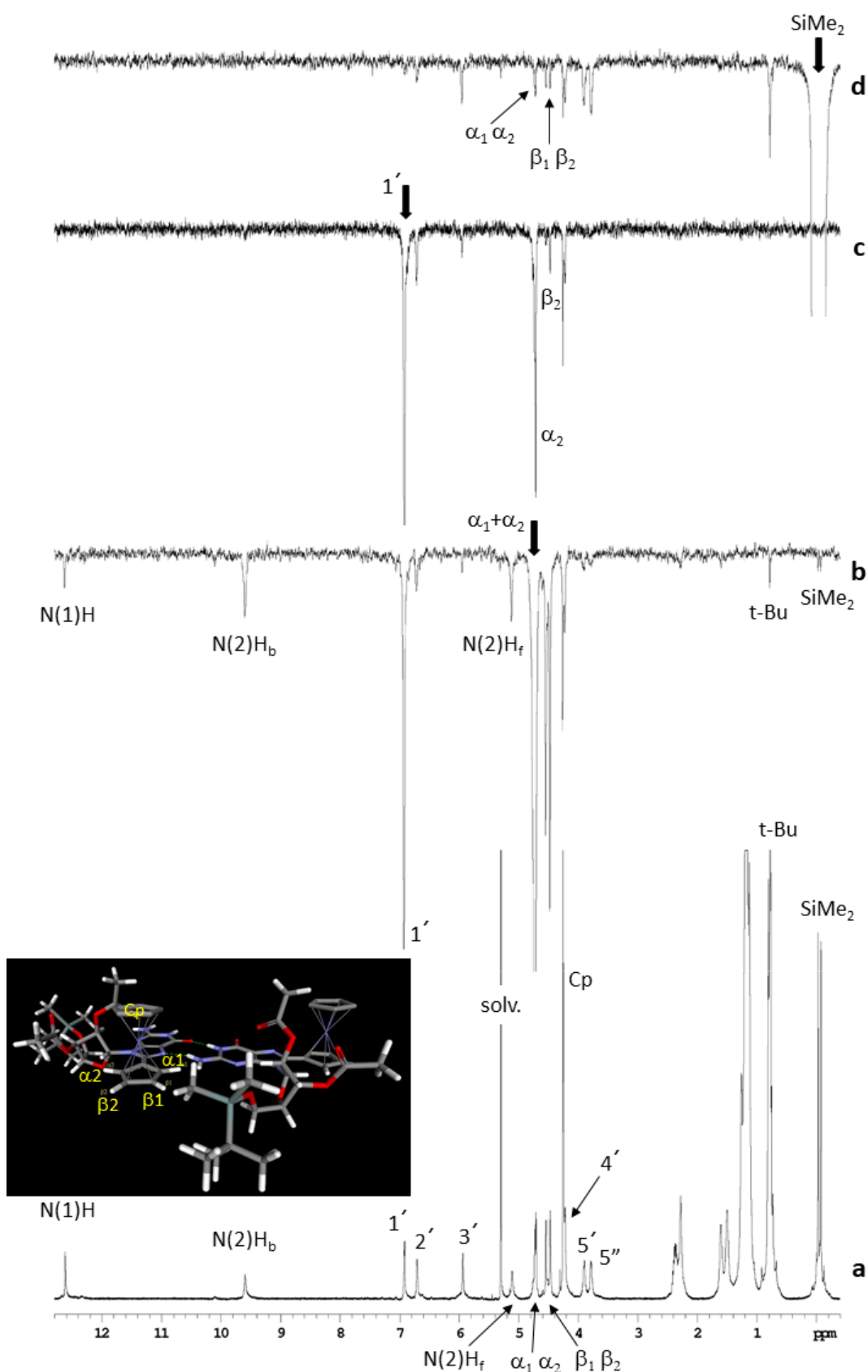
**Figure S11.** a) Downfield portion of the 600 MHz  $^1\text{H}$ -NMR spectrum of **8Ph5Si** in  $\text{CD}_2\text{Cl}_2$  (6 mM) and signals assignment (diastereotopic protons were not assigned); b), c): selected NOESY-1D spectra of the same sample. Irradiated frequencies are indicated by an arrow. In each NOE spectrum were used at least 512 coadded transients, a recycle delay of 1 sec, a mixing time of 0.2 sec and a 20-50Hz shaped pulse. All spectra were recorded at  $-40^\circ\text{C}$ .



**Figure S12.** a) Downfield portion of the 600 MHz  $^1\text{H}$ -NMR spectrum of **8Ph5C10** in  $\text{CD}_2\text{Cl}_2$  (7 mM) and signals assignment (diastereotopic protons were not assigned); b), c): selected NOESY-1D spectra of the same sample. Irradiated signals are indicated by an arrow. In each NOE spectrum were used at least 256 coadded transients, a recycle delay of 1 sec, a mixing time of 0.4 sec and a 20-50Hz shaped pulse. All spectra were recorded at  $-40^\circ\text{C}$ .

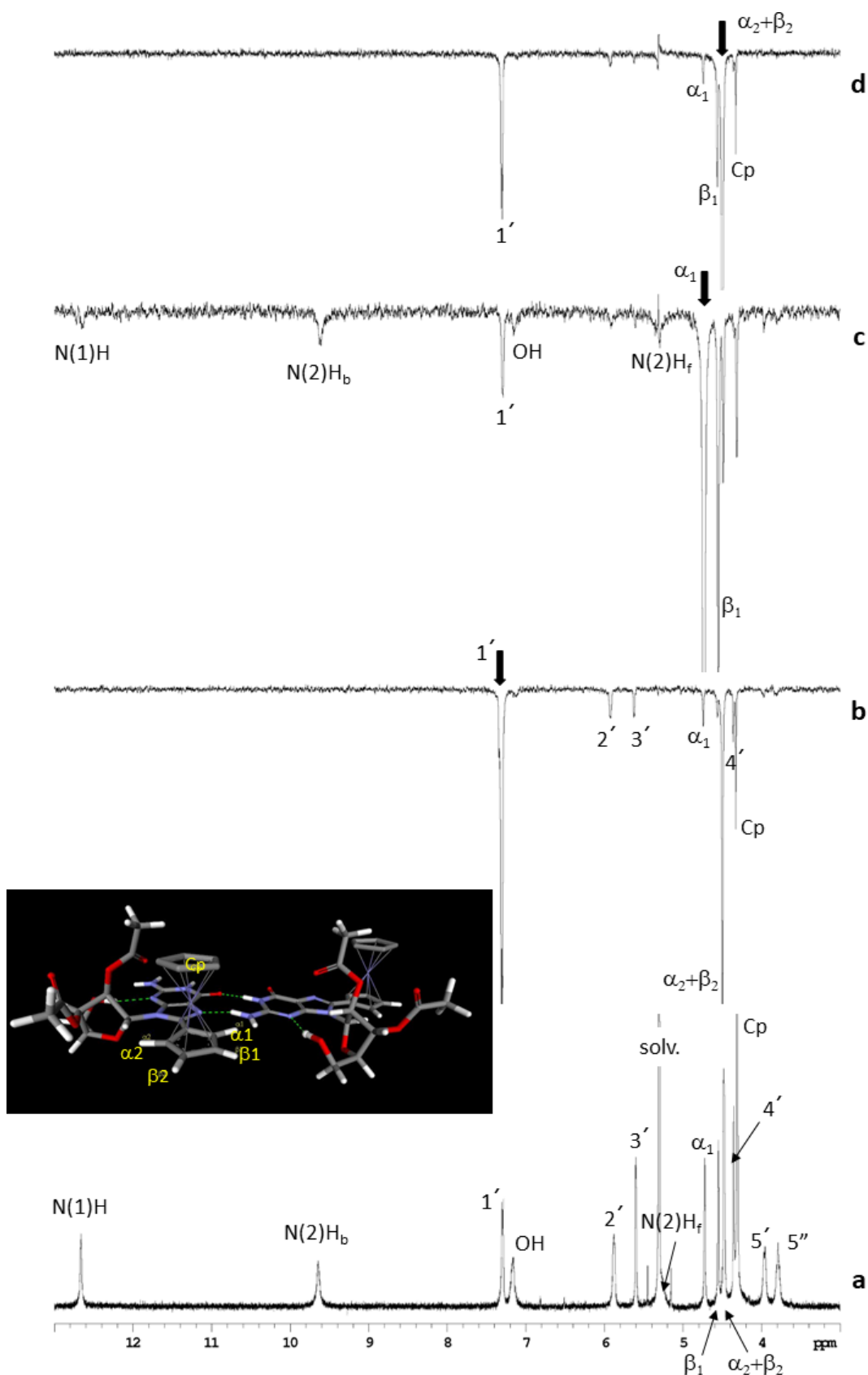


**Figure S13.** a) Downfield portion of the 600 MHz  $^1\text{H}$ -NMR spectrum of **8Fc5C10** in  $\text{CD}_2\text{Cl}_2$  (4.5mM) and signals assignment (diastereotopic protons were not assigned); b), c): selected NOESY-1D spectra of the same sample. Irradiated frequencies are indicated by an arrow. In each NOE spectrum were used 512 coadded transients, a recycle delay of 0.6 sec, a mixing time of 0.6 sec and a 50Hz shaped pulse. All spectra were recorded at  $-40^\circ\text{C}$ .

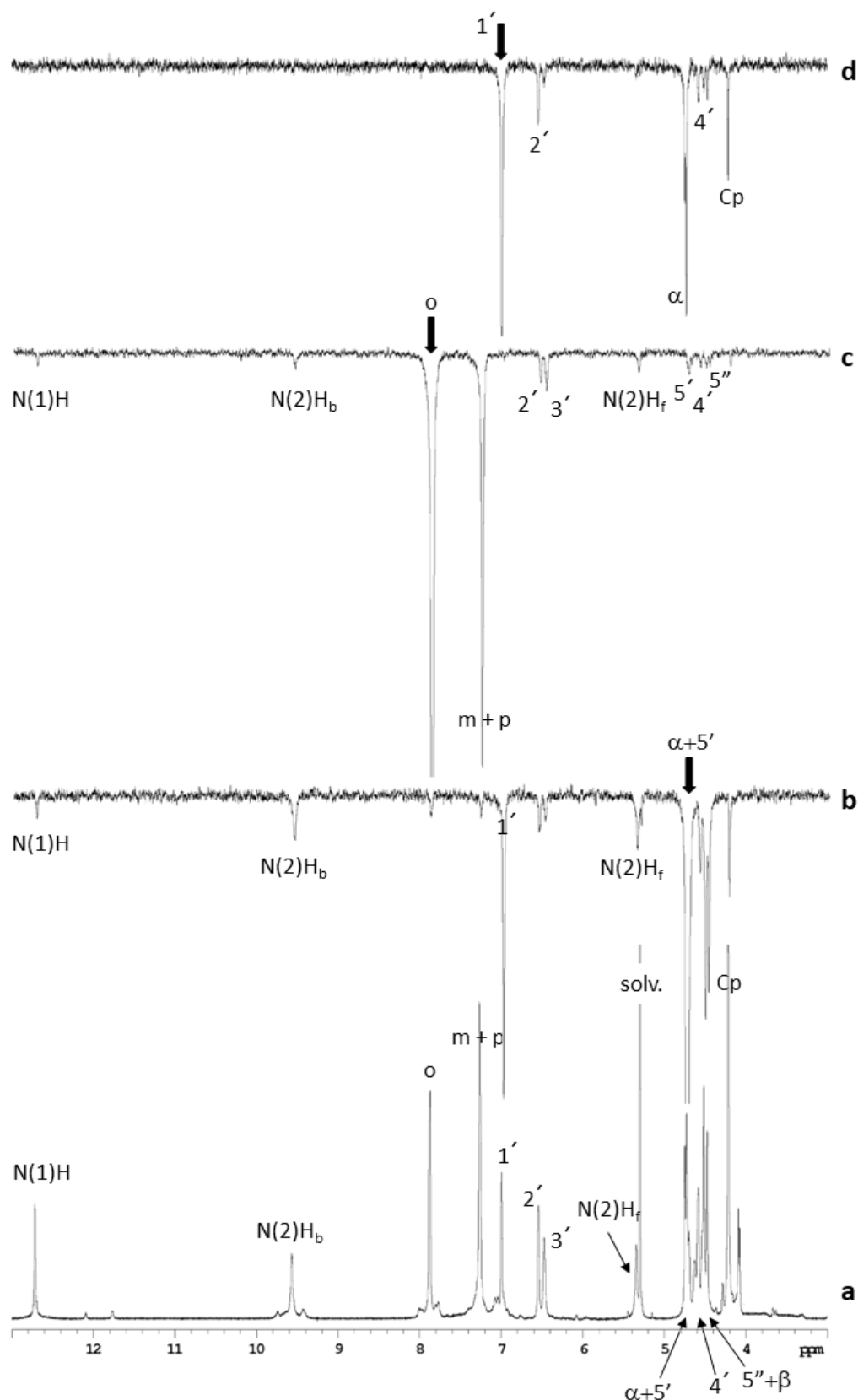


**Figure S14.** a) 600 MHz  $^1\text{H}$ -NMR spectrum of **8Fe5Si** in  $\text{CD}_2\text{Cl}_2$  (6 mM) and signals assignment (sugar diastereotopic protons were not assigned); b), c), d): selected NOESY-1D spectra of the same sample. Irradiated signals are indicated by an arrow. See inset for proton labeling. In each NOE spectrum were used at least 256 coadded transients, a recycle delay of 1 sec, a mixing time of 0.35 sec and a 20-80Hz shaped pulse. All spectra were recorded at  $-50^\circ\text{C}$ .

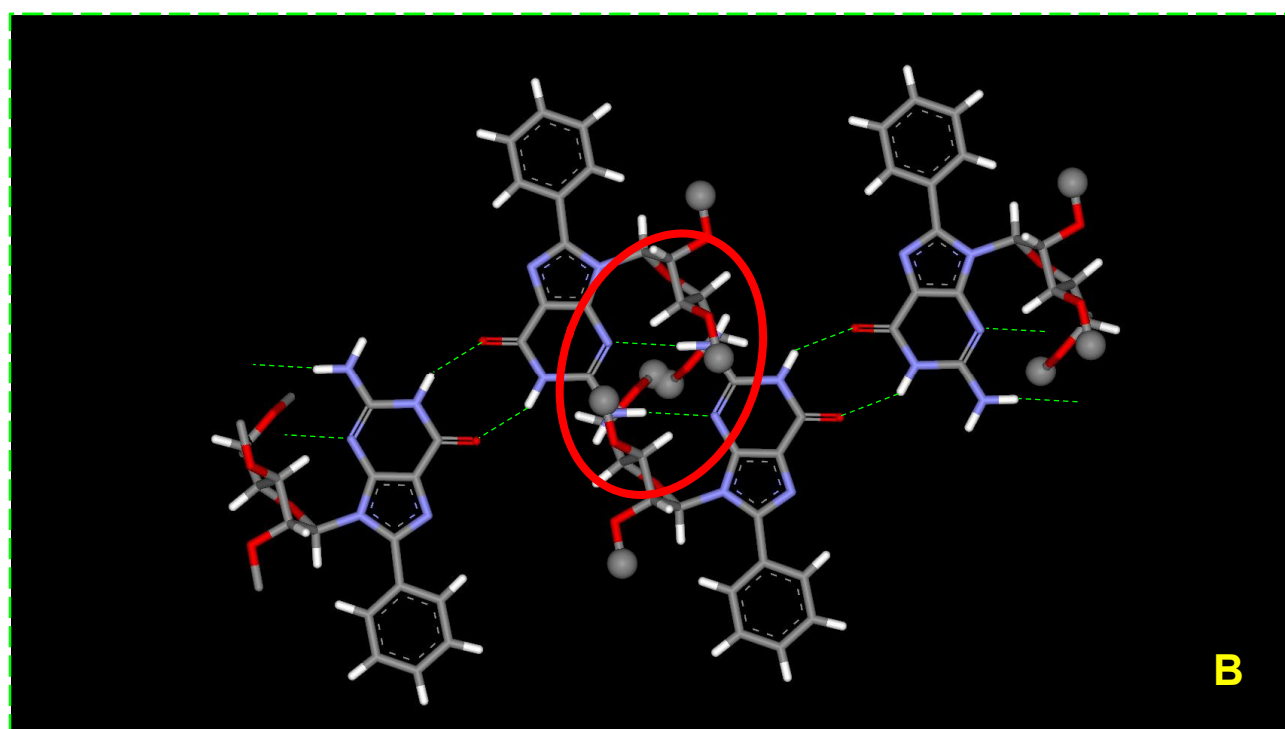
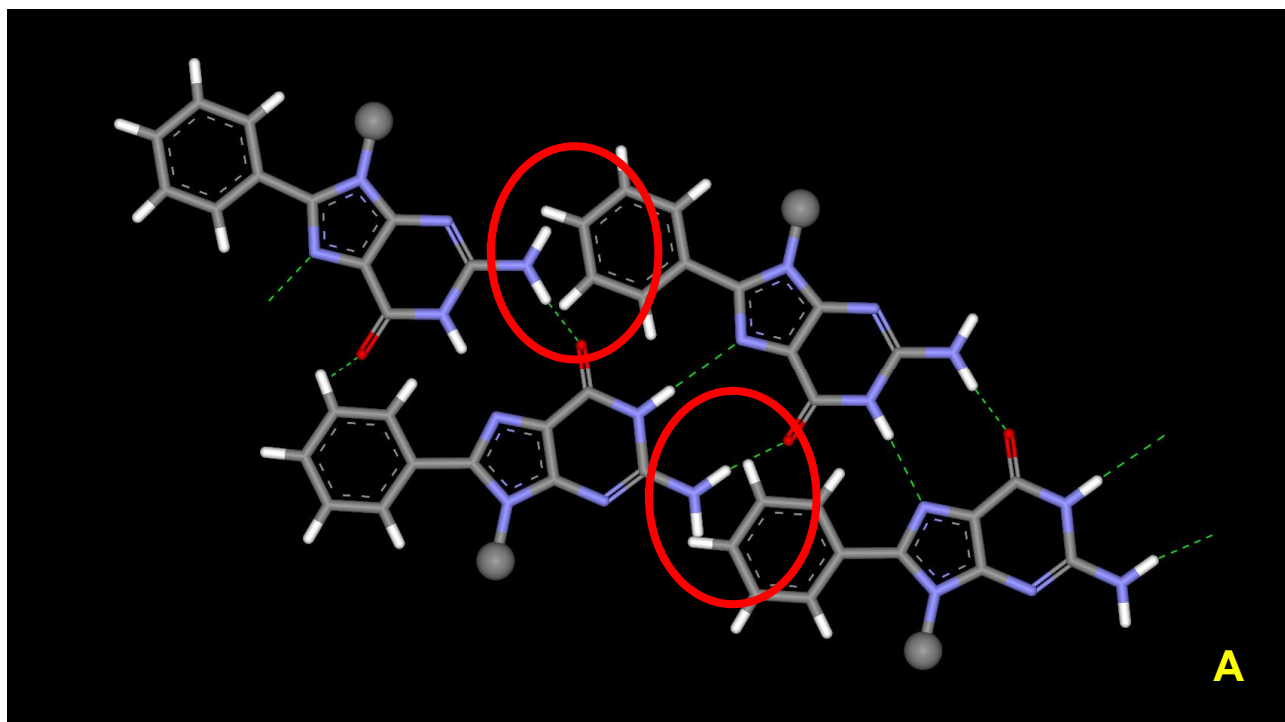




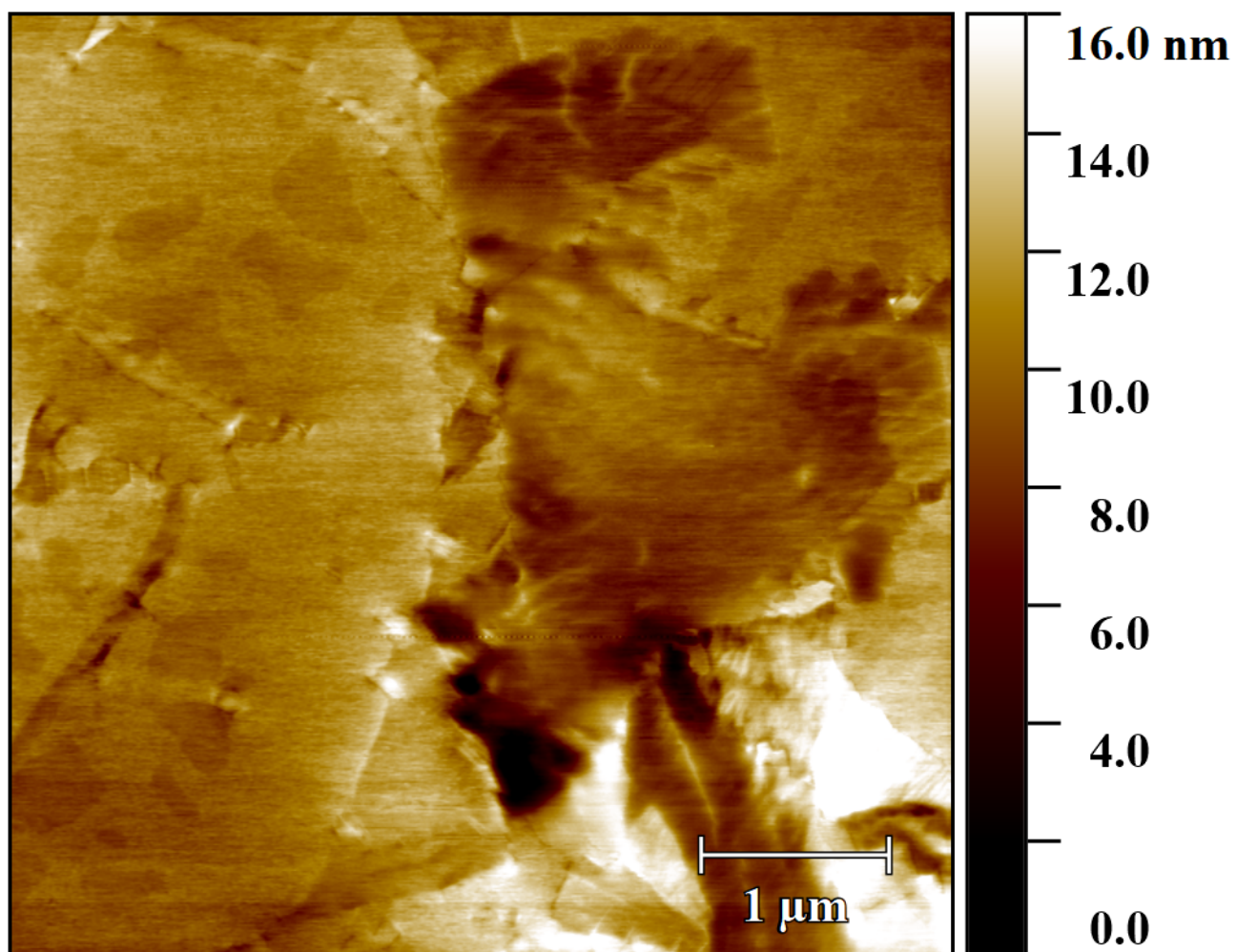
**Figure S15.** a) Downfield portion of the 600 MHz  $^1\text{H}$ -NMR spectrum of **8Fc5OH** in  $\text{CD}_2\text{Cl}_2$  (7 mM) and signals assignment (ribose diastereotopic protons were not assigned); b), c), d): selected NOESY-1D spectra of the same sample. Irradiated frequencies are indicated by an arrow. See inset for proton labeling. In each NOE spectrum were used at least 128 coadded transients, a recycle delay of 1 sec, a mixing time of 0.4 sec and a 20-50Hz shaped pulse. All spectra were recorded at  $-50^\circ\text{C}$ .



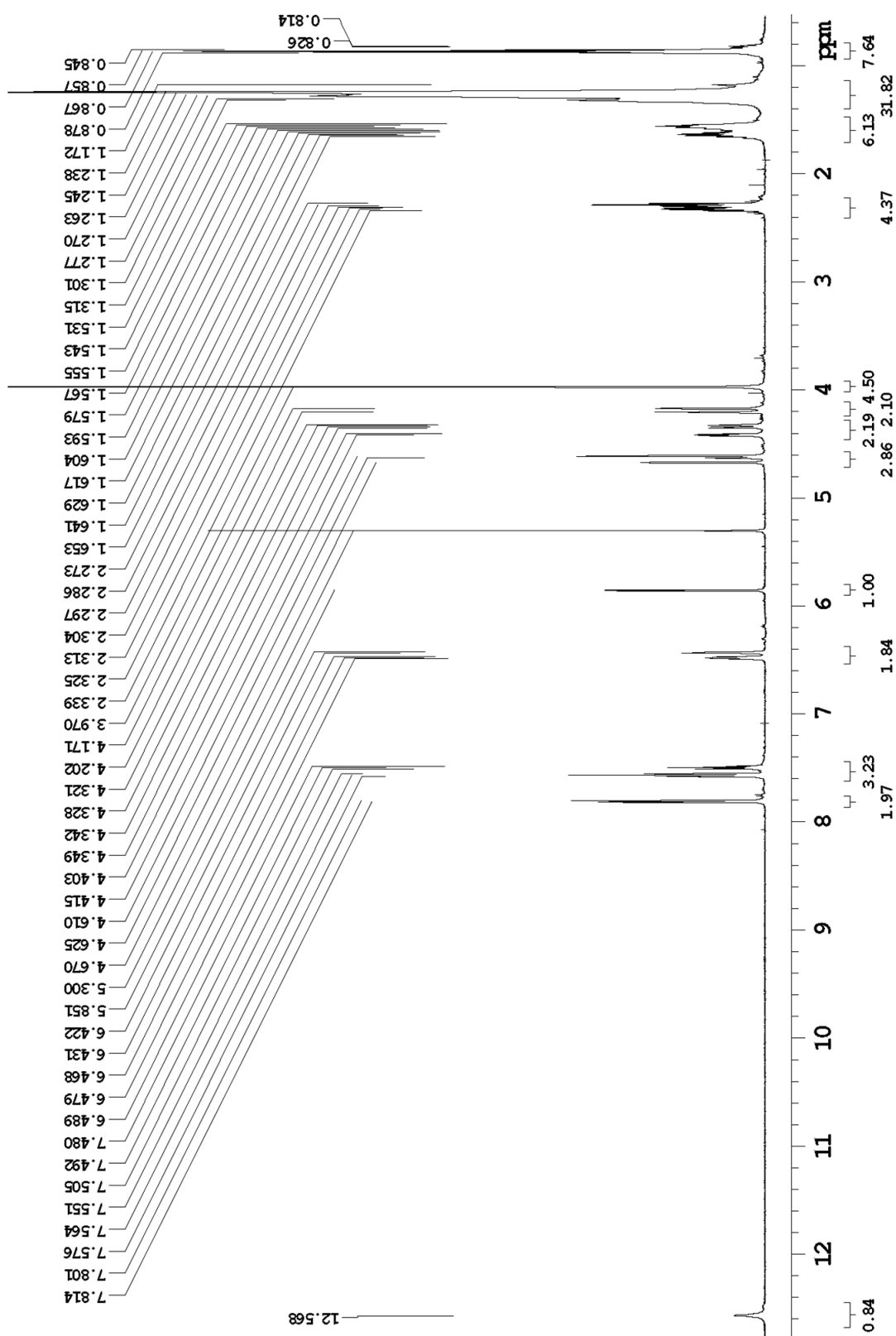
**Figure S16.** a) Downfield portion of the 600 MHz  $^1\text{H}$ -NMR spectrum of **8Fc5Ph** in  $\text{CD}_2\text{Cl}_2$  (4 mM) and signals assignment (diastereotopic protons were not assigned); b), c), d): selected NOESY-1D spectra of the same sample. Irradiated protons are indicated by an arrow. In each NOE spectrum were used at least 256 coadded transients, a recycle delay of 1 sec, a mixing time of 0.4 sec and a 20-50Hz shaped pulse. All spectra were recorded at  $-50^\circ\text{C}$ .



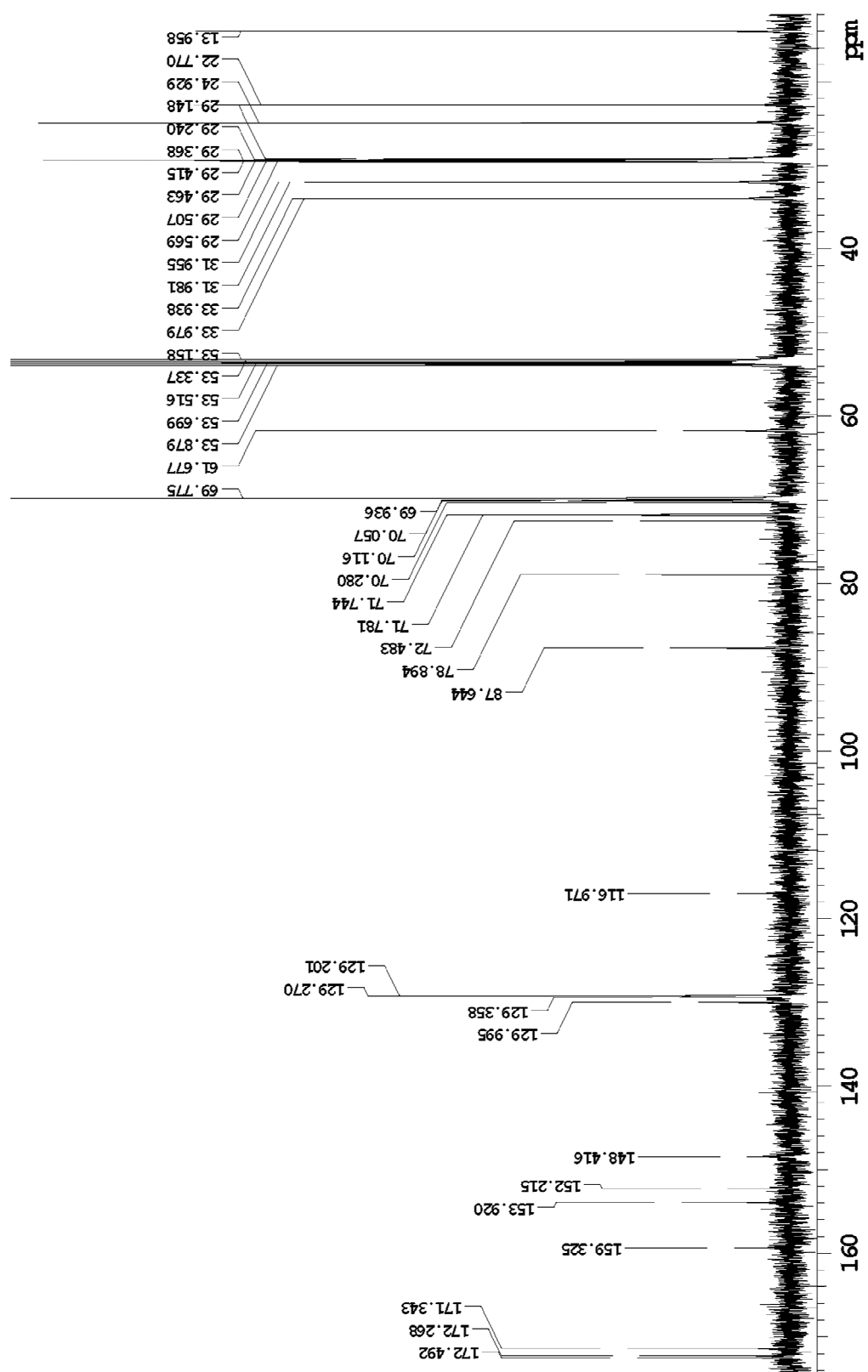
**Figure S17.** Sketches of ribbon-like structures A and B hypothetically formed by 8-substituted guanosine in syn conformation. Sterically overcrowded areas are circled. Furthermore, both structures are incompatible with observed NOEs.



**Figure S18.** AFM image of the amorphous film obtained from **8Ph5C10** by drop-casting from  $\text{CH}_2\text{Cl}_2$ .



<sup>1</sup>H-NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Ph5Fc**



<sup>13</sup>C{<sup>1</sup>H} NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Ph5Fc**

i600 std parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.000 sec  
Acq. time 0.213 sec  
Width 9611.9 Hz  
2D Width 9611.9 Hz  
2 repetitions  
256 increments

OBSERVE HL, 599.7287472 MHz

DATA PROCESSING

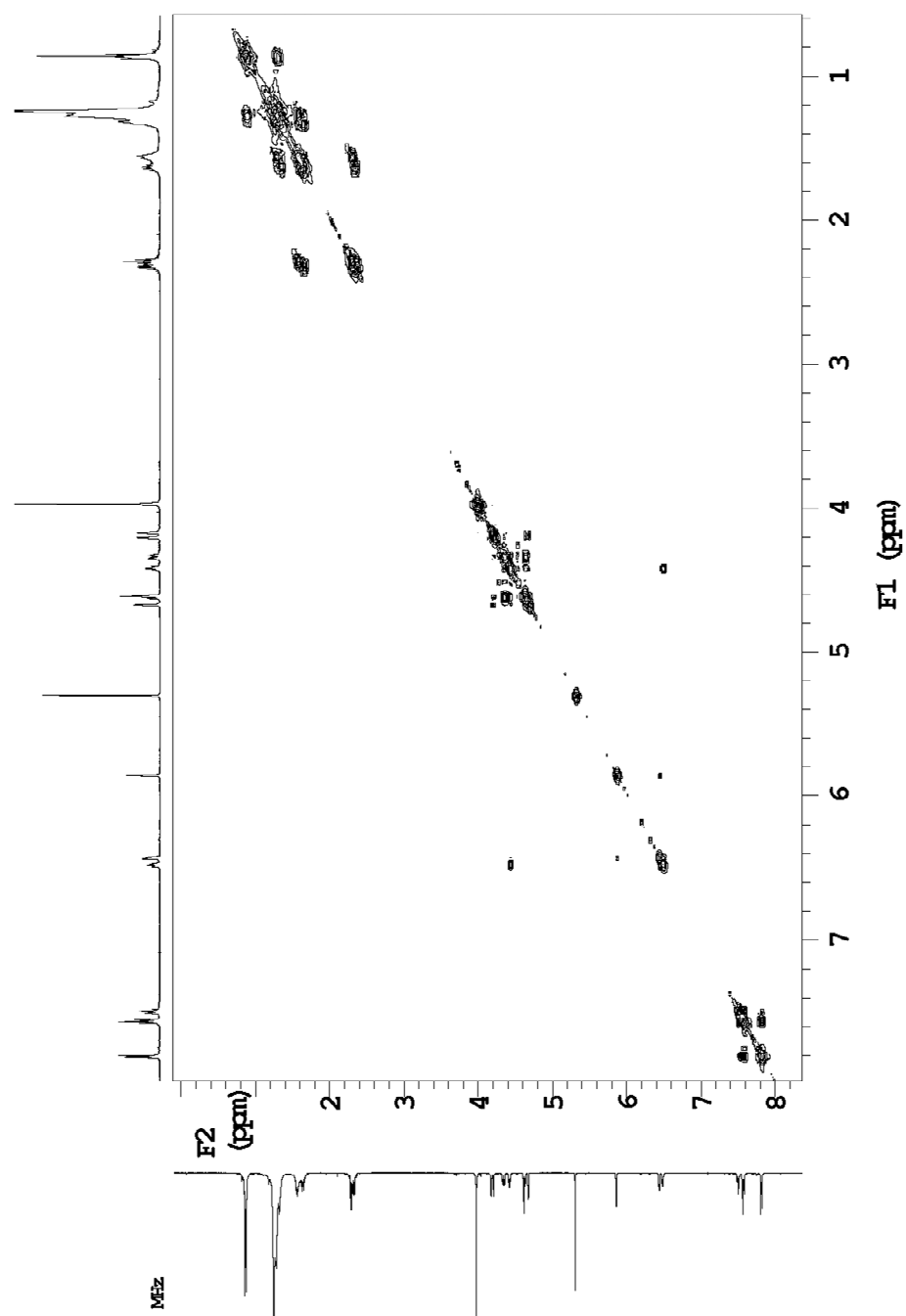
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Total time 0 min 0 sec



gCOSY spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Ph5Fc**

i600 std parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

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Acq. time 0.199 sec  
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2D Width 25632.8 Hz  
8 repetitions

2 x 256 increments  
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DECOUPLE C13, 150.8132215 MHz  
Power 45 dB

on during acquisition  
off during delay  
W40 Triple modulated

DATA PROCESSING

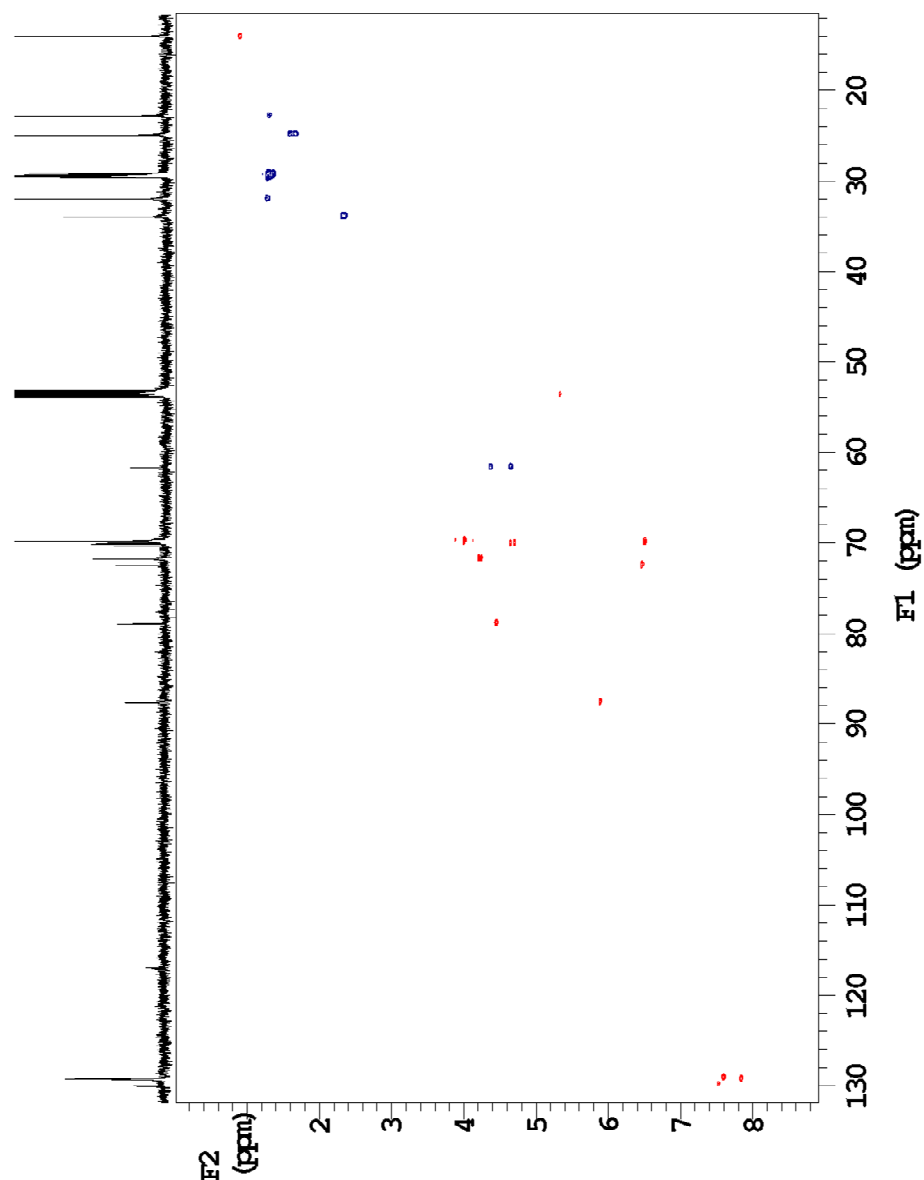
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Total time 0 min 0 sec



gHSQC spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Ph5Fc**



i600 std parameters

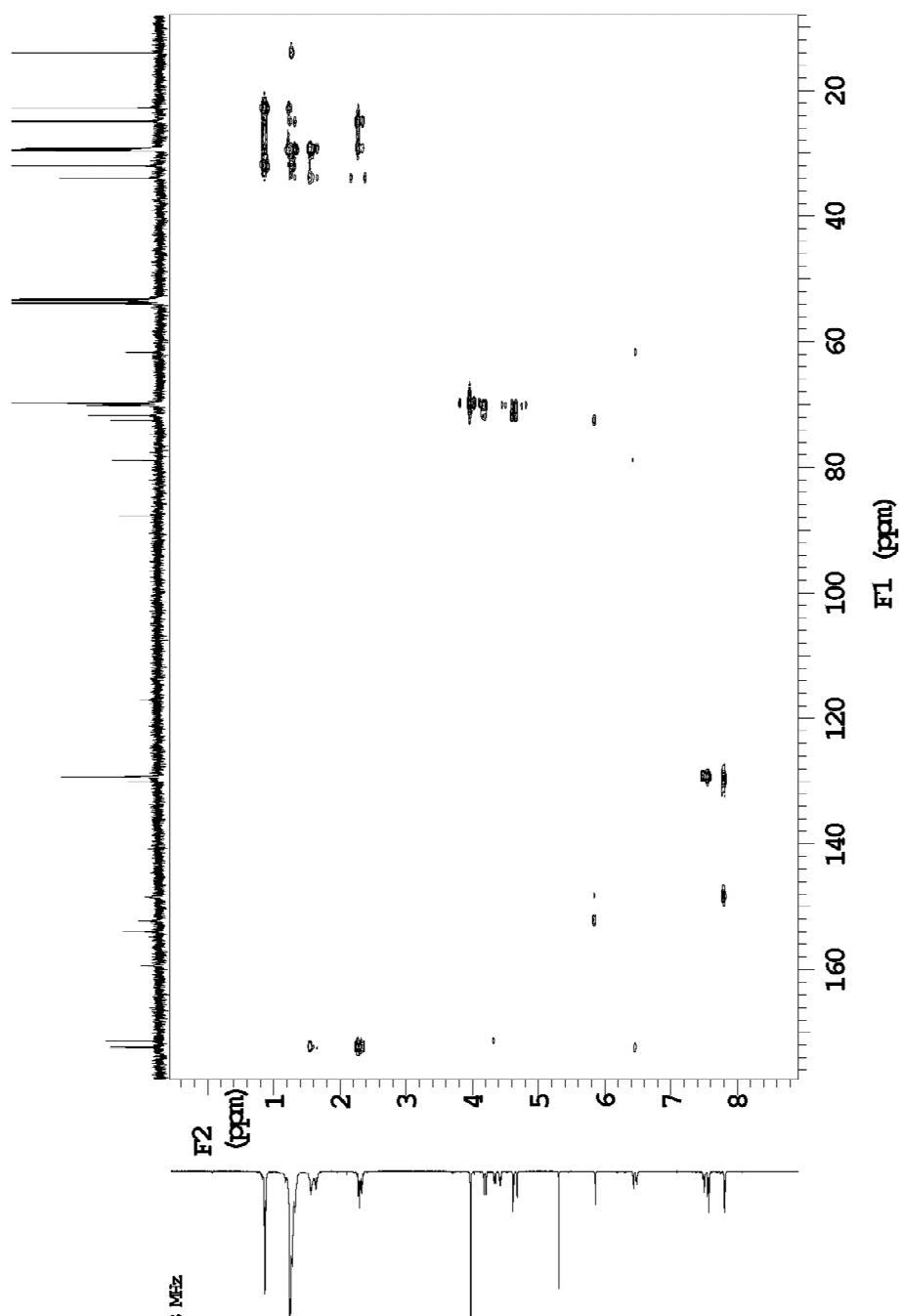
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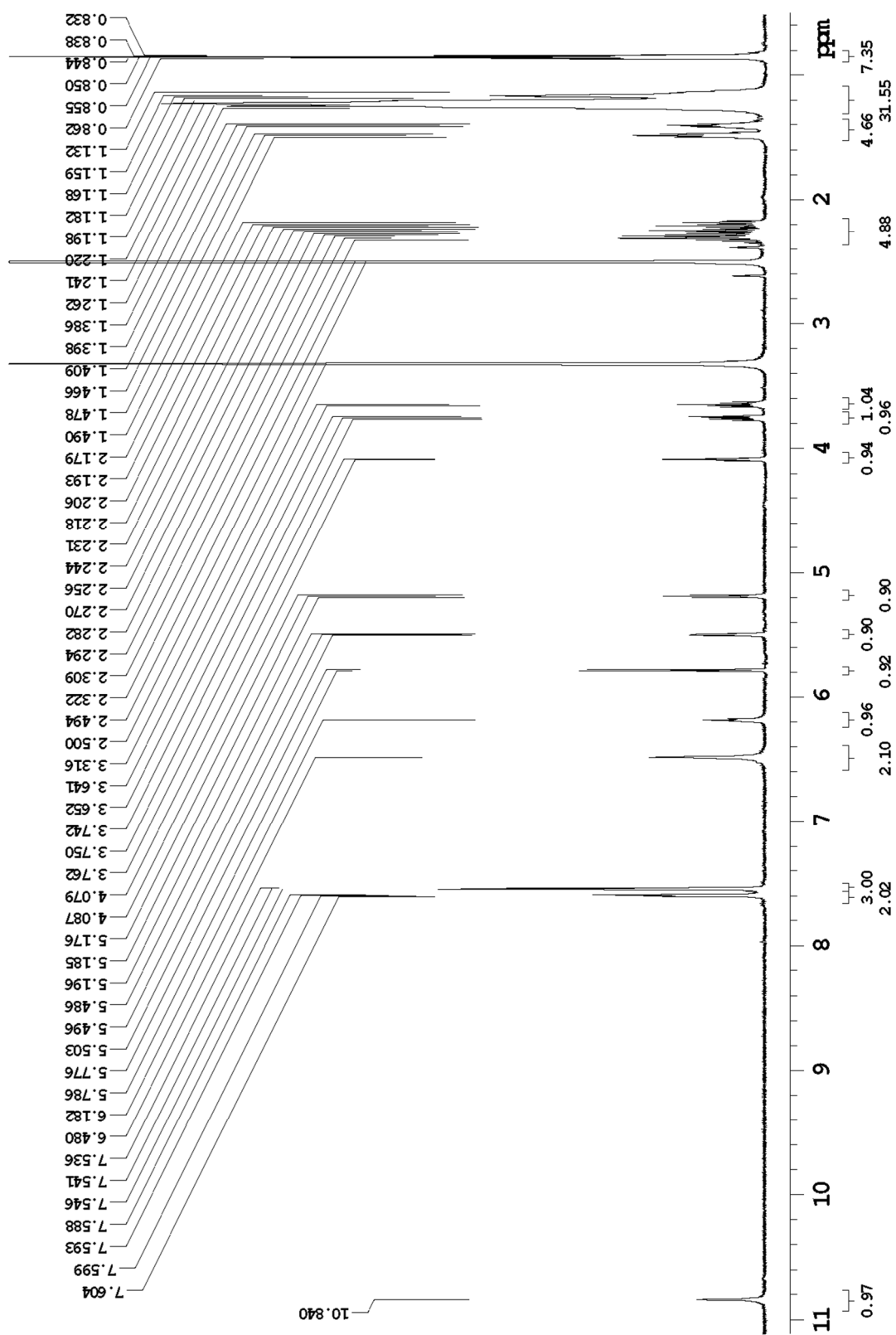
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Operator: sangiac

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2D Width 36199.1 Hz  
16 repetitions

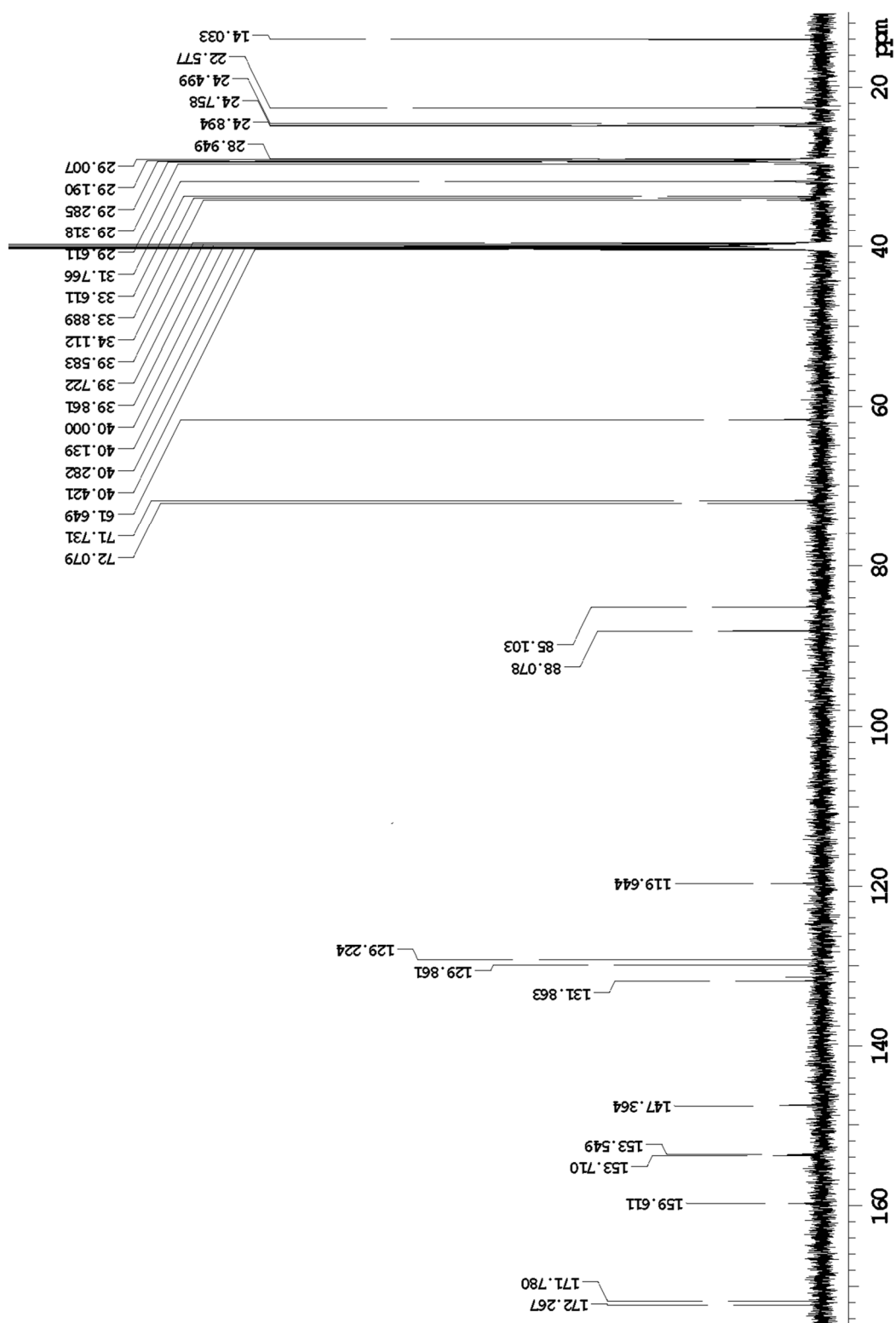
256 increments  
OBSERVE H1, 599.7287678 MHz

DATA PROCESSING  
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F1 DATA PROCESSING  
Sine ball 0.007 sec  
F1 size 4096 x 2048  
Total time 0 min 0 sec





<sup>1</sup>H-NMR (dms0-d<sub>6</sub>, 600 MHz) of **8Ph5OH**



$^{13}\text{C}\{^1\text{H}\}$  NMR (dmsol- $d_6$ , 600 MHz) of **8Ph5OH**

i600 std parameters

File:

Temp. 10.0 C / 283.1 K  
Operator: sangiac

Relax. delay 1.000 sec  
Acq. time 0.213 sec  
Width 9611.9 Hz  
2D Width 9611.9 Hz  
2 repetitions  
200 increments

OBSERVE HL, 599.7352686 MHz

DATA PROCESSING

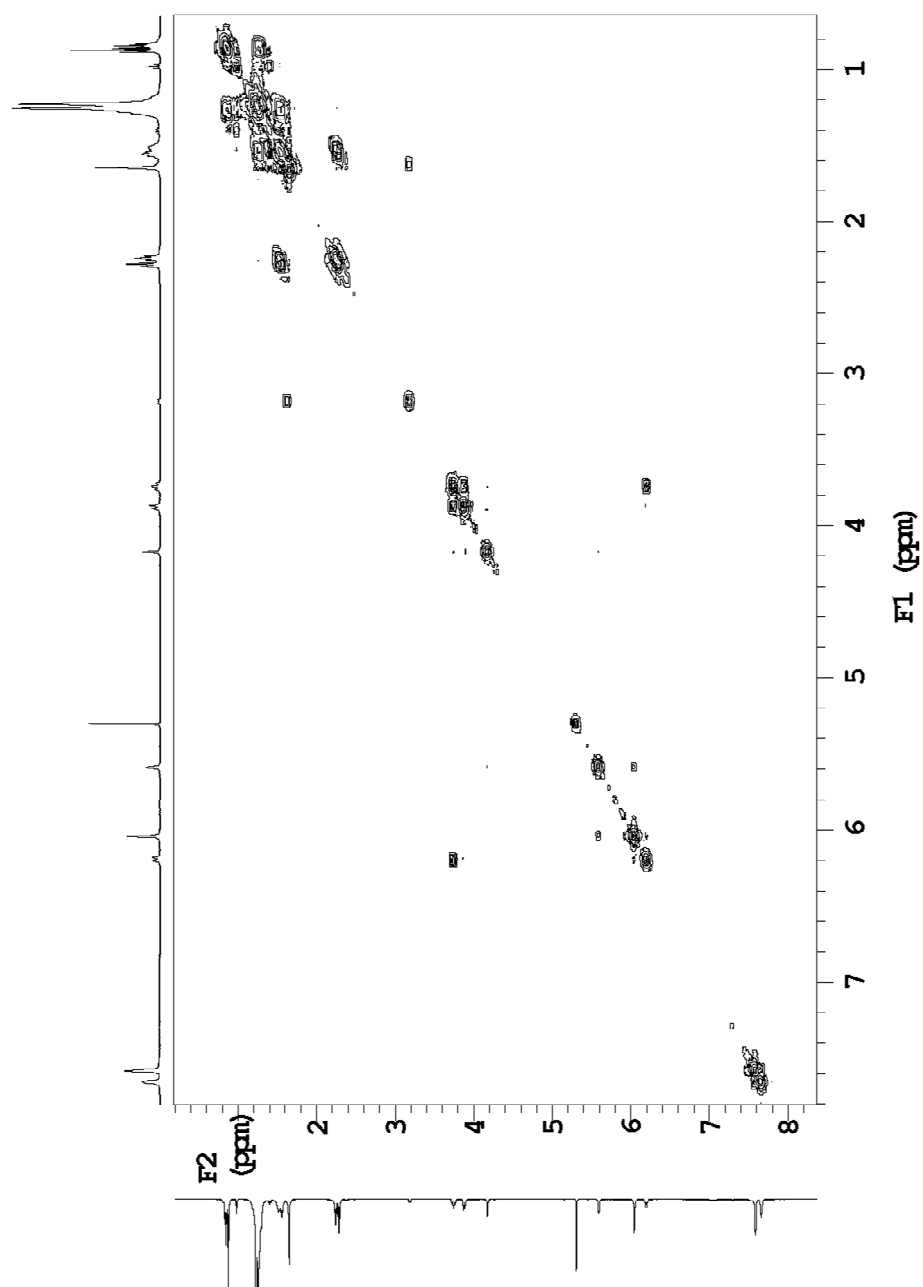
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Sine ball 0.021 sec

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Total time 0 min 0 sec



gCOSY spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Ph5OH**

i600 std parameters

File:

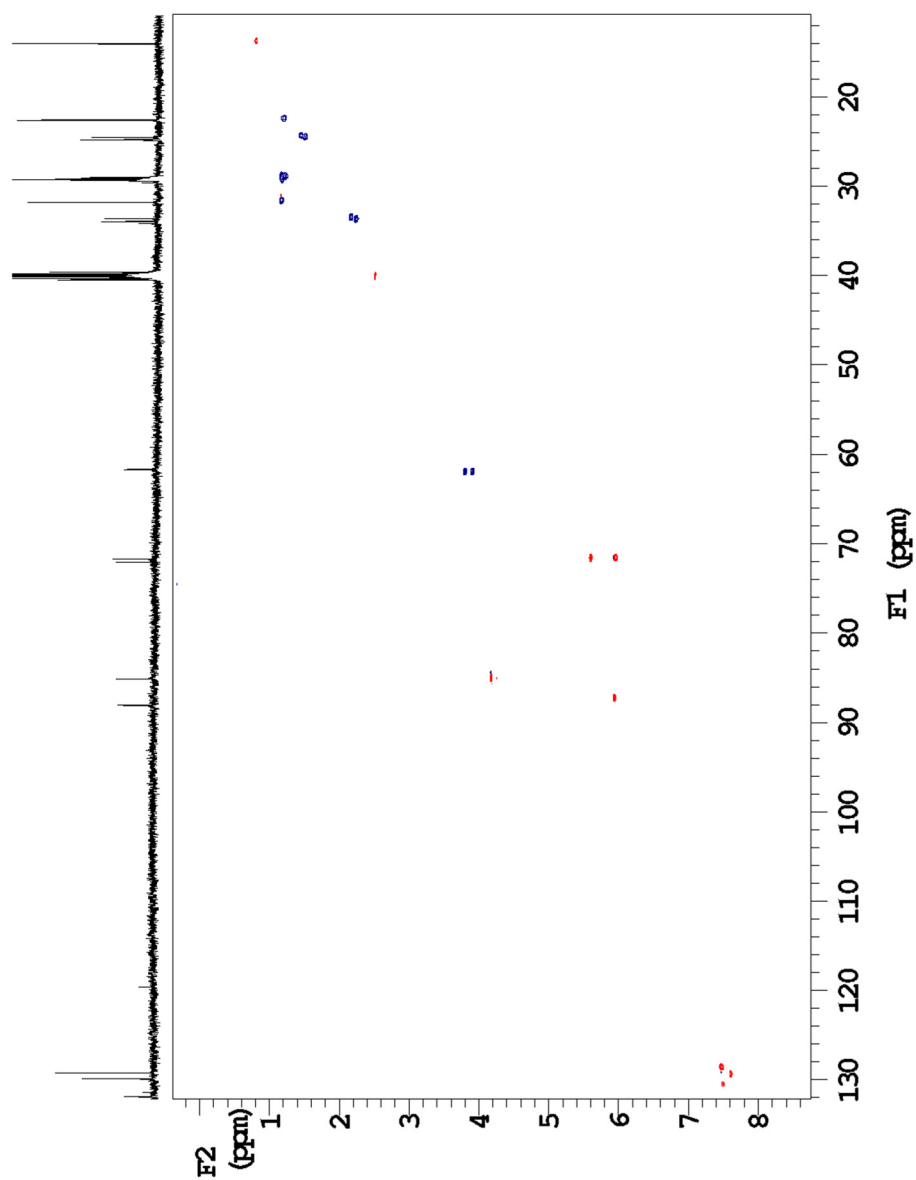
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Operator: sangiac

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2D Width 25632.8 Hz  
32 repetitions

2 x 256 increments  
OBSERVE HL, 599.7340972 MHz  
DECOUPLE CL3, 150.8145701 MHz

Power 45 dB  
on during acquisition  
off during delay  
W40 Triple modulated  
DATA PROCESSING

Gauss apodization 0.092 sec  
F1 DATA PROCESSING  
Gauss apodization 0.009 sec  
FT size 8192 x 2048  
Total time 0 min 0 sec



gHSQC spectrum (dmsso-d<sub>6</sub>, 600 MHz) of **8Ph5OH**

i600 std parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.200 sec  
Mixing 0.080 sec  
Acq. time 0.128 sec  
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2D Width 36199.1 Hz  
64 repetitions  
256 increments

CESERVE H1, 599.7340871 MHz

DATA PROCESSING

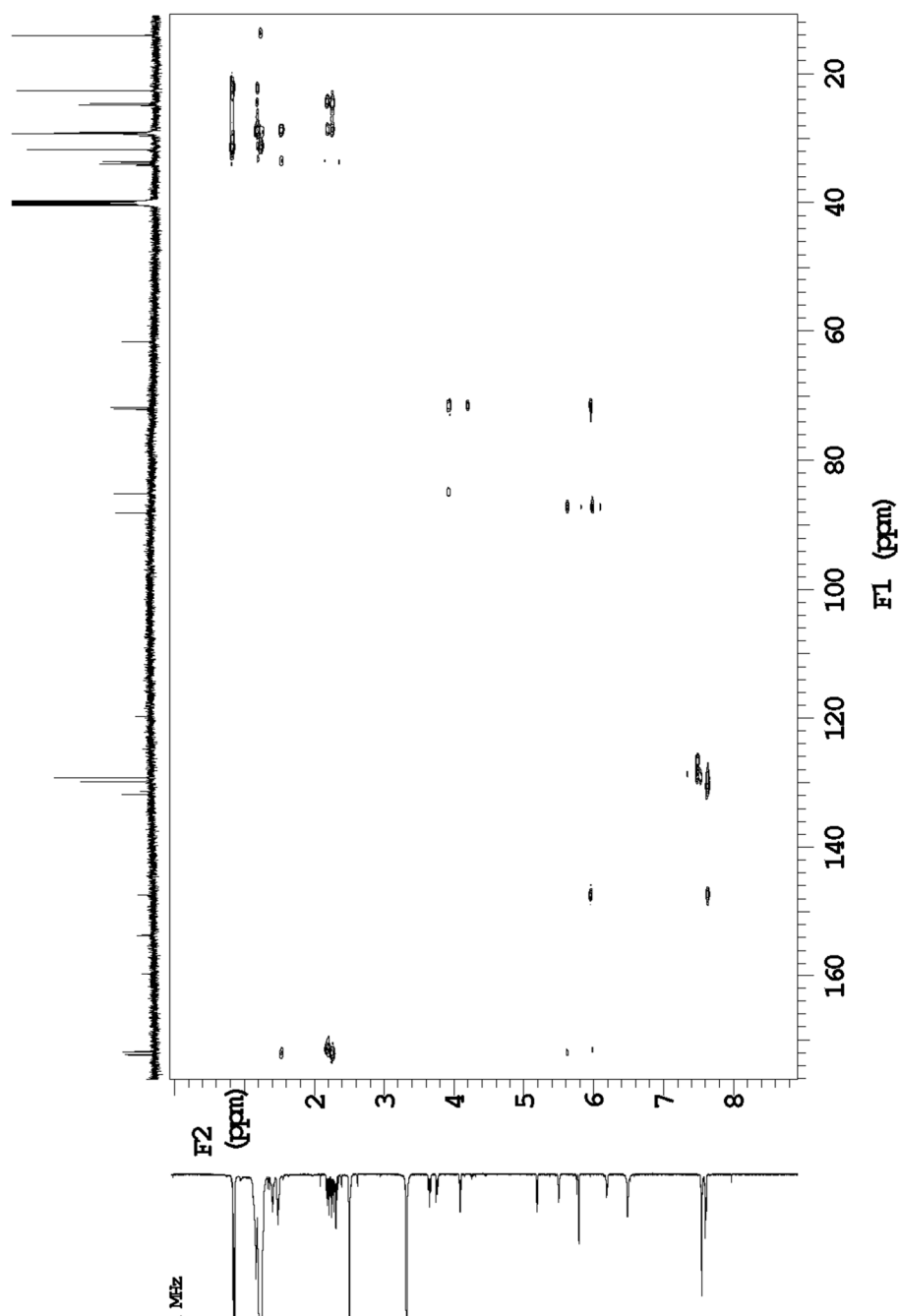
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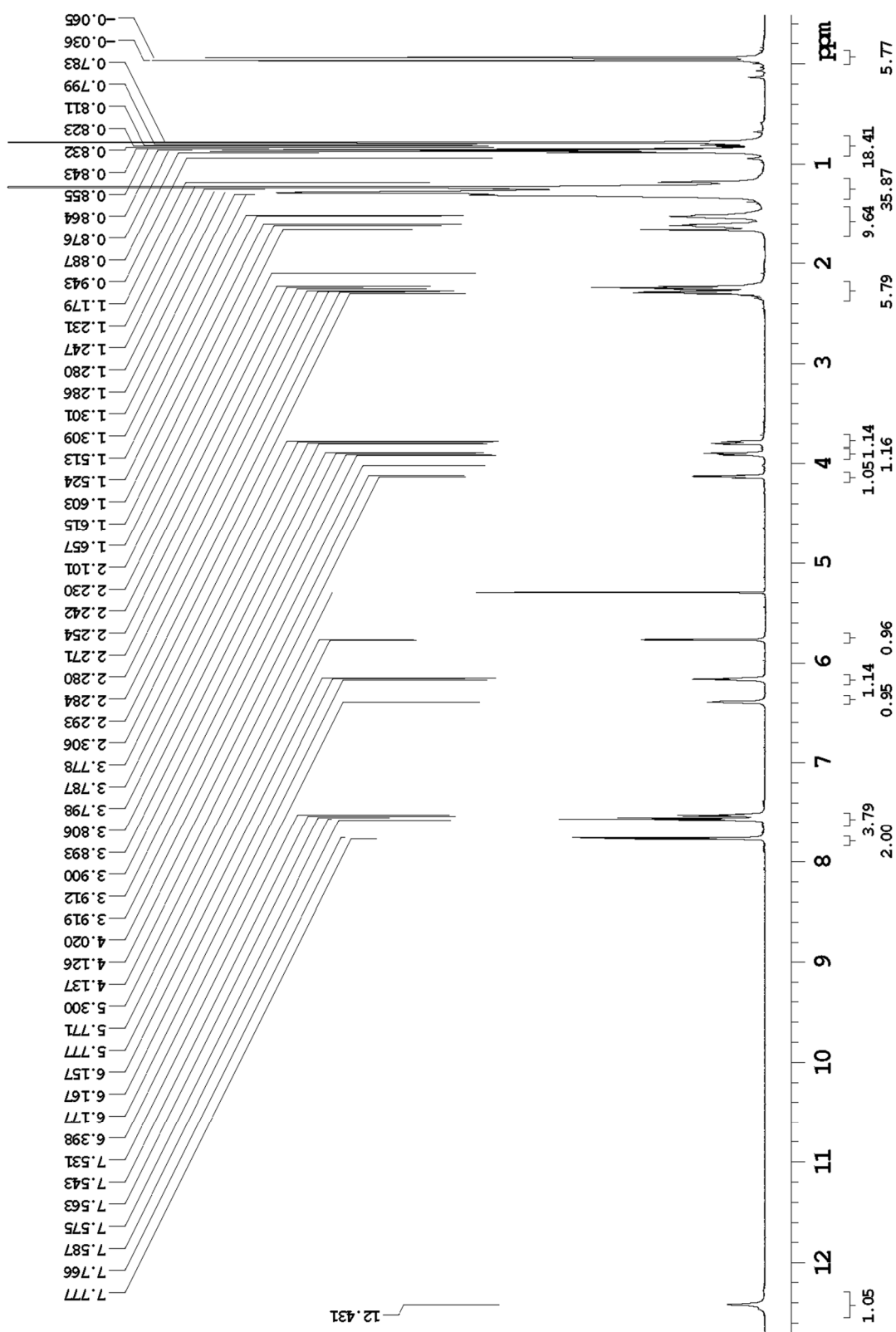
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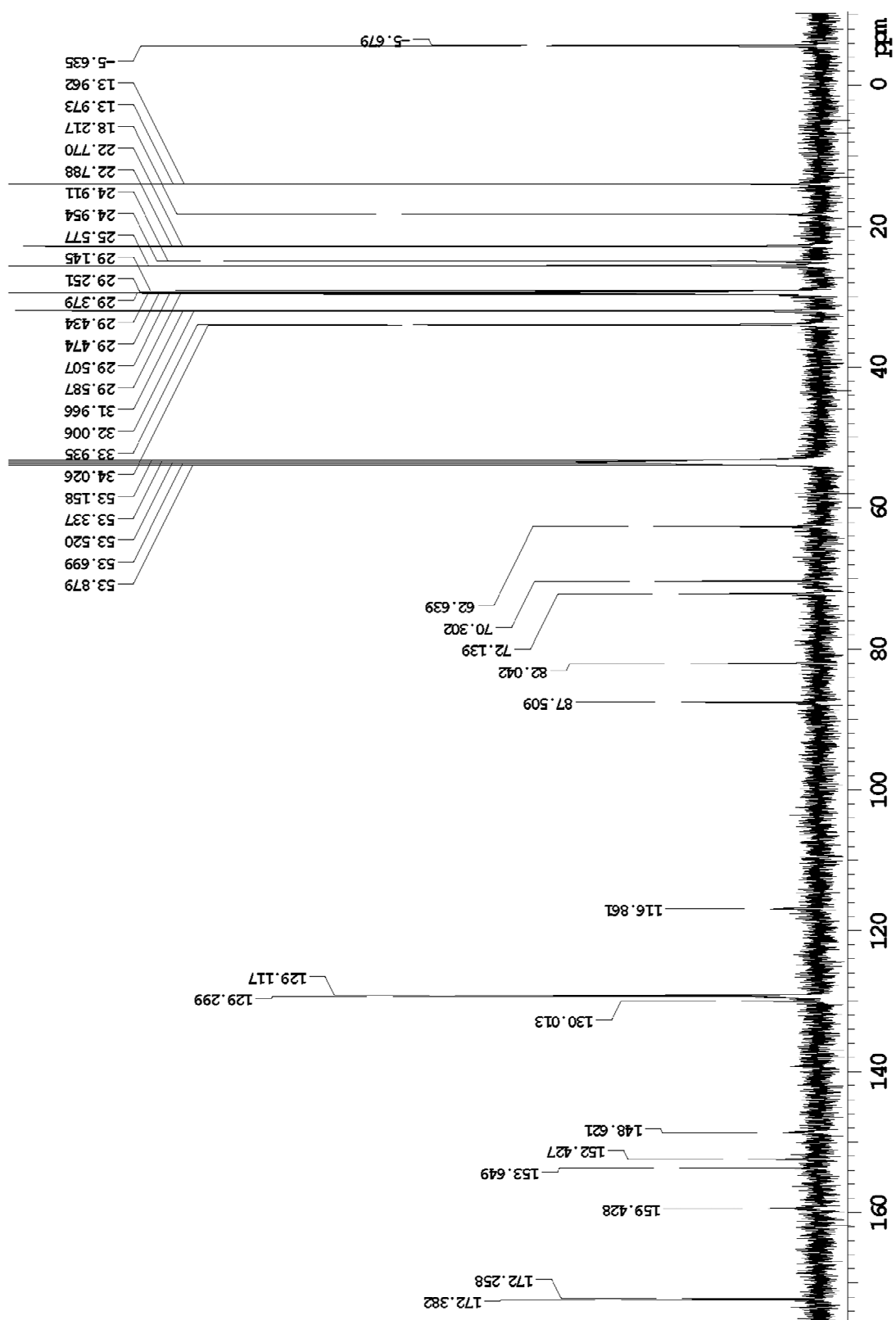
Total time 0 min 0 sec



gHMBC spectrum (dms0-d<sub>6</sub>, 600 MHz) of **8Ph5OH**



<sup>1</sup>H-NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Ph5Si**



$^{13}\text{C}\{^1\text{H}\}$  NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Ph5Si**



i600 std parameters

File:

Temp. 25.0 C / 298.1 K

Operator: sangiac

Relax. delay 1.000 sec

Mixing 0.080 sec

Acq. time 0.213 sec

Width 9611.9 Hz

2D Width 9611.9 Hz

2 repetitions

256 increments

OBSERVE H1, 599.7352616 MHz

DATA PROCESSING

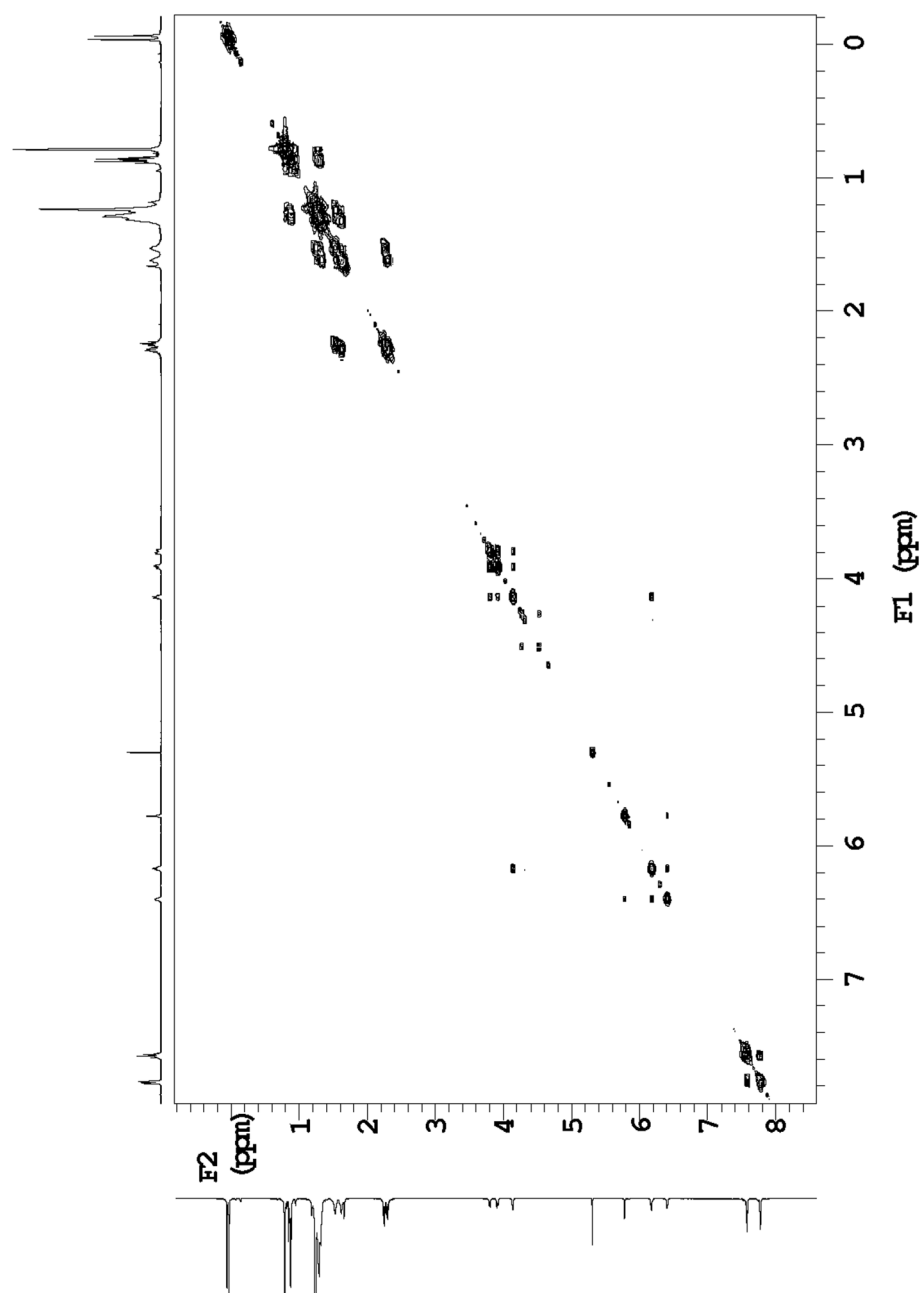
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F1 DATA PROCESSING

Sine bell 0.027 sec

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Total time 0 min 0 sec



gCOSY spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Ph5Si**

i600 std parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.000 sec  
Acq. time 0.199 sec  
Width 9611.9 Hz  
2D Width 25632.8 Hz  
4 repetitions

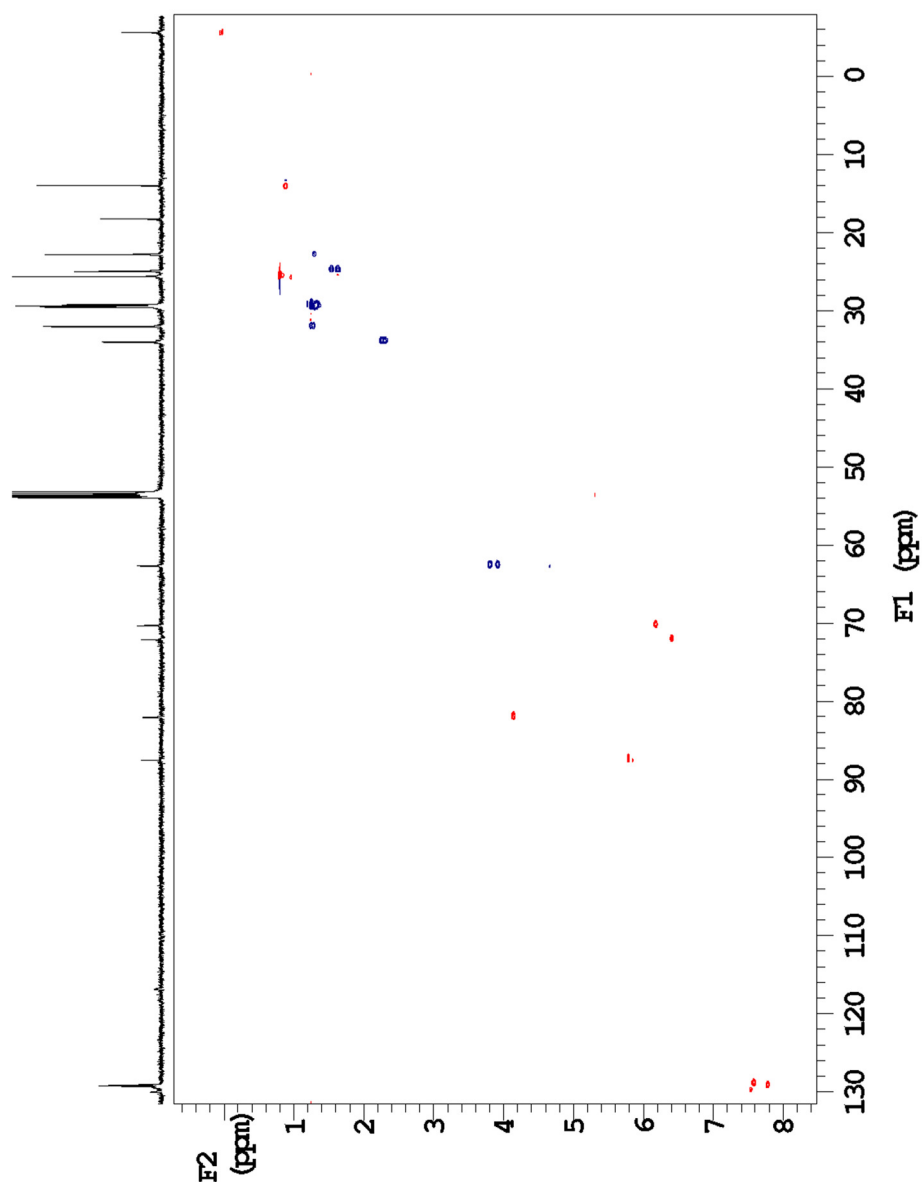
2 x 200 increments  
OBSERVE H1, 599.7352613 MHz  
DECOUPLE C13, 150.8148596 MHz

Power 46 dB  
on during acquisition  
off during delay  
W40 Triple modulated

DATA PROCESSING

Gauss apodization 0.092 sec  
F1 DATA PROCESSING

Gauss apodization 0.007 sec  
F1 size 8192 x 2048  
Total time 0 min 0 sec



gHSQC spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Ph5Si**

i600 std parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.000 sec  
Mixing 0.080 sec  
Acq. time 0.128 sec  
Width 9611.9 Hz  
2D Width 36199.1 Hz  
8 repetitions  
200 increments

OBSERVE HL, 599.7351982 MHz

DATA PROCESSING

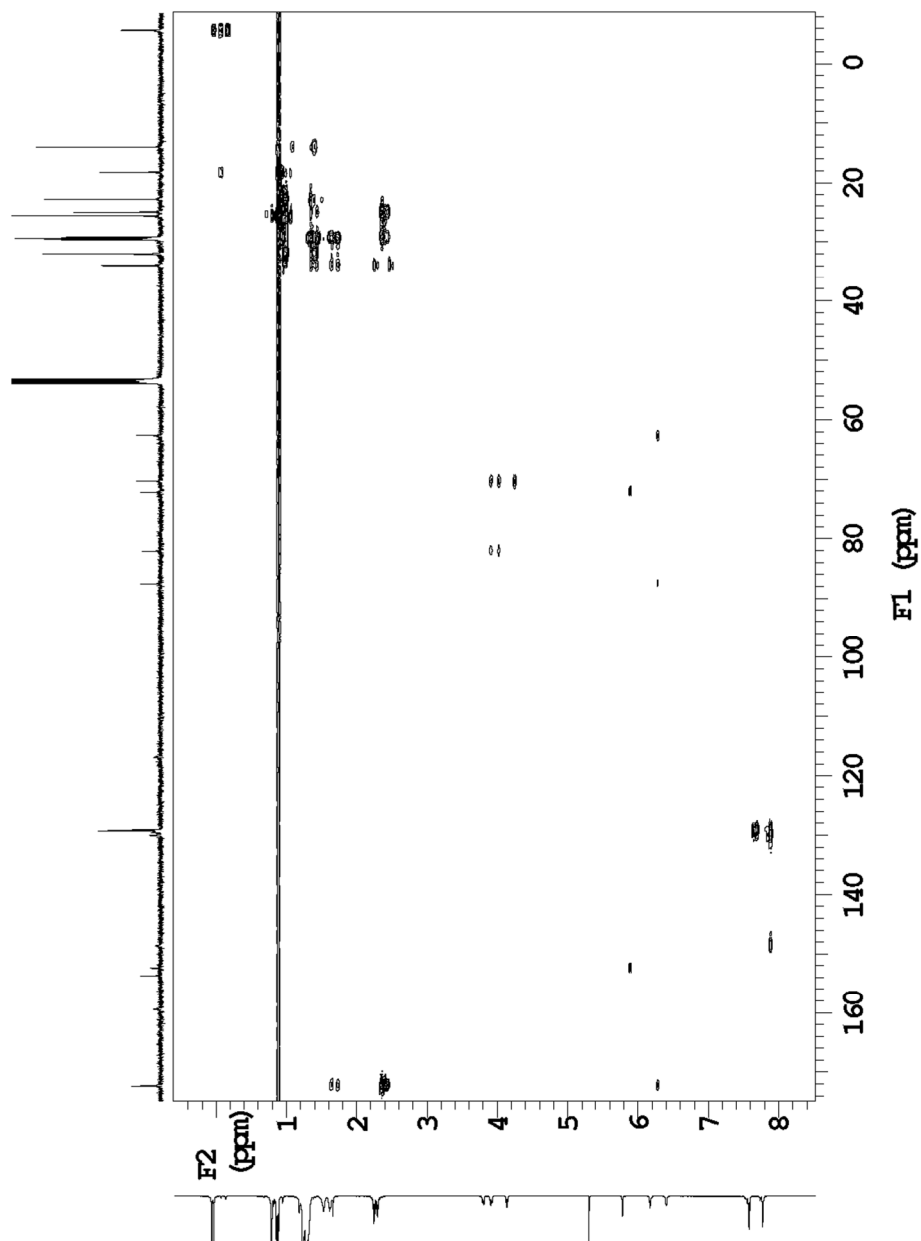
Sine ball 0.064 sec

F1 DATA PROCESSING

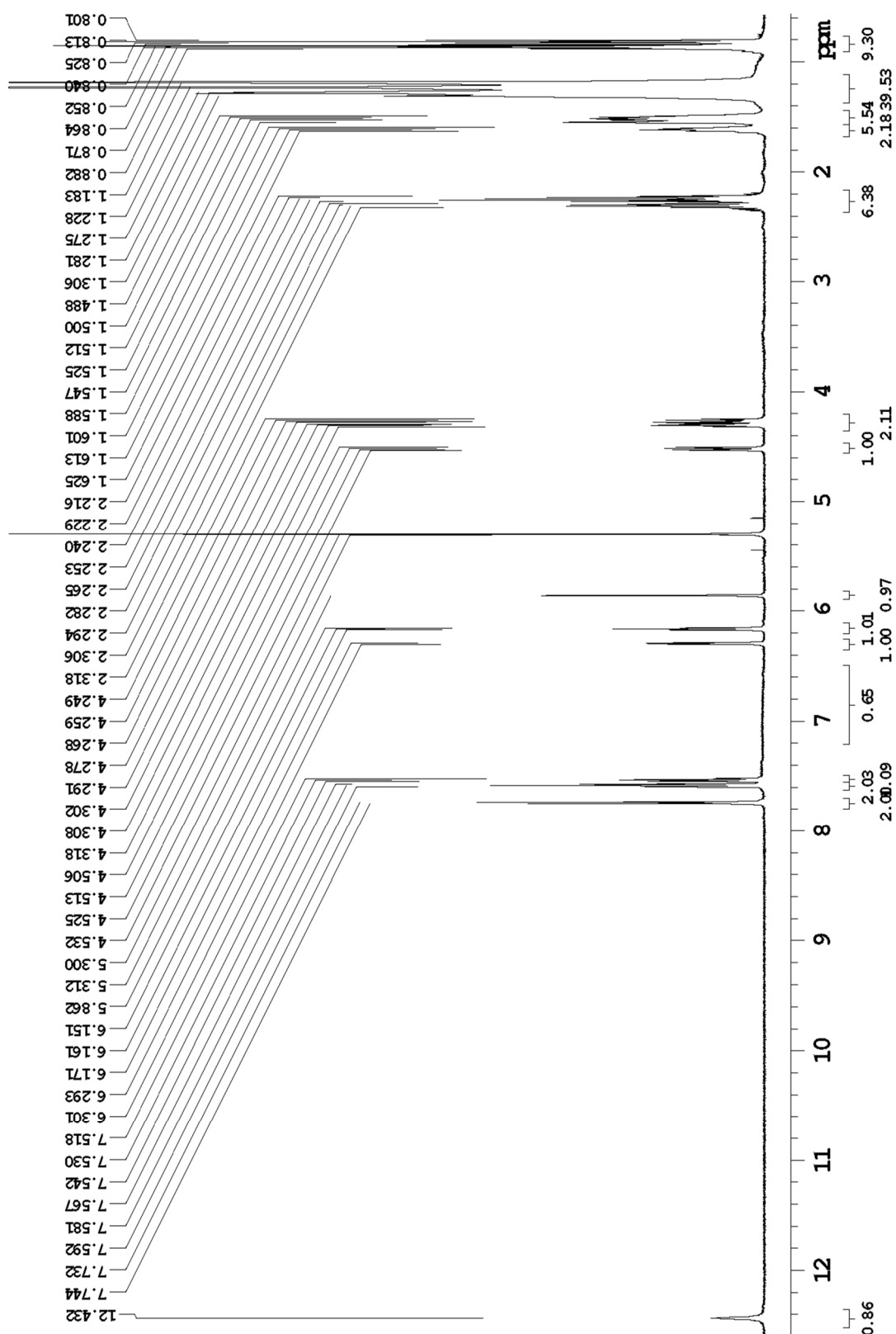
Sine ball 0.006 sec

FT size 4096 x 2048

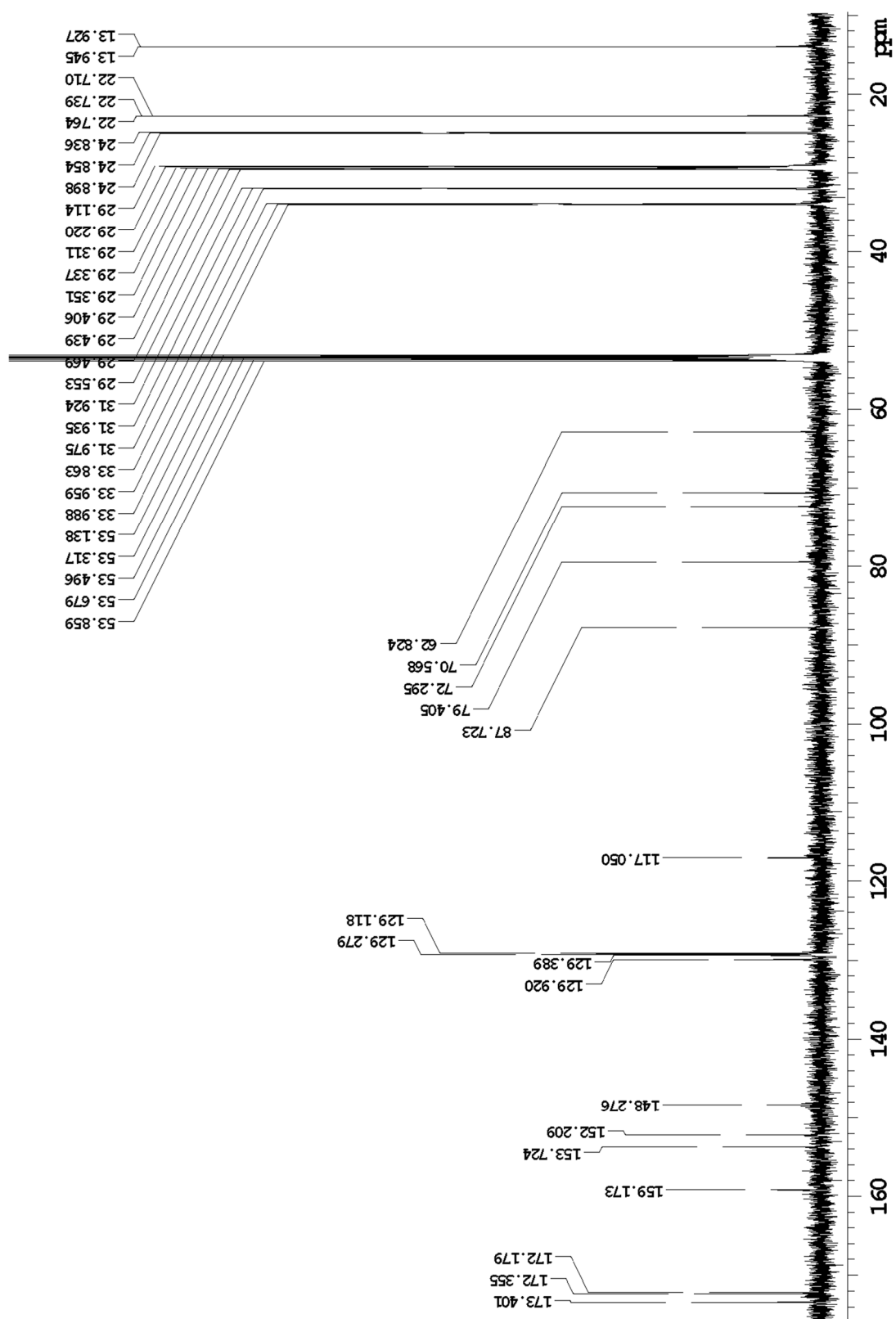
Total time 0 min 0 sec



gHMBC spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Ph5Si**



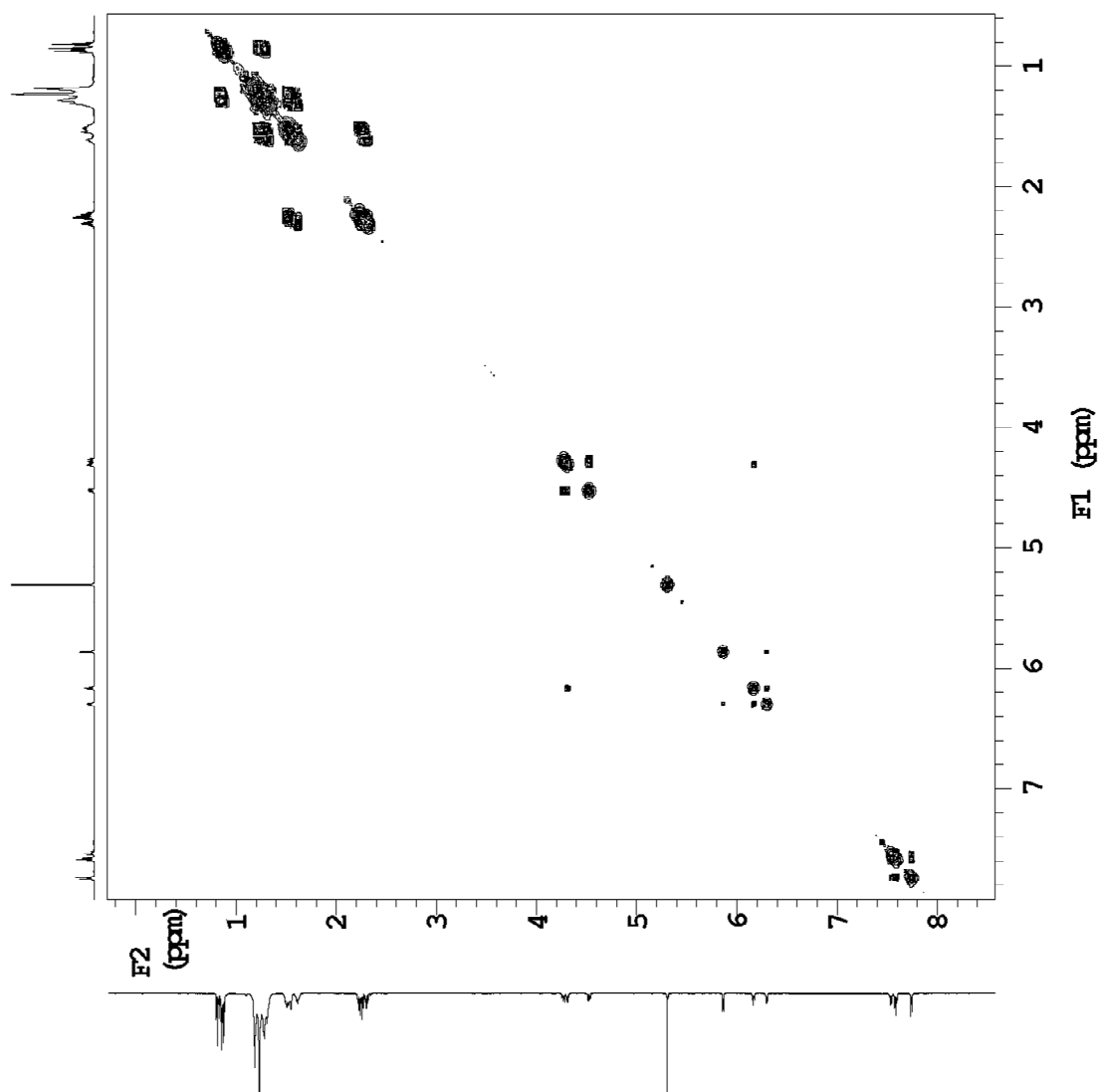
<sup>1</sup>H-NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Ph5C10**



<sup>13</sup>C{<sup>1</sup>H} NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Ph5C10**

STANDARD PROTON PARAMETERS

File:  
 Temp. 25.0 C / 298.1 K  
 Operator: sangiac  
 Relax. delay 1.000 sec  
 Acq. time 0.213 sec  
 Width 9611.9 Hz  
 2D Width 9611.9 Hz  
 2 repetitions  
 200 increments  
 OBSERVE F1, 599.7352635 MHz  
 DATA PROCESSING  
 Sine bell 0.107 sec  
 F1 DATA PROCESSING  
 Sine bell 0.021 sec  
 FT size 4096 x 4096  
 Total time 0 min 0 sec



gCOSY spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Ph5C10**

# STANDARD PROTON PARAMETERS

File:

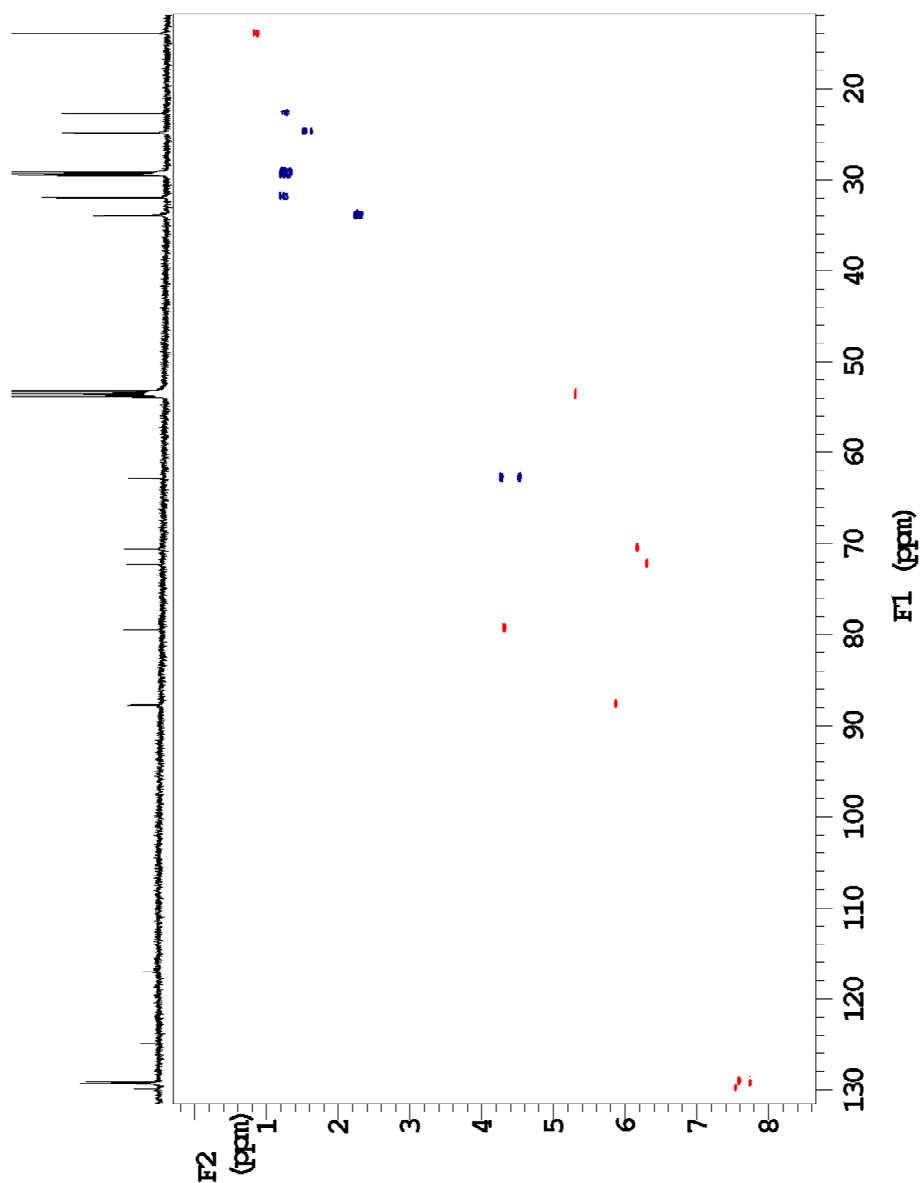
Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.000 sec  
Acq. time 0.199 sec  
Width 9611.9 Hz  
2D Width 25632.8 Hz  
4 repetitions  
2 x 200 increments

CESRVE HL, 599.7352625 MHz  
DECOUPLE C13, 150.8148596 MHz

Power 45 dB  
on during acquisition  
off during delay  
W40 Triple modulated

DATA PROCESSING  
Gauss apodization 0.092 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
Ft size 8192 x 2048  
Total time 0 min 0 sec



gHSQC spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Ph5C10**

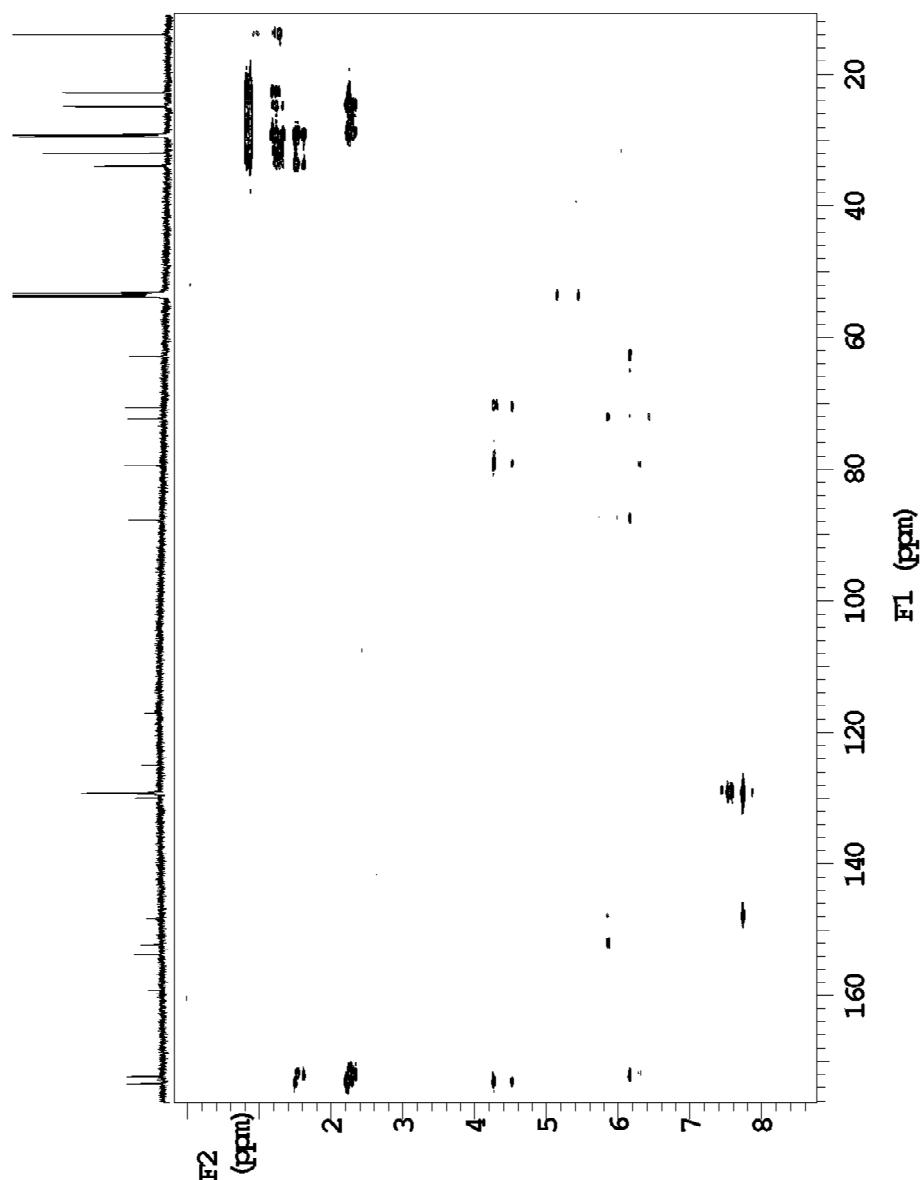
# STANDARD PROTON PARAMETERS

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

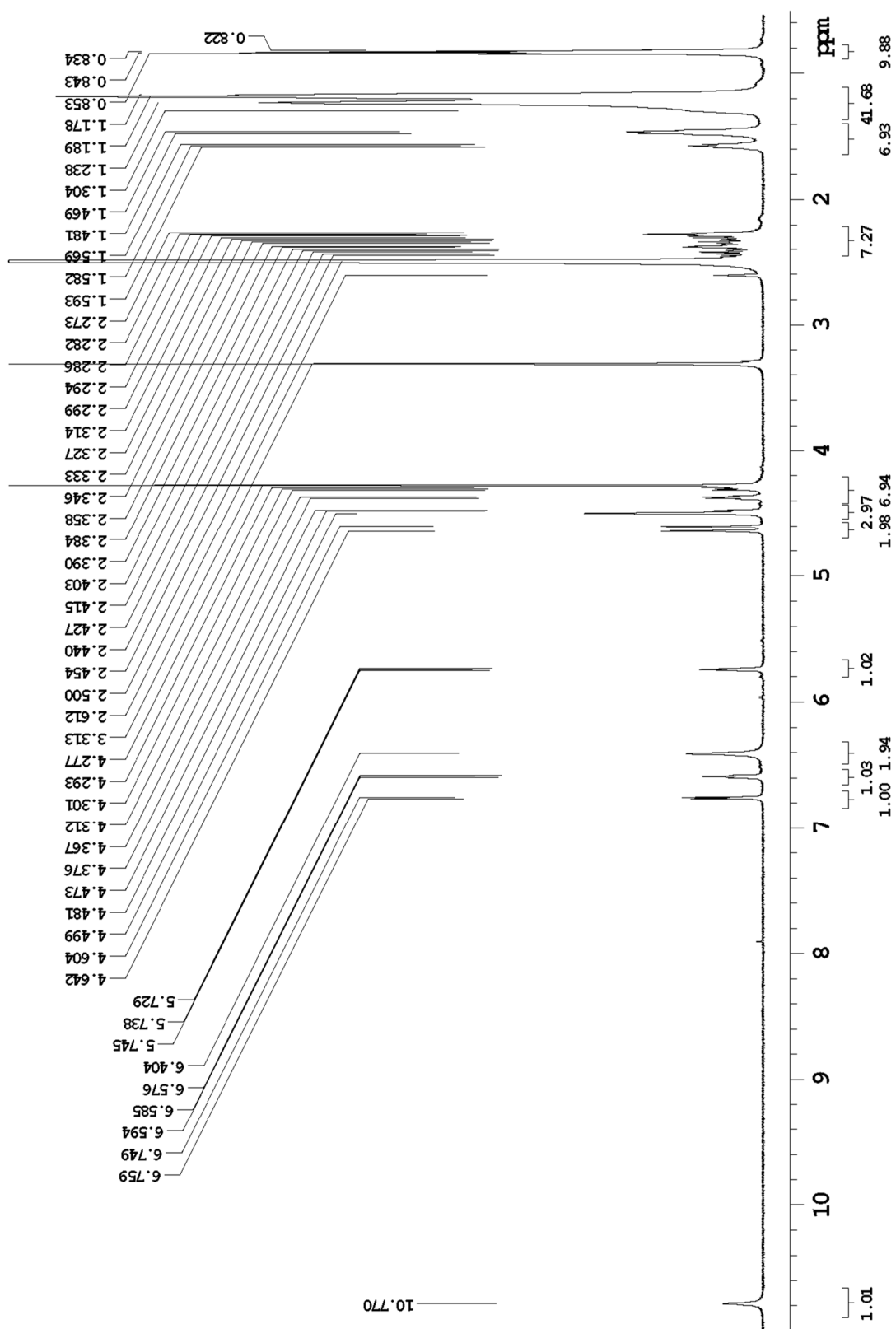
Relax. delay 1.000 sec  
Mixing 0.080 sec  
Acq. time 0.128 sec  
Width 9611.9 Hz  
2D Width 36199.1 Hz  
16 repetitions  
256 increments

CESRVE HL, 599.7352679 MHz  
DATA PROCESSING  
Sine bell 0.064 sec  
F1 DATA PROCESSING  
Sine bell 0.007 sec  
F1 size 4096 x 2048  
Total time 0 min 0 sec

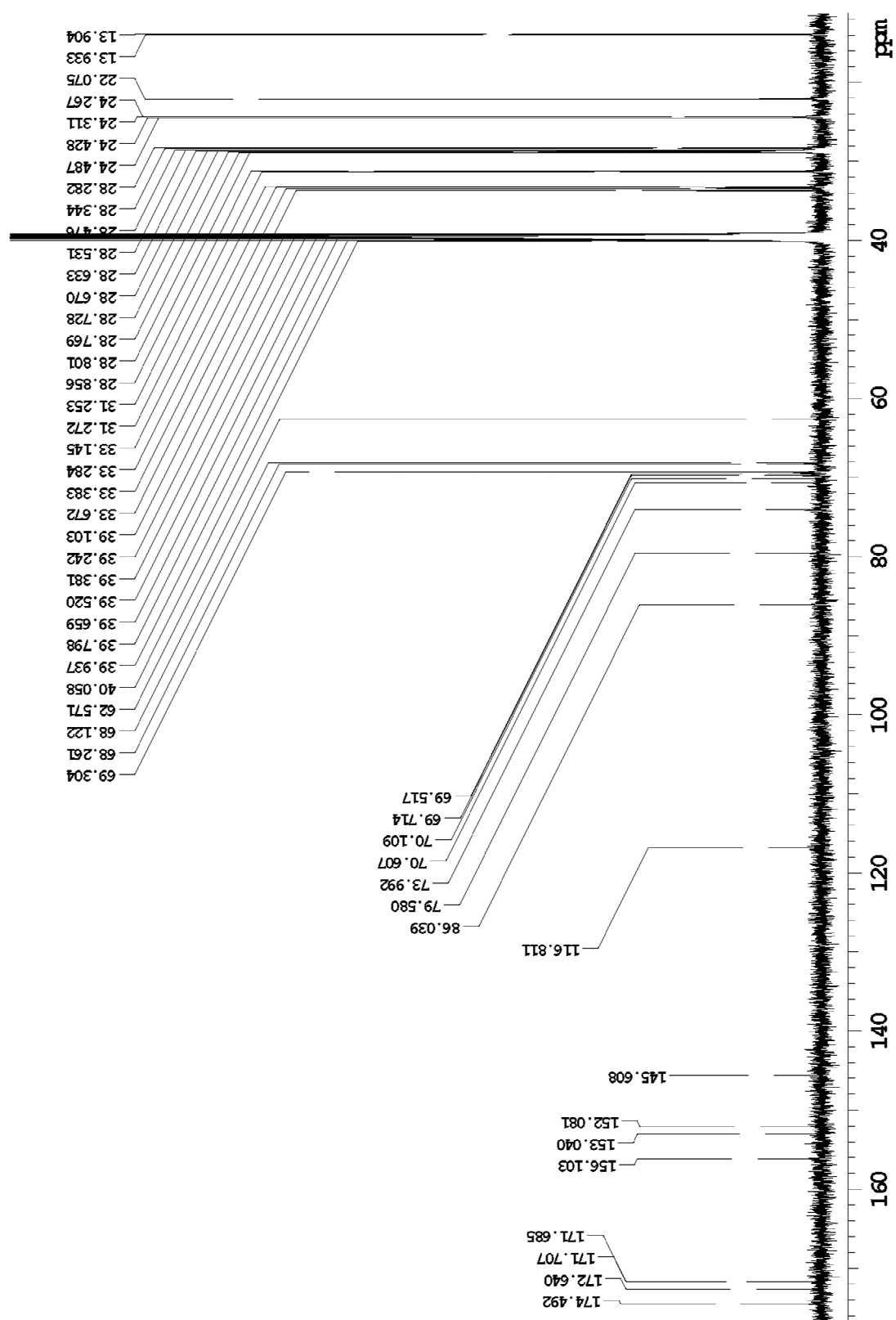


gHMBC spectrum (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Ph5C10**





<sup>1</sup>H-NMR (dms0-d<sub>6</sub>, 600 MHz) of **8Fc5C10**



$^{13}\text{C}\{^1\text{H}\}$  NMR (dms0-d<sub>6</sub>, 600 MHz) of **8Fc5C10**

1600 std parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.000 sec  
Acq. time 0.213 sec  
Width 9611.9 Hz  
2D Width 9611.9 Hz  
4 repetitions  
256 increments

OBSERVE H1, 599.7304254 MHz

DATA PROCESSING

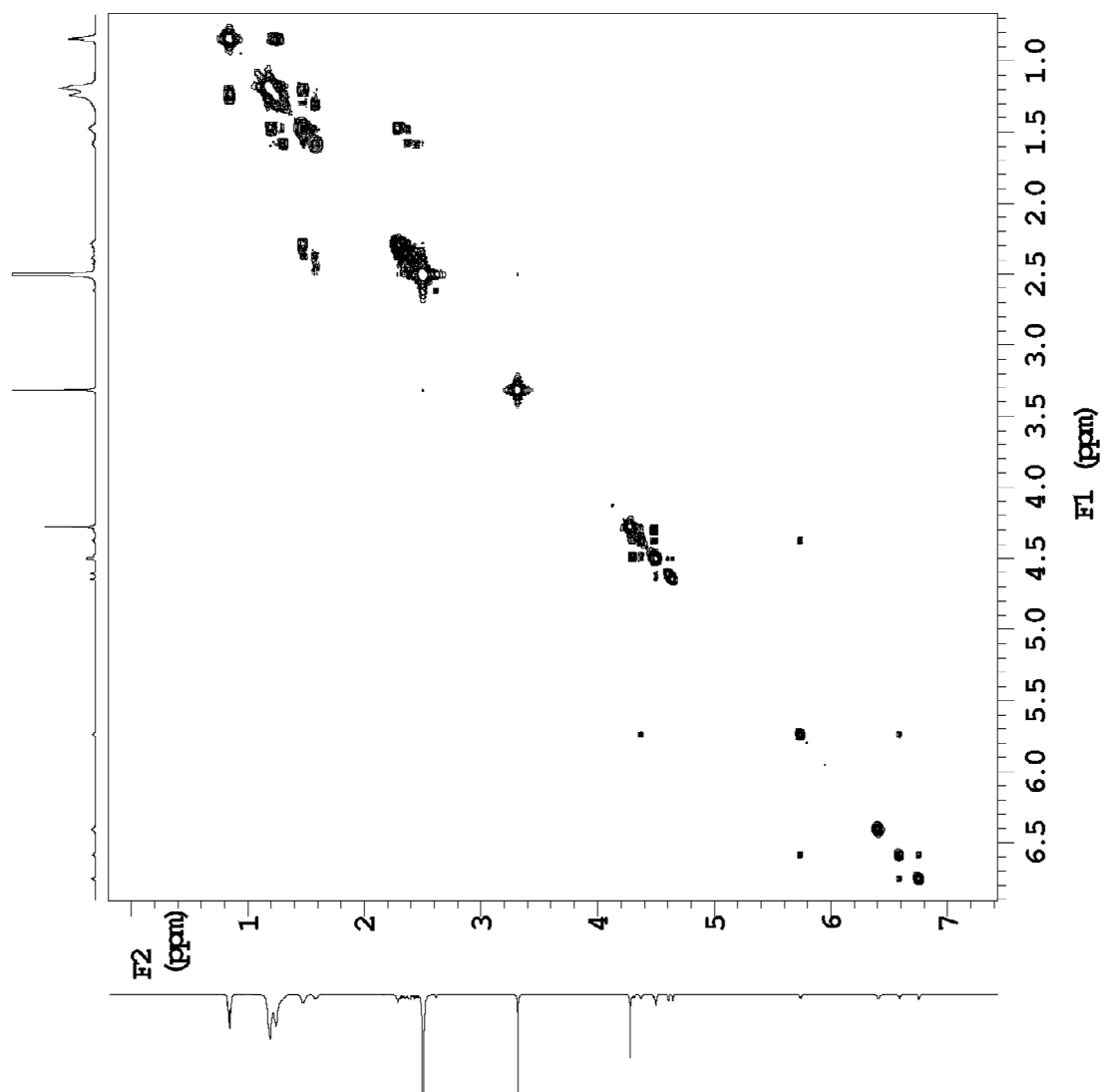
Sine ball 0.107 sec

F1 DATA PROCESSING

Sine ball 0.027 sec

FT size 4096 x 4096

Total time 0 min 0 sec



gCOSY spectrum (dms0-d<sub>6</sub>, 600 MHz) of **8Fe5C10**

i600 std parameters

File:

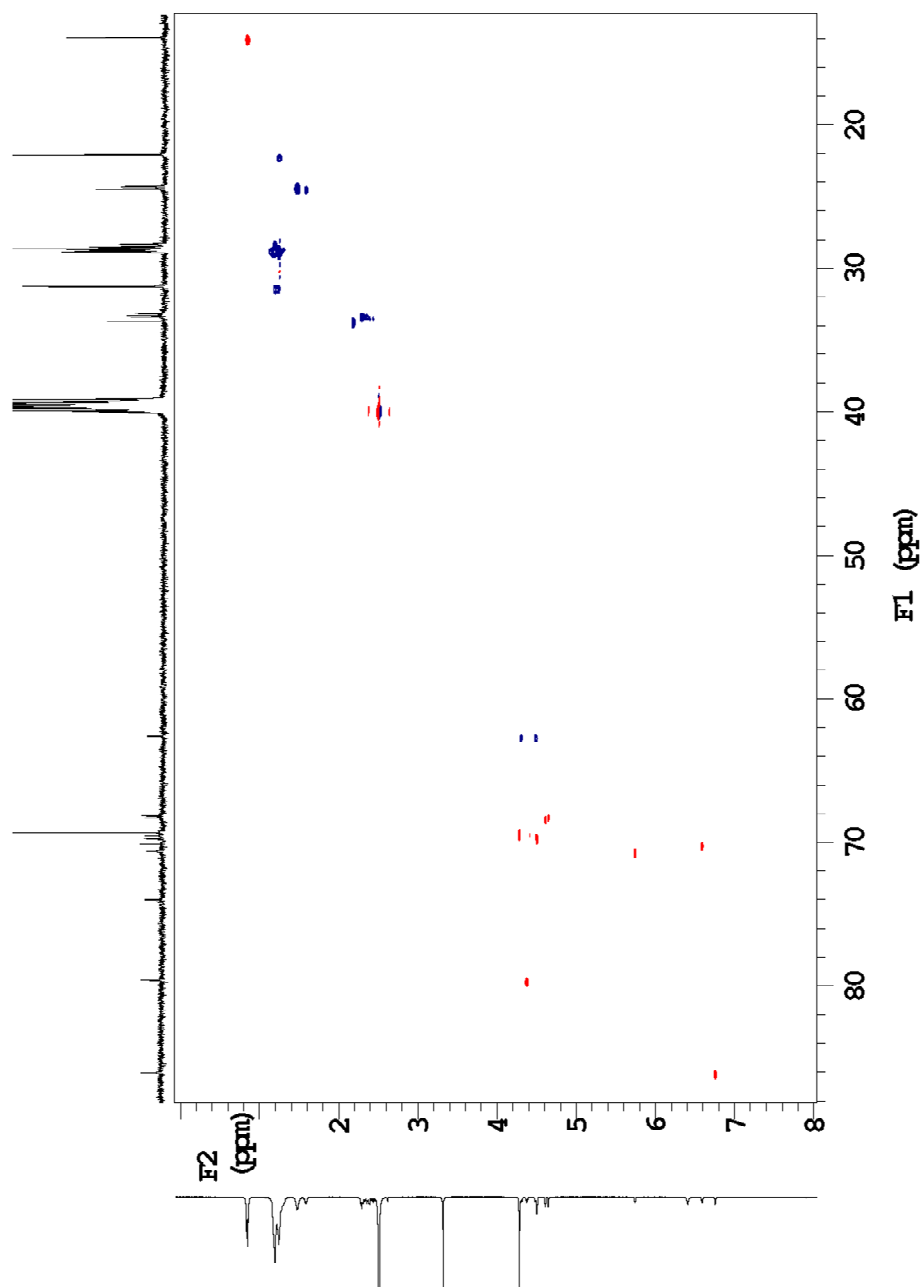
Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.000 sec  
Acq. time 0.199 sec  
Width 9611.9 Hz  
2D Width 25632.8 Hz  
8 repetitions

2 x 256 increments  
OBSERVE H1, 599.7304200 MHz  
DECOUPLE C13, 150.8136483 MHz  
Power 43 dB

on during acquisition  
off during delay  
W40 Triple modulated  
DATA PROCESSING

Gauss apodization 0.092 sec  
F1 DATA PROCESSING  
Gauss apodization 0.009 sec  
F1 size 8192 x 2048  
Total time 0 min 0 sec



gHSQC spectrum (dms0-d<sub>6</sub>, 600 MHz) of 8Fc5C10

i600 std parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.000 sec  
Mixing 0.080 sec  
Acq. time 0.128 sec  
Width 9611.9 Hz  
2D Width 36199.1 Hz  
32 repetitions  
256 increments

OBSERVE F1, 599.7304284 MHz

DATA PROCESSING

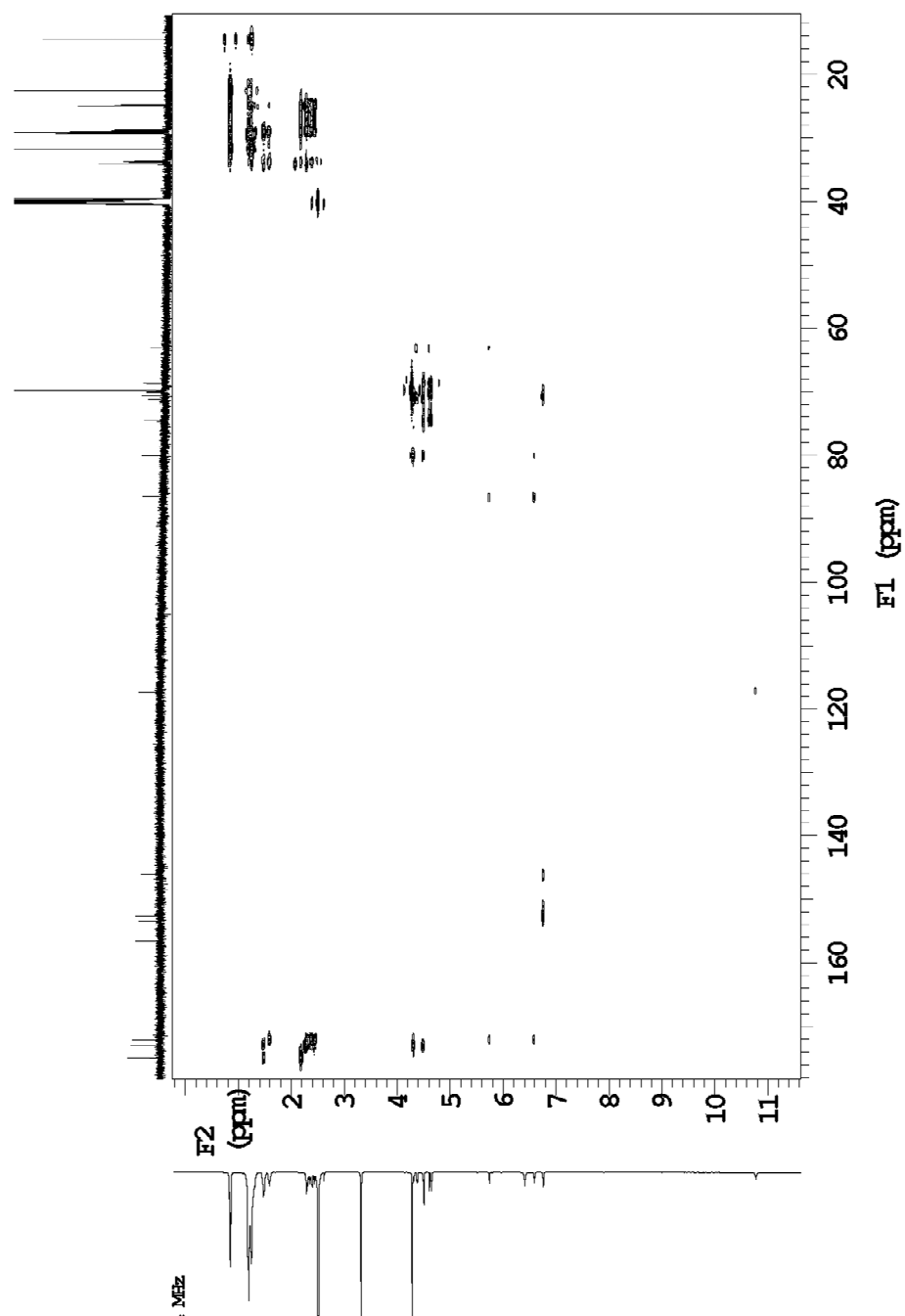
Sine bell 0.064 sec

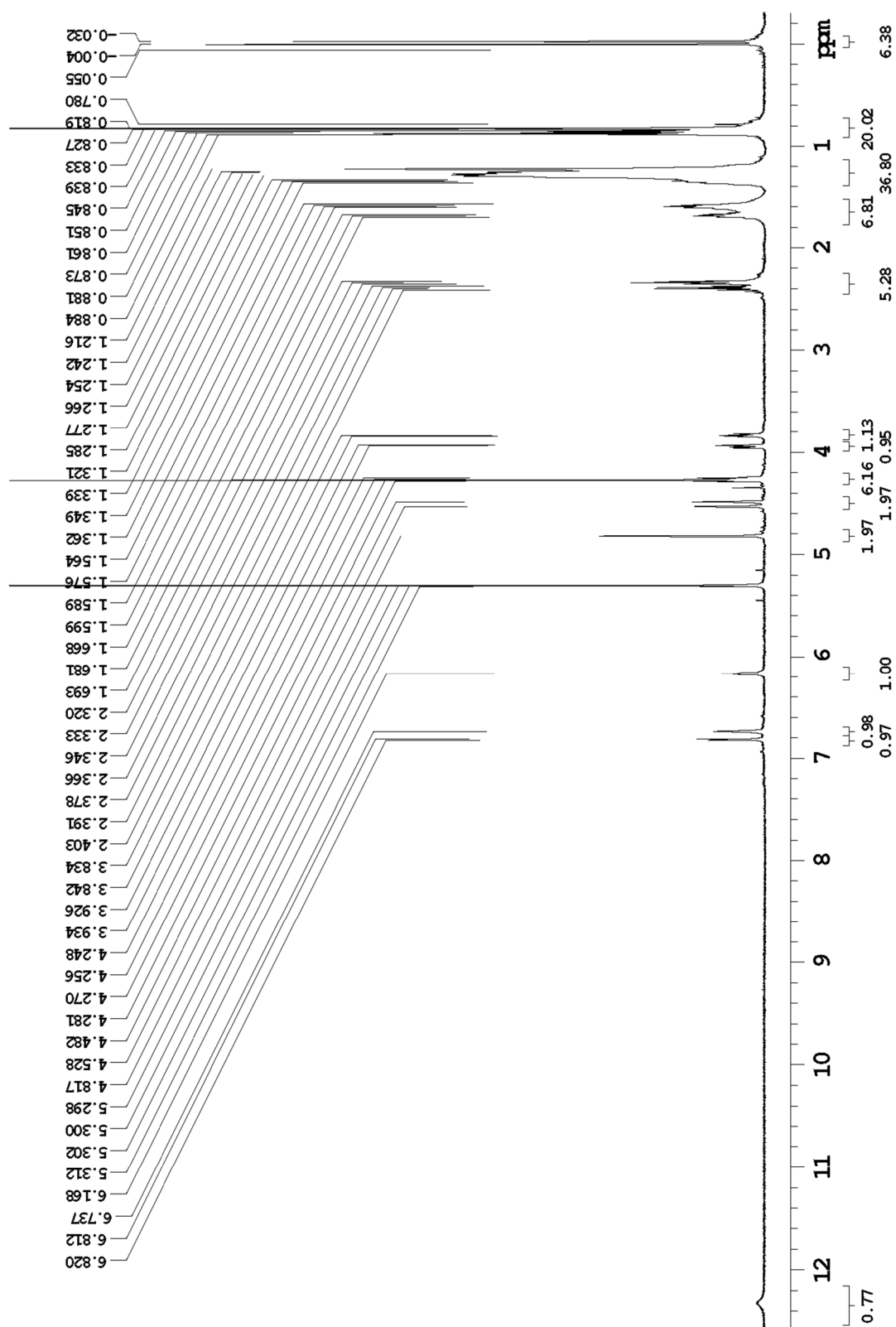
F1 DATA PROCESSING

Sine bell 0.007 sec

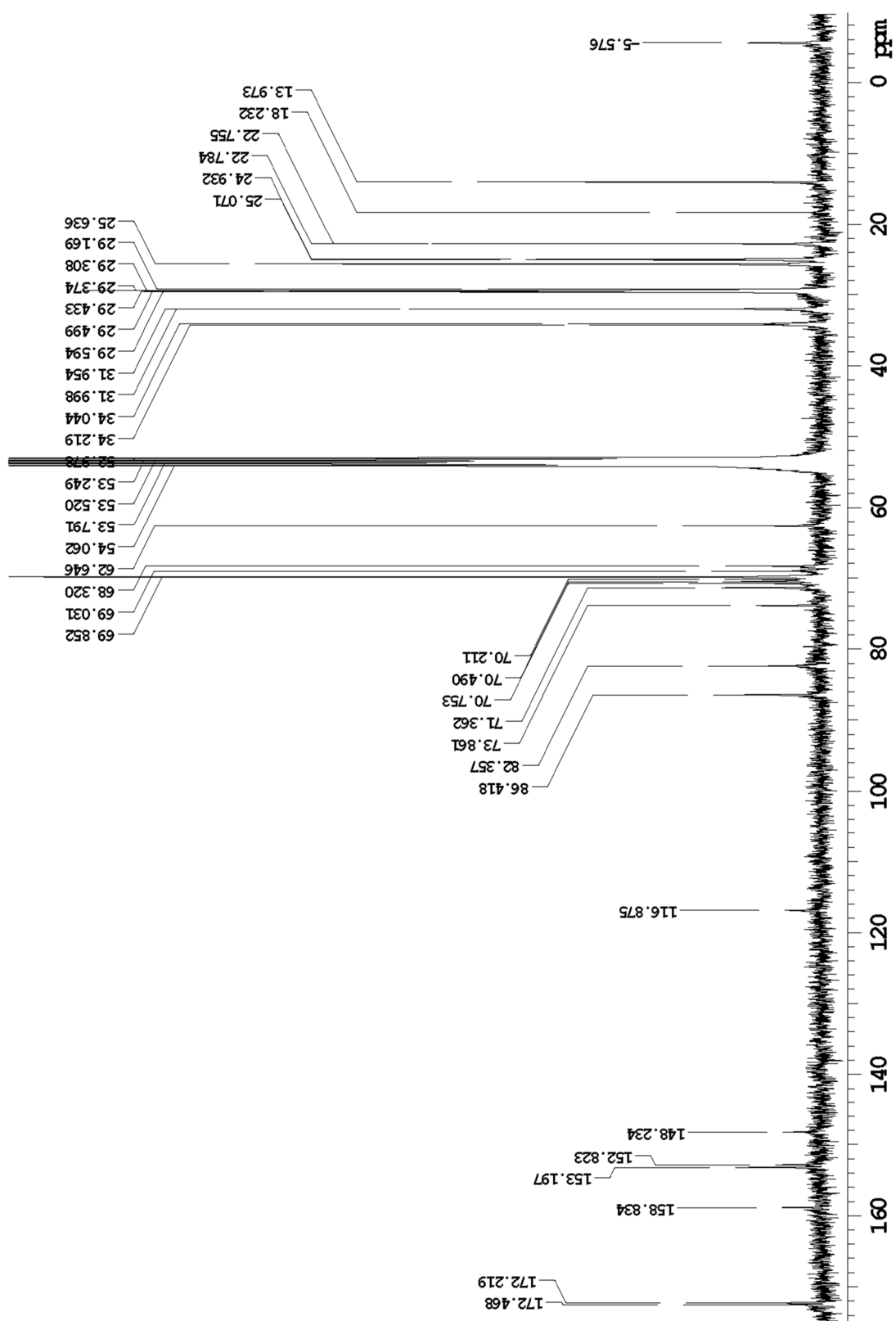
FT size 4096 x 2048

Total time 0 min 0 sec





<sup>1</sup>H-NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Fc5Si**



$^{13}\text{C}\{^1\text{H}\}$  NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Fc5Si**

Std proton

File:

Temp. 25.0 C / 298.1 K

Operator: sangiac

Relax. delay 1.000 sec

Acq. time 0.213 sec

Width 9611.9 Hz

2D Width 9611.9 Hz

2 repetitions

200 increments

OBSERVE F1, 599.7352614 MHz

DATA PROCESSING

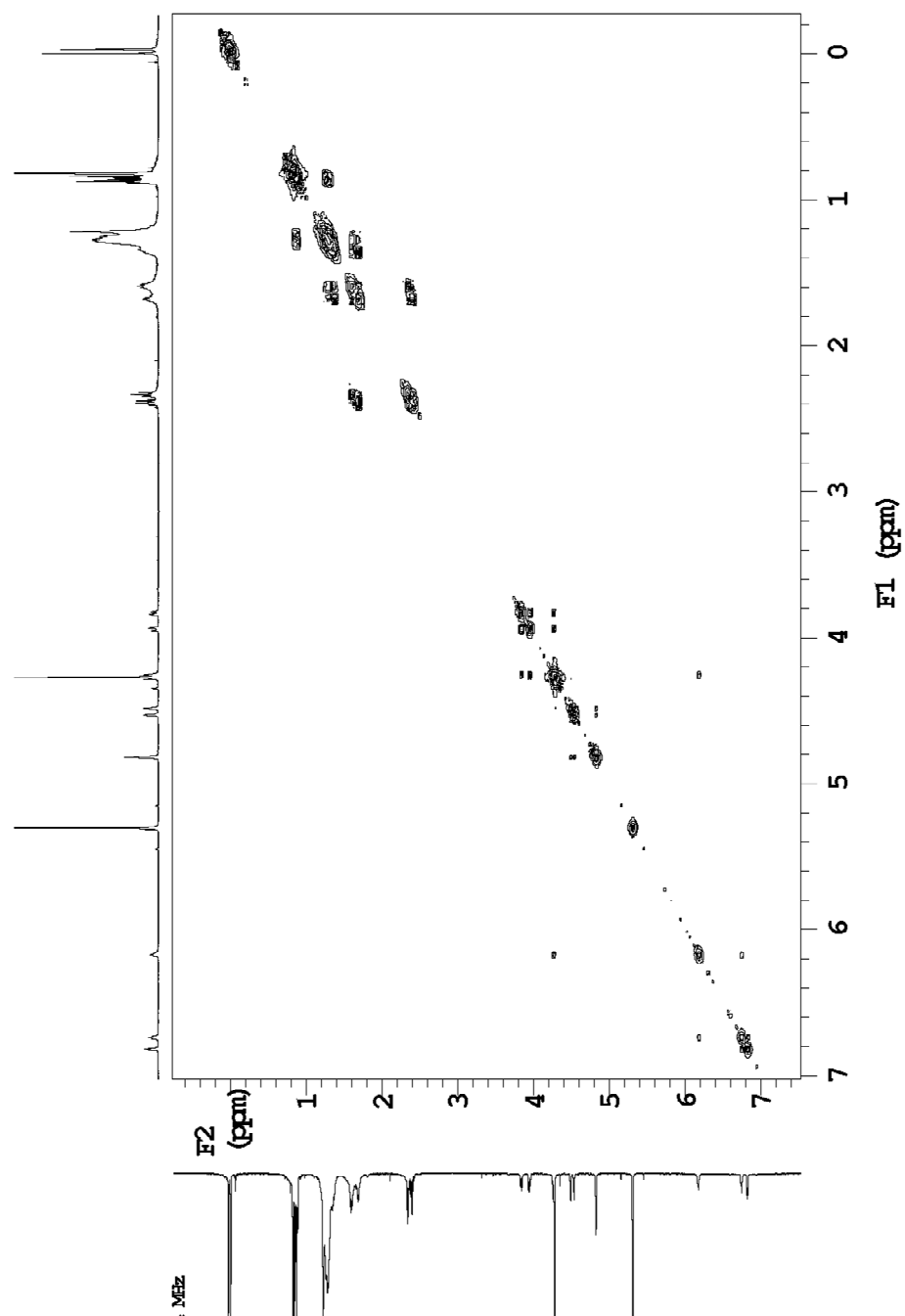
Sine bell 0.107 sec

F1 DATA PROCESSING

Sine bell 0.021 sec

FT size 4096 x 4096

Total time 0 min 0 sec



gCOSY spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Fc5Si**



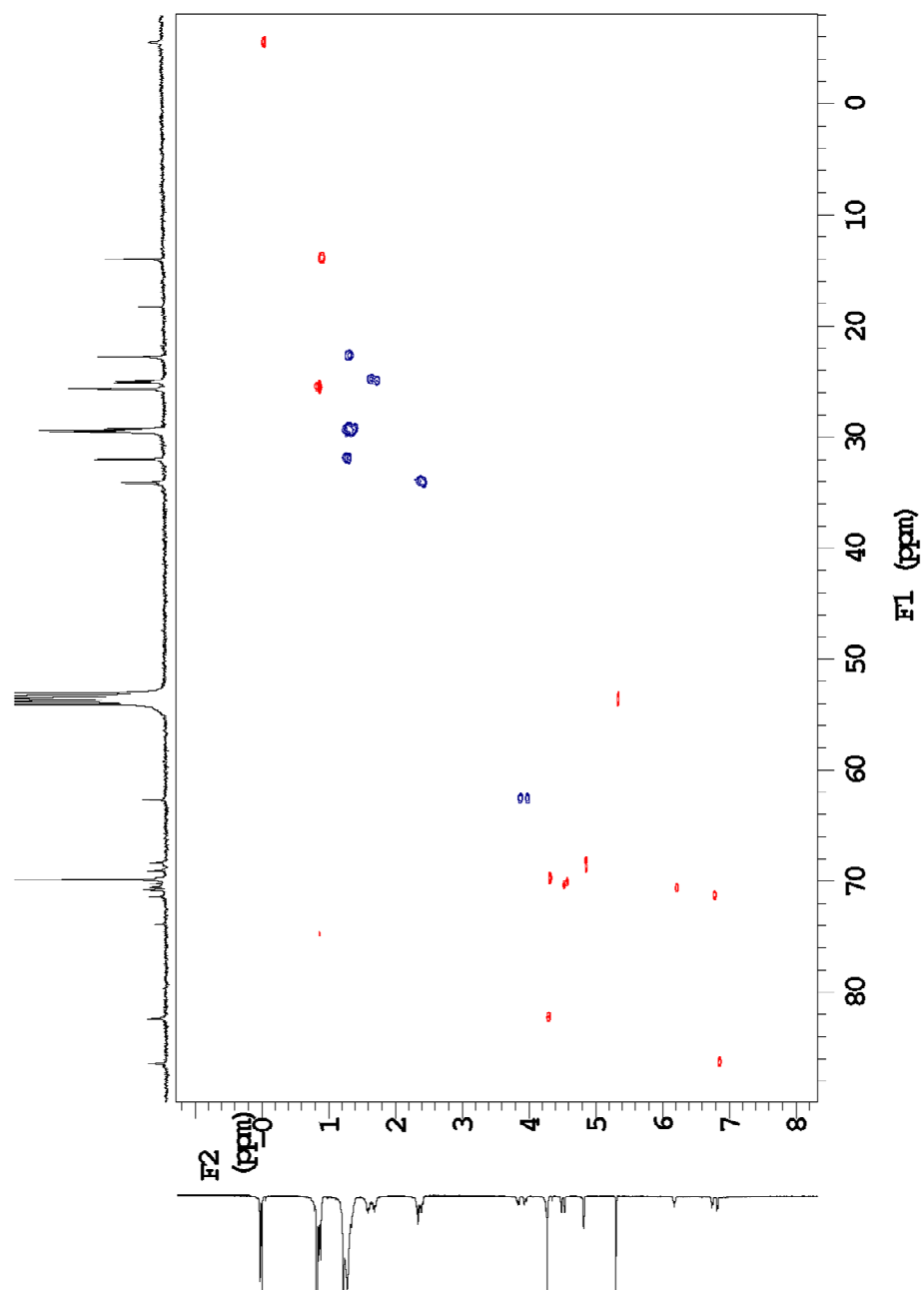
Std Proton parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sandonat  
INOVA-600 "F4EL"

Relax. delay 1.301 sec  
Acq. time 0.199 sec  
Width 6398.0 Hz  
2D Width 17094.0 Hz  
8 repetitions  
2 x 200 increments

OBSERVE H1, 399.9253434 MHz  
DECOUPLE C13, 100.5688497 MHz  
Power 46 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.092 sec  
F1 DATA PROCESSING  
Gauss apodization 0.011 sec  
FT size 4096 x 2048  
Total time 0 min 0 sec



gHSQC spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Fc5Si**

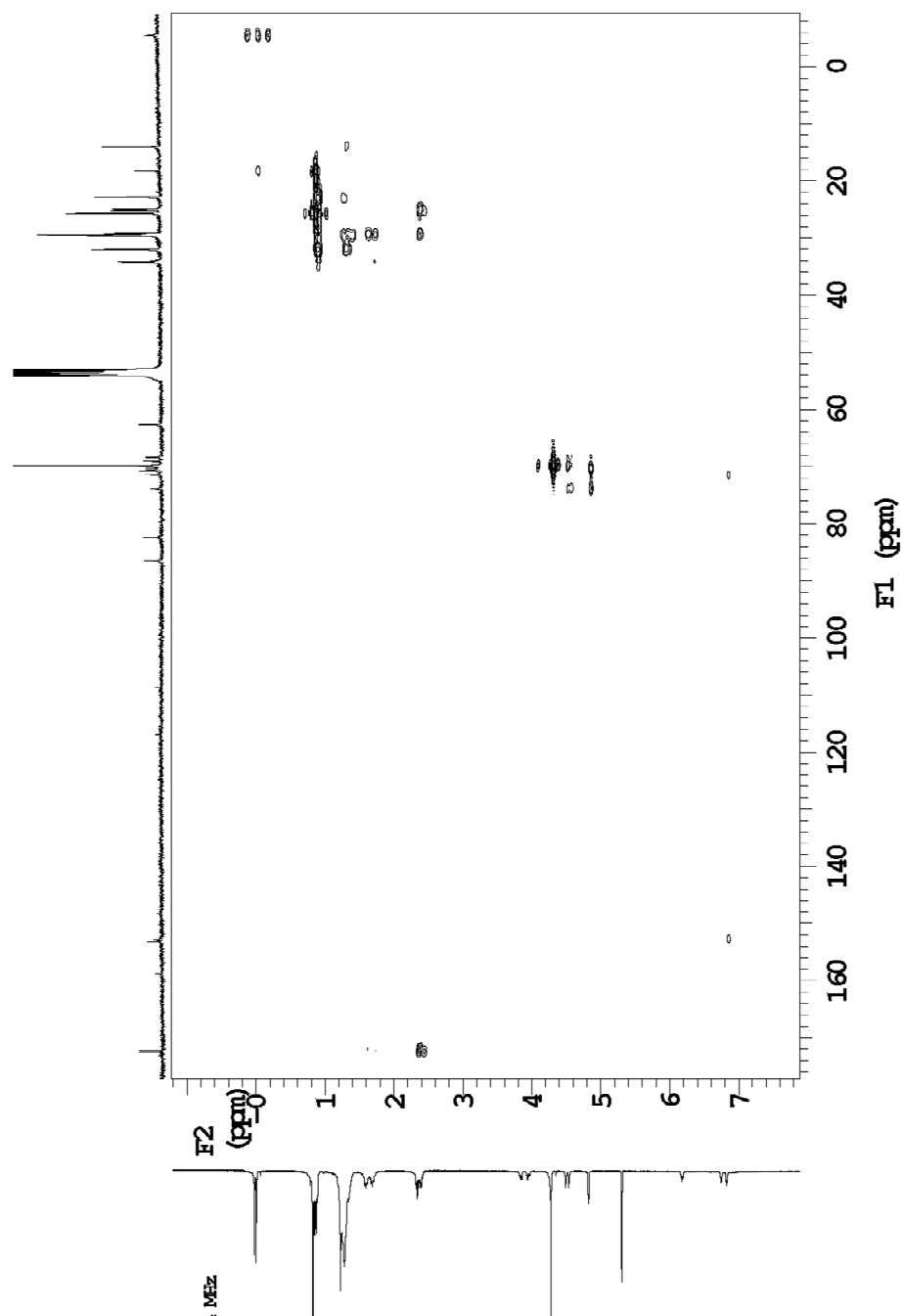
Std Proton parameters

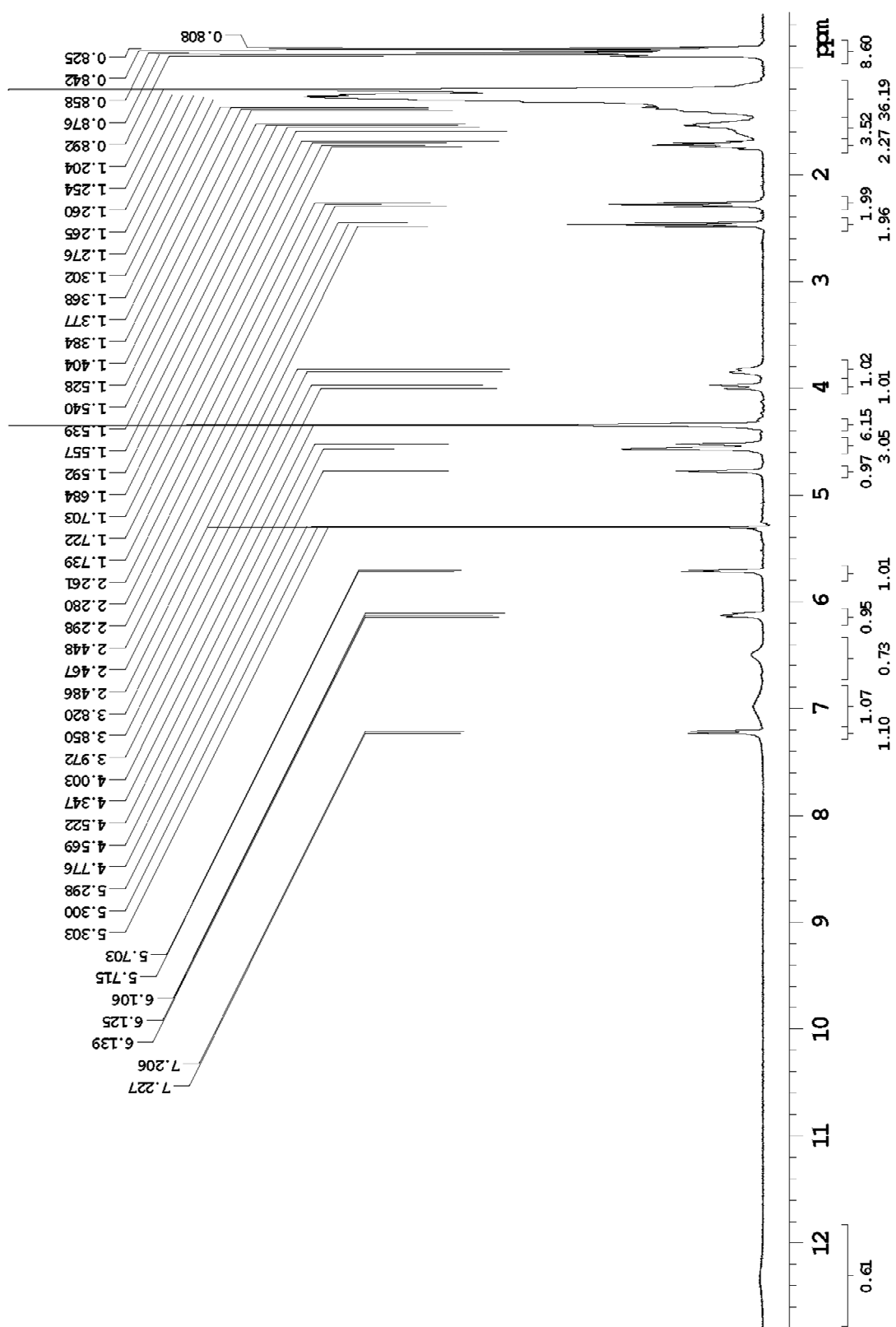
File:

Temp. 25.0 C / 298.1 K  
Operator: sandonat  
INNOVA-600 "FUEL"

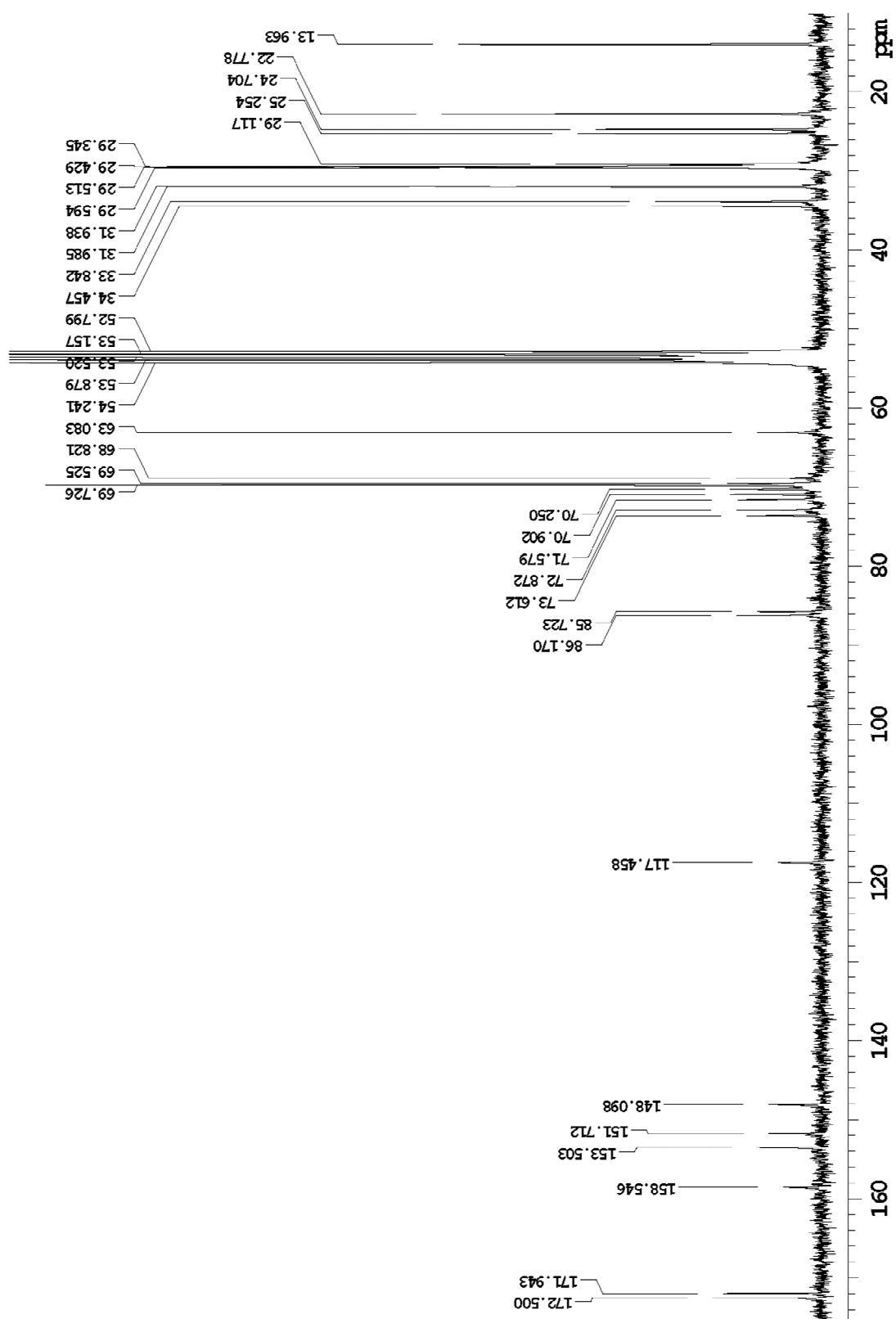
Relax. delay 1.500 sec  
Mixing 0.080 sec  
Acq. time 0.128 sec  
Width 6398.0 Hz  
2D Width 24132.7 Hz  
16 repetitions

200 increments  
OBSERVE H1, 399.9253434 MHz  
DATA PROCESSING  
Sine ball 0.064 sec  
F1 DATA PROCESSING  
Sine ball 0.008 sec  
F1 size 4096 x 2048  
Total time 0 min 0 sec





<sup>1</sup>H-NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Fc5OH**



<sup>13</sup>C{<sup>1</sup>H}NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Fc5OH**.

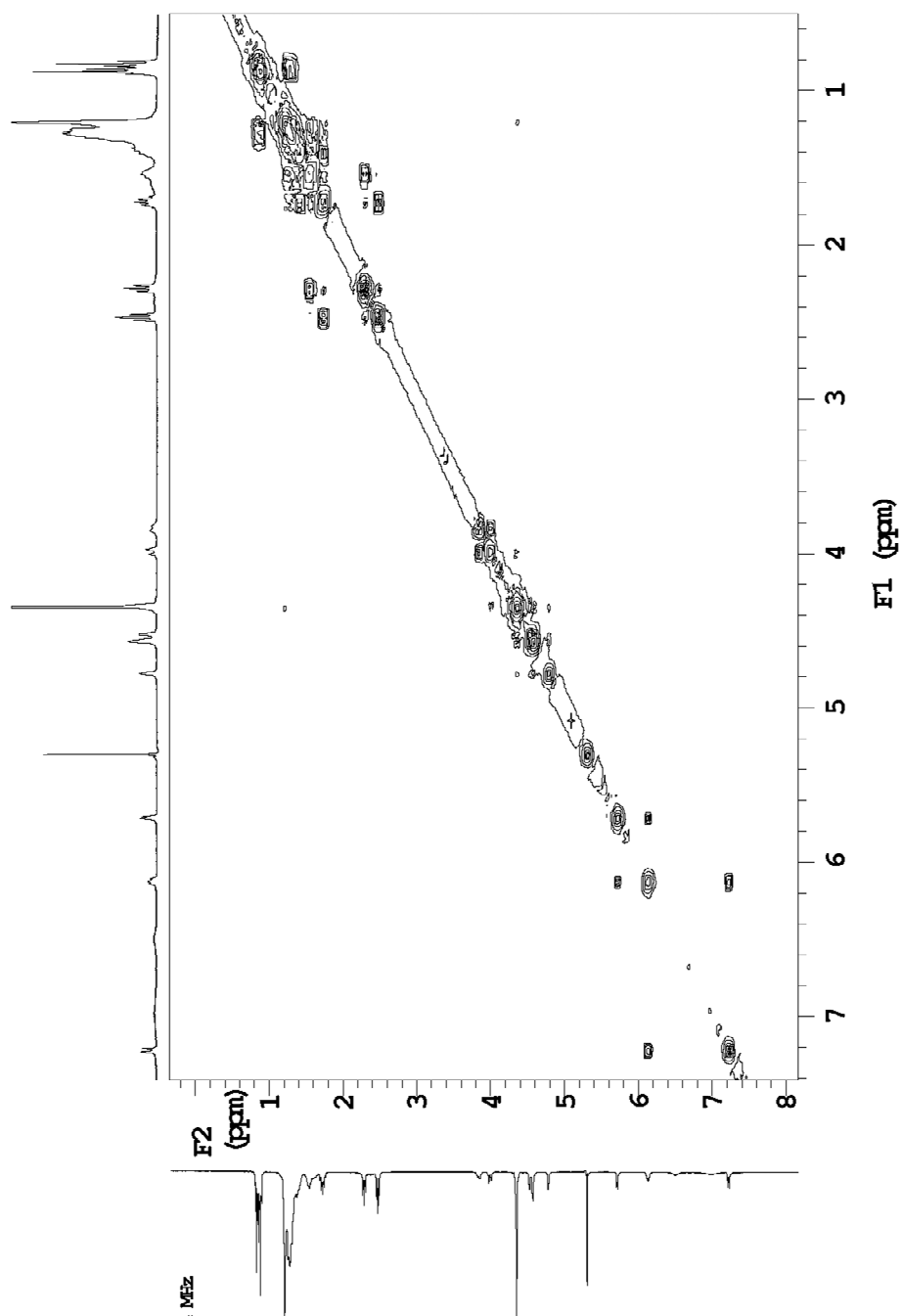
Std Proton parameters

File:

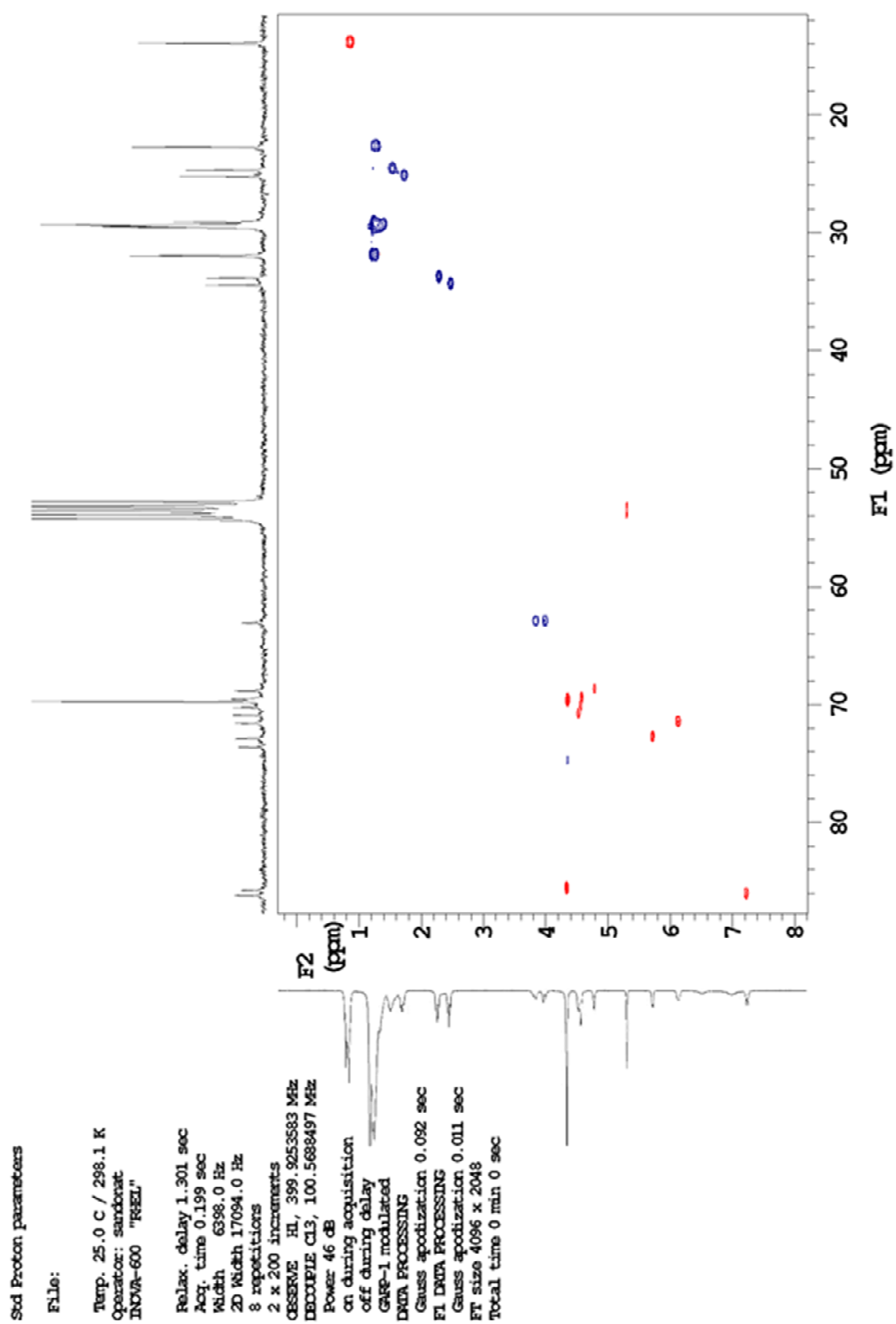
Temp. 25.0 C / 298.1 K  
Operator: sandorat  
INOVA-600 "RHEI"

Relax. delay 1.301 sec  
Acq. time 0.160 sec  
Width 6398.0 Hz  
2D Width 6398.0 Hz  
4 repetitions  
128 increments

OBSERVE H1, 399.9253544 MHz  
DATA PROCESSING  
Sine ball 0.080 sec  
F1 DATA PROCESSING  
Sine ball 0.020 sec  
F1 size 2048 x 2048  
Total time 0 min 0 sec



gCOSY spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Fc5OH**



gHSQC spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Fc5OH**

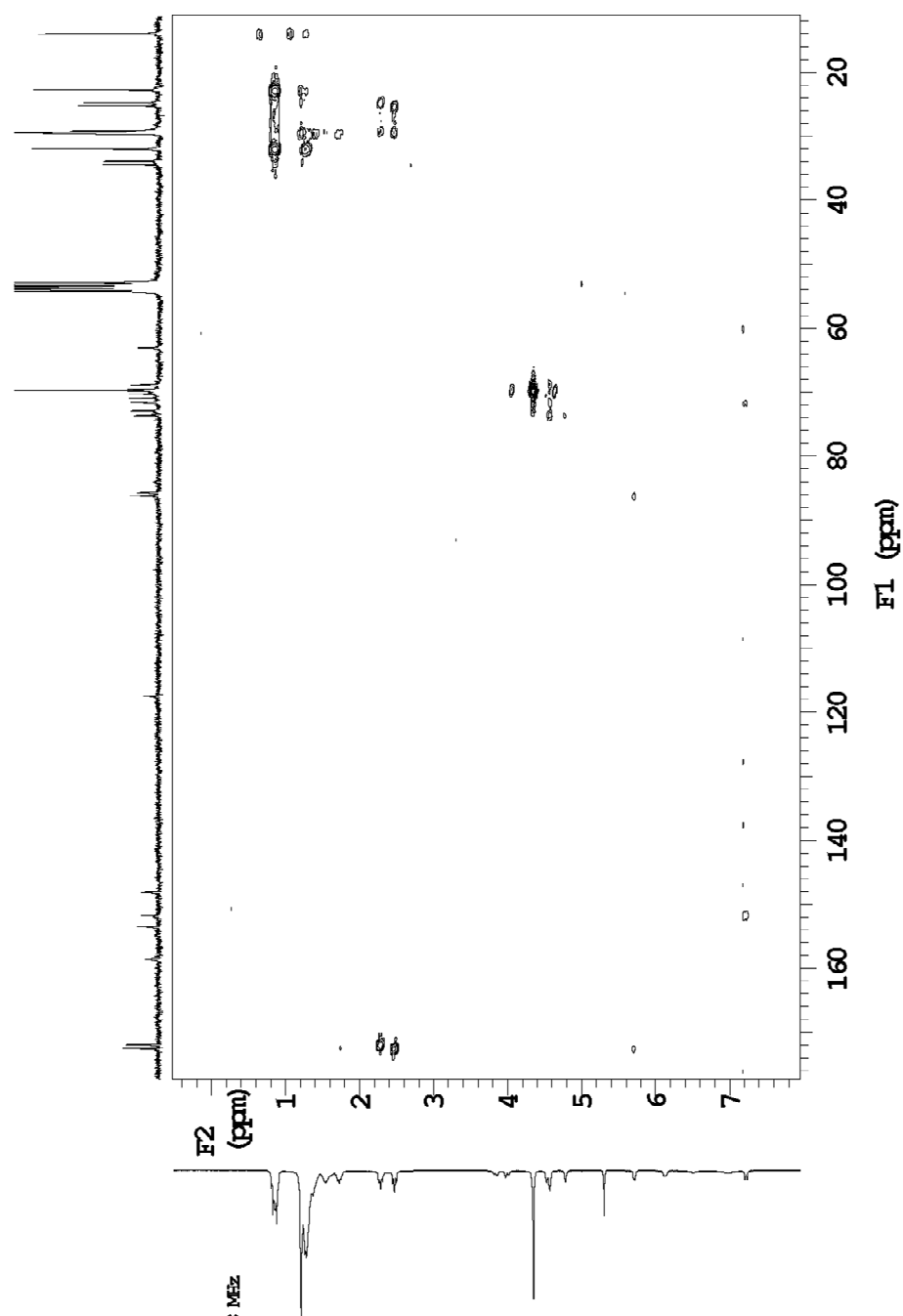
Standard Proton Parameters - i300@fci.unibo.it

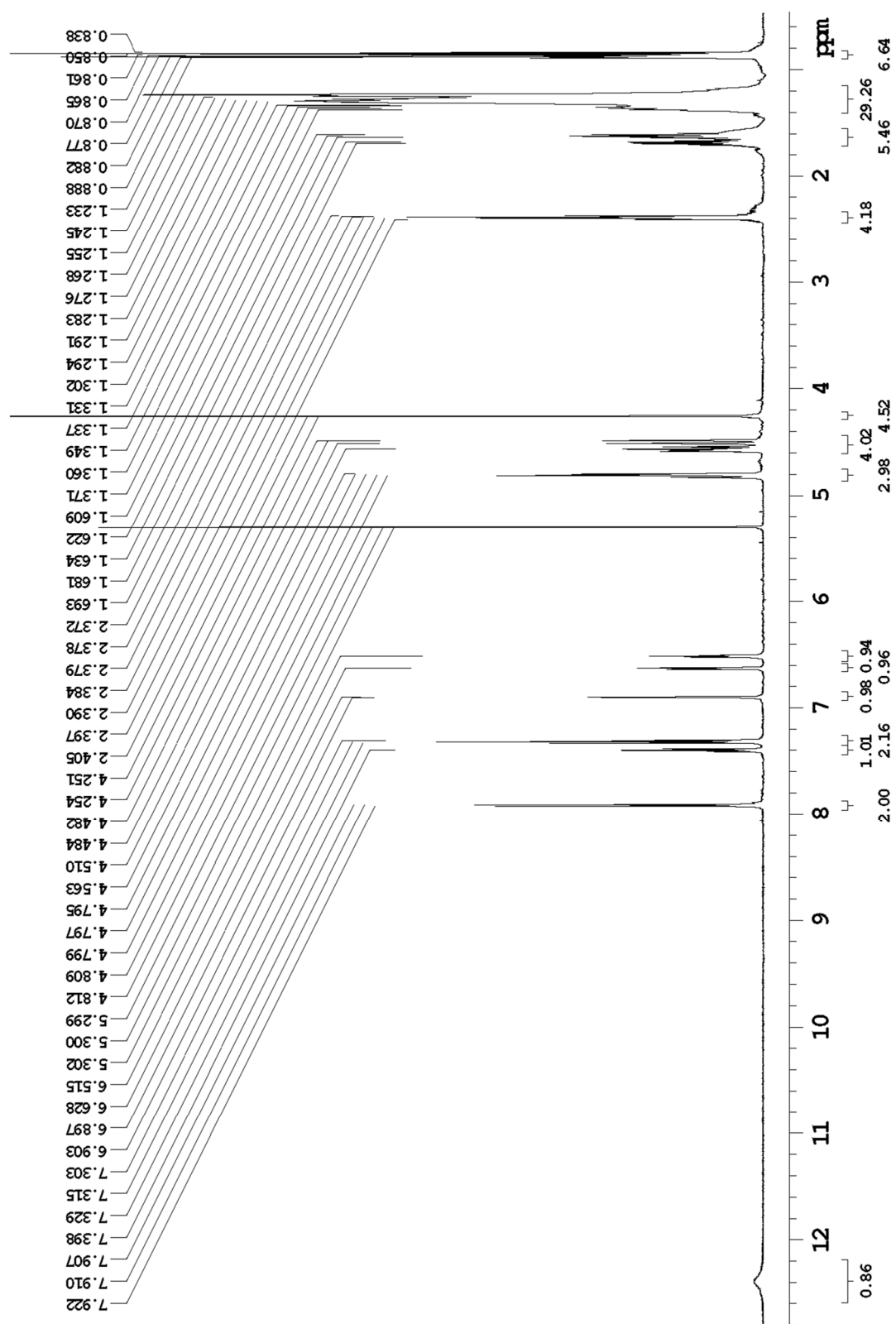
File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac  
INOVA-600 "RHEL"

Relax. delay 2.000 sec  
Mixing 0.080 sec  
Acq. time 0.200 sec  
Width 4800.5 Hz  
2D Width 18107.7 Hz  
32 repetitions  
256 increments

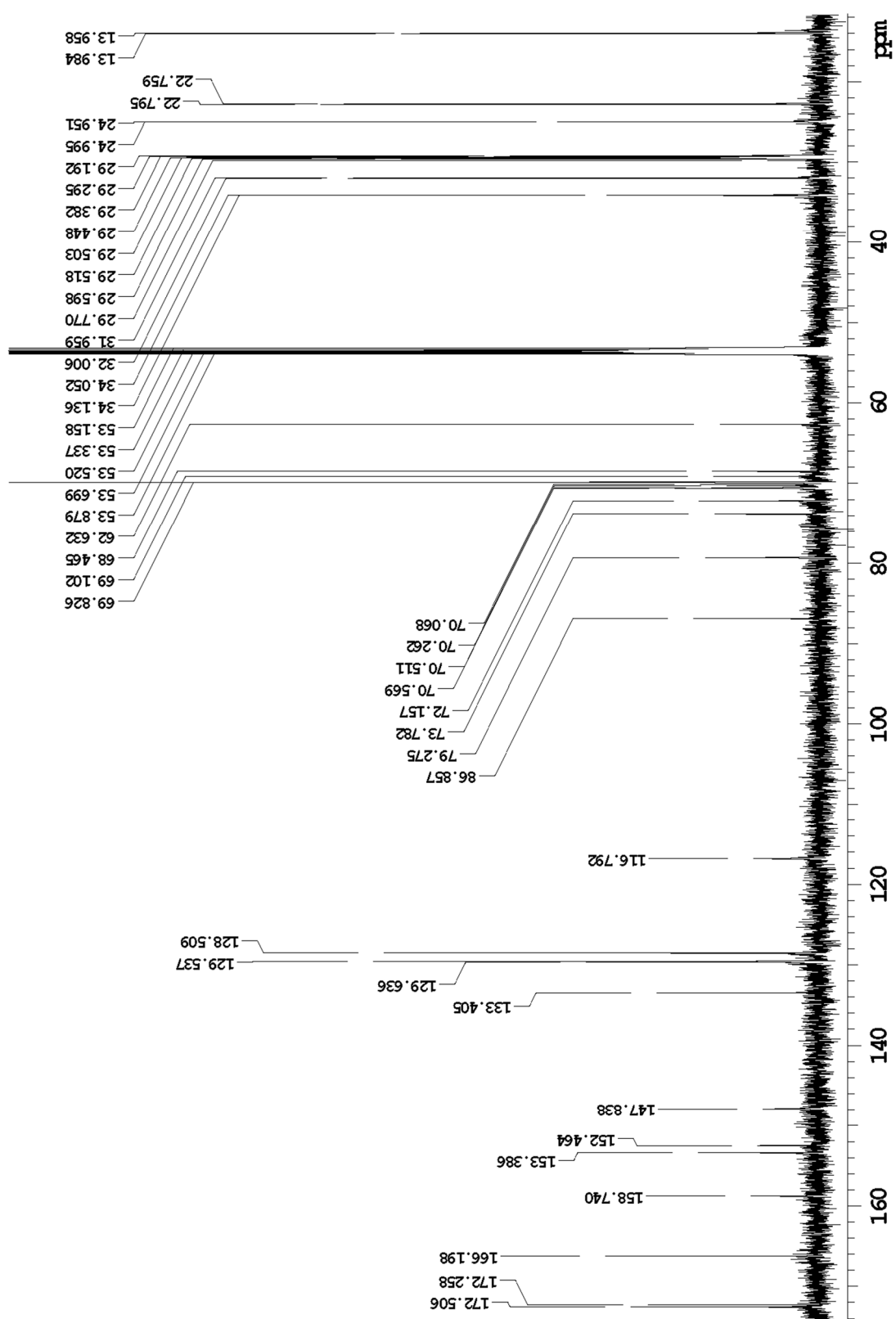
CESRVE H1, 300.0321273 MHz  
DATA PROCESSING  
Sine ball 0.100 sec  
F1 DATA PROCESSING  
Sine ball 0.014 sec  
F1 size 4096 x 2048  
Total time 0 min 0 sec





<sup>1</sup>H-NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Fc5Ph**





$^{13}\text{C}\{^1\text{H}\}$  NMR (CD<sub>2</sub>Cl<sub>2</sub>, 600 MHz) of **8Fc5Ph**.

i600 std parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.000 sec  
Acq. time 0.213 sec  
Width 9611.9 Hz  
2D Width 9611.9 Hz  
8 repetitions  
200 increments

OBSERVE F1, 599.7352630 MHz

DATA PROCESSING

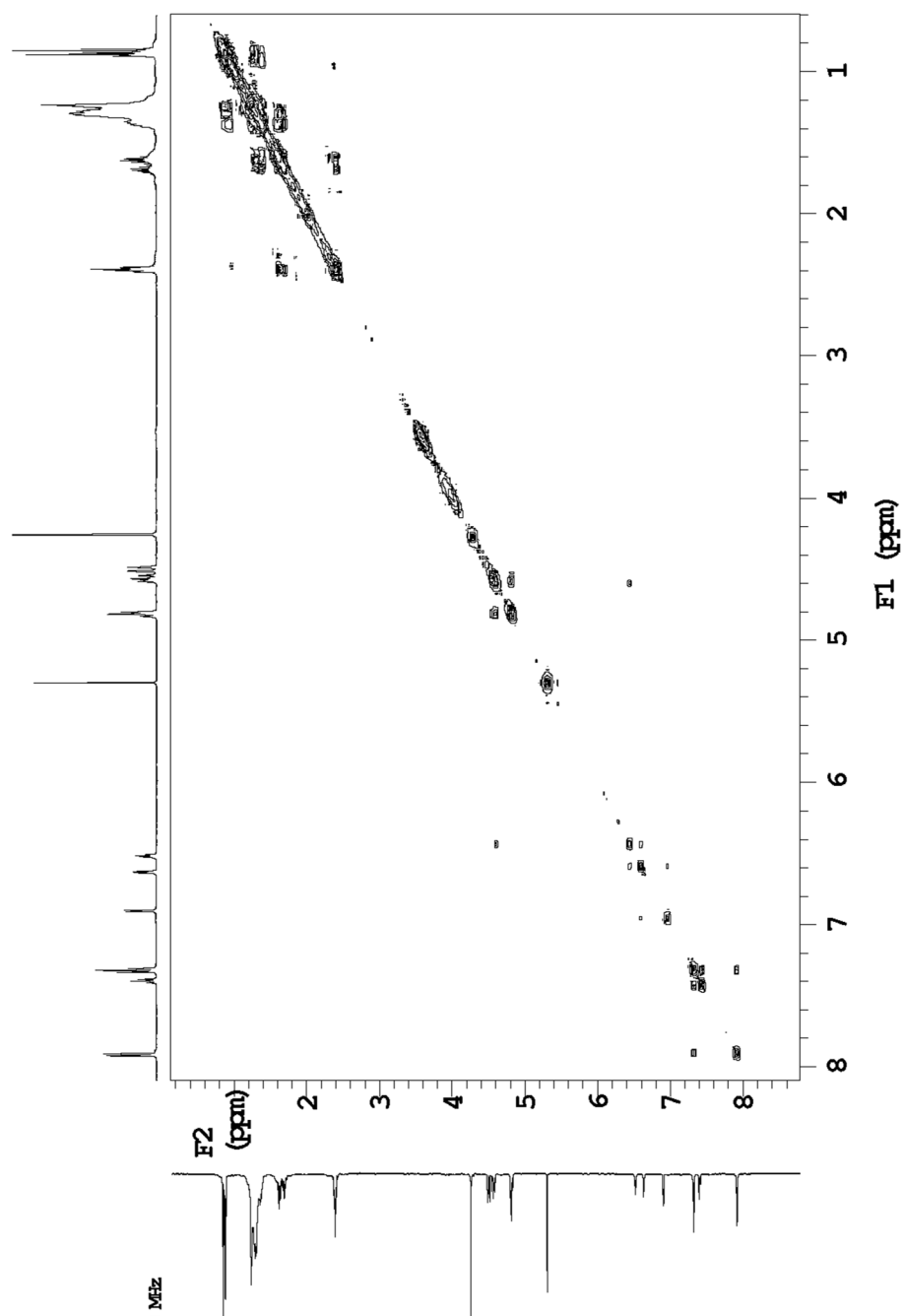
Sine bell 0.107 sec

F1 DATA PROCESSING

Sine bell 0.021 sec

FT size 4096 x 4096

Total time 0 min 0 sec



gCOSY spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Fc5Ph**

i600 std parameters

File:

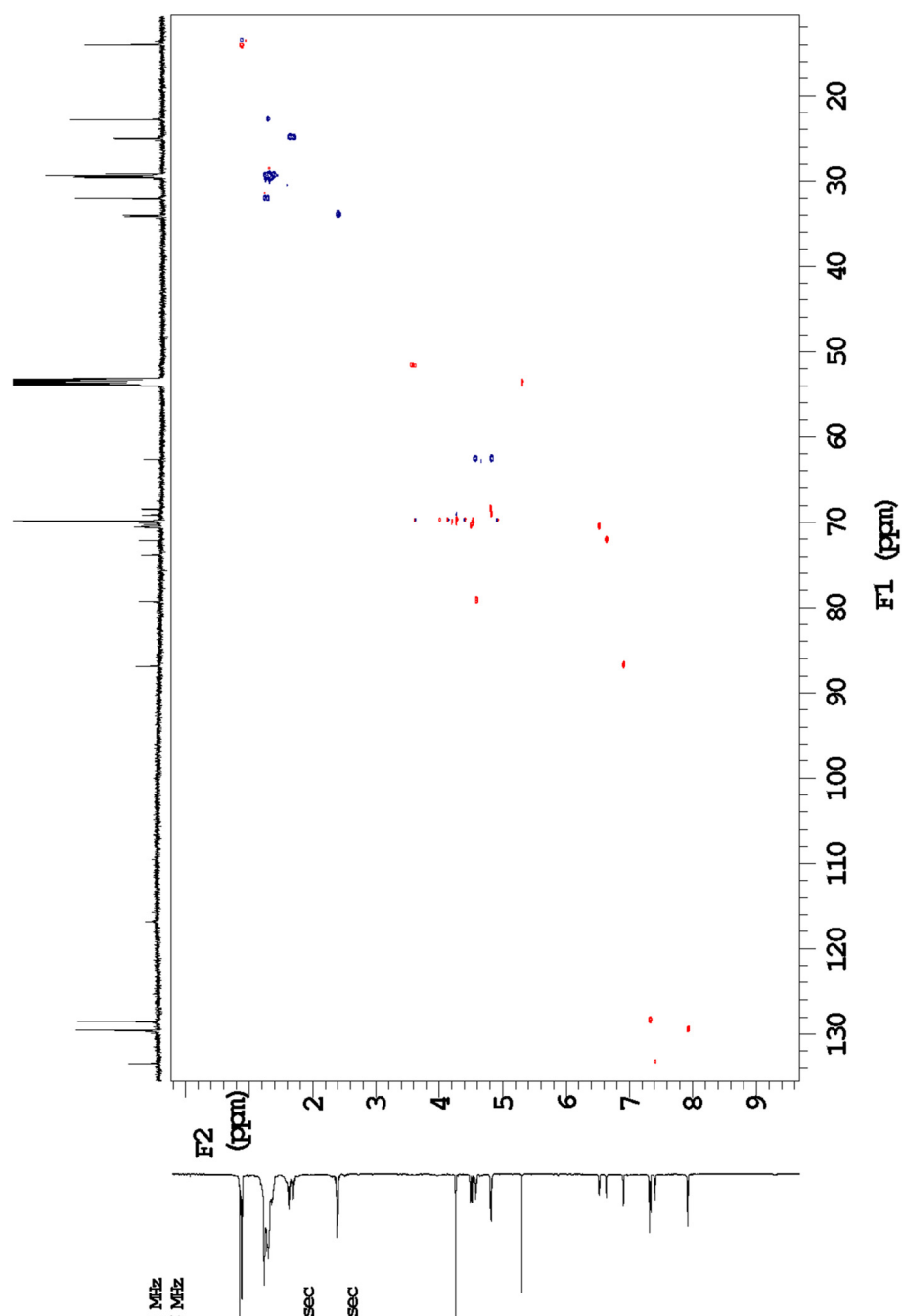
Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.400 sec  
Acq. time 0.199 sec  
Width 9611.9 Hz  
2D Width 25632.8 Hz  
8 repetitions

2 x 256 increments  
OBSERVE H1, 599.7352602 MHz  
DECOUPLE C13, 150.8148596 MHz  
Power 46 dB

on during acquisition  
off during delay  
W40 Triple modulated  
DATA PROCESSING

Gauss apodization 0.092 sec  
F1 DATA PROCESSING  
Gauss apodization 0.009 sec  
FT size 8192 x 2048  
Total time 0 min 0 sec



gHSQC spectrum ( $\text{CD}_2\text{Cl}_2$ , 600 MHz) of **8Fc5Ph**

i600 std parameters

File:

Temp. 25.0 C / 298.1 K  
Operator: sangiac

Relax. delay 1.400 sec  
Mixing 0.080 sec  
Acq. time 0.128 sec  
Width 9611.9 Hz  
2D Width 36199.1 Hz  
16 repetitions  
256 increments

OBSERVE HL, 599.7352598 MHz

DATA PROCESSING

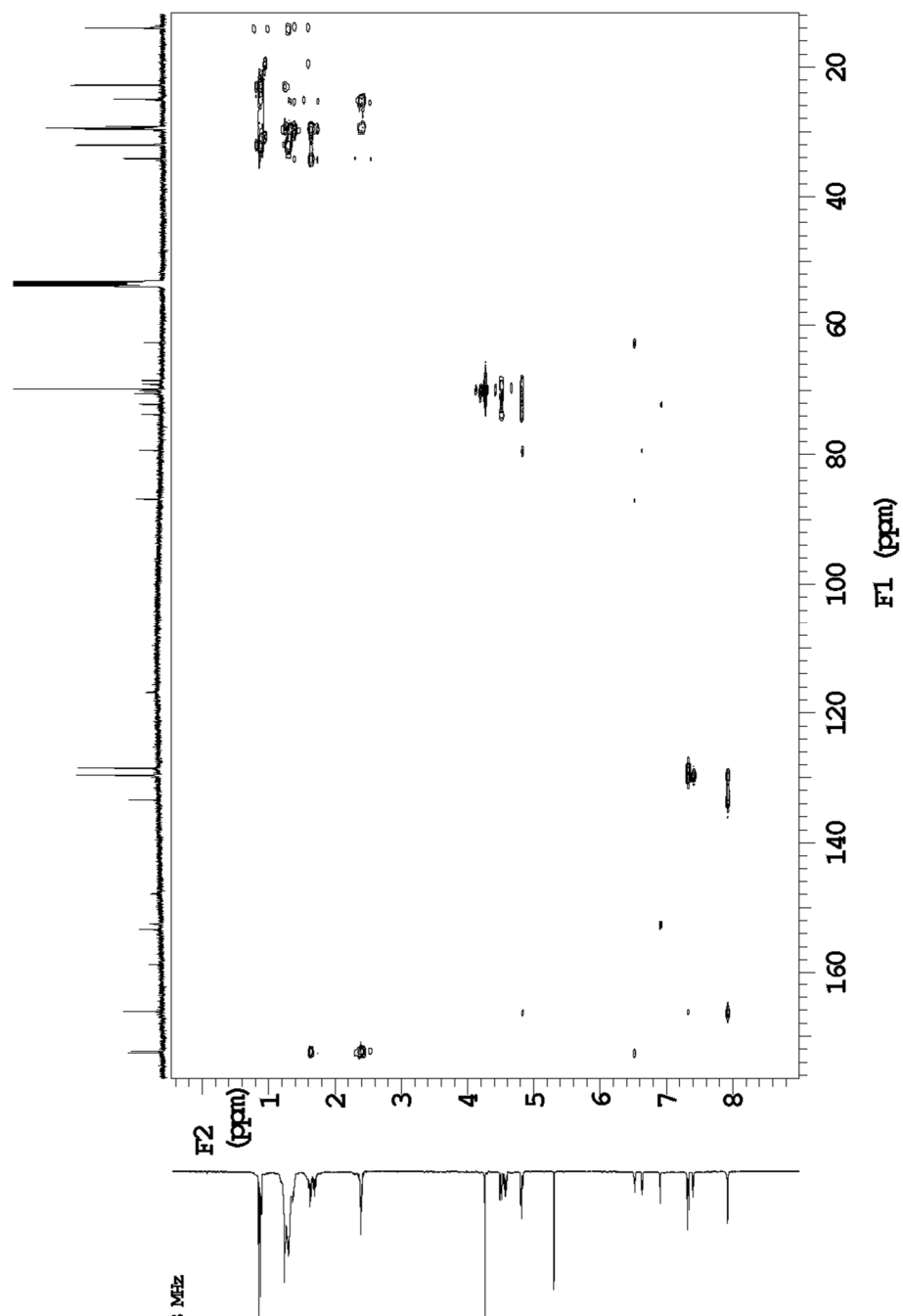
Sine bell 0.064 sec

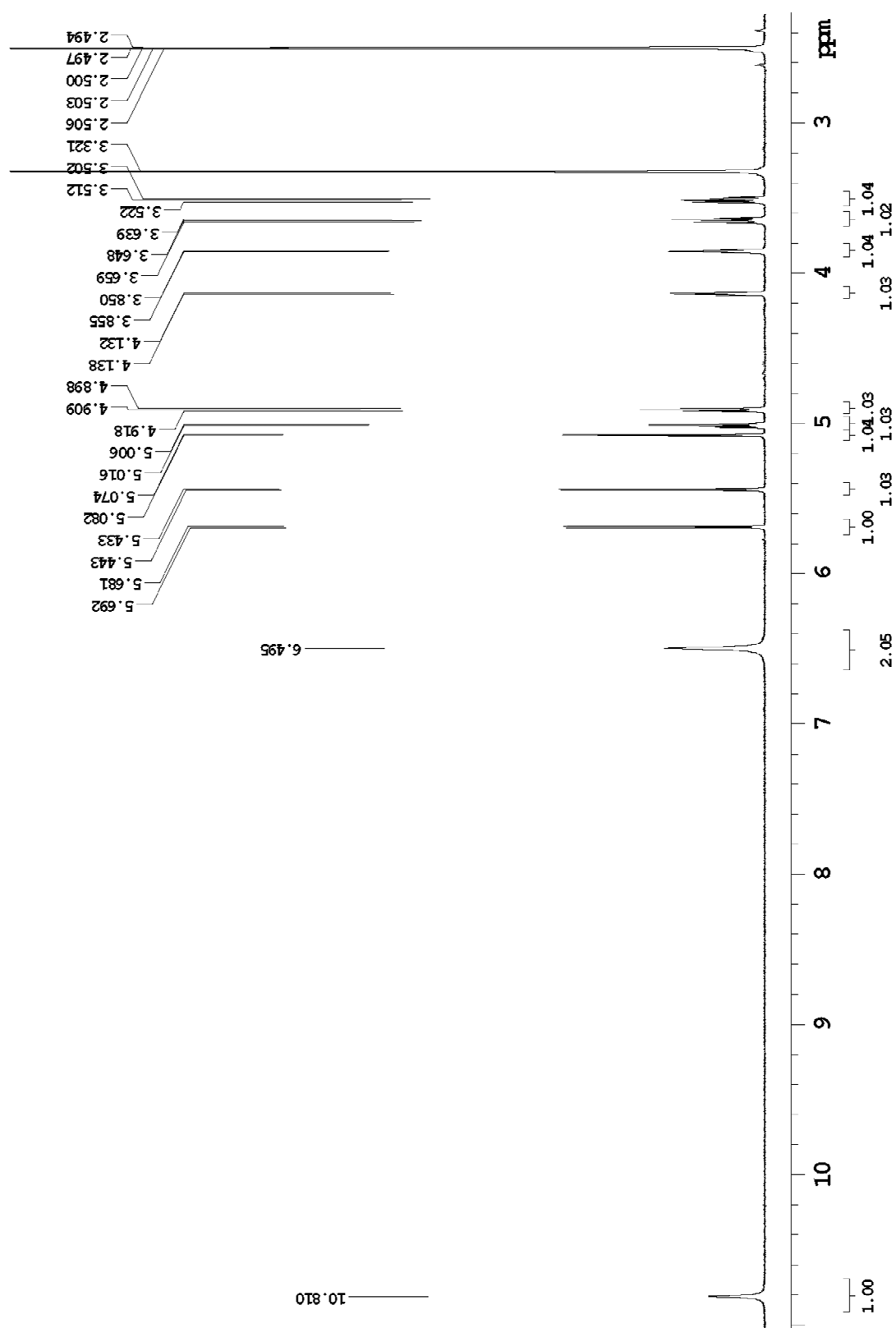
F1 DATA PROCESSING

Sine bell 0.007 sec

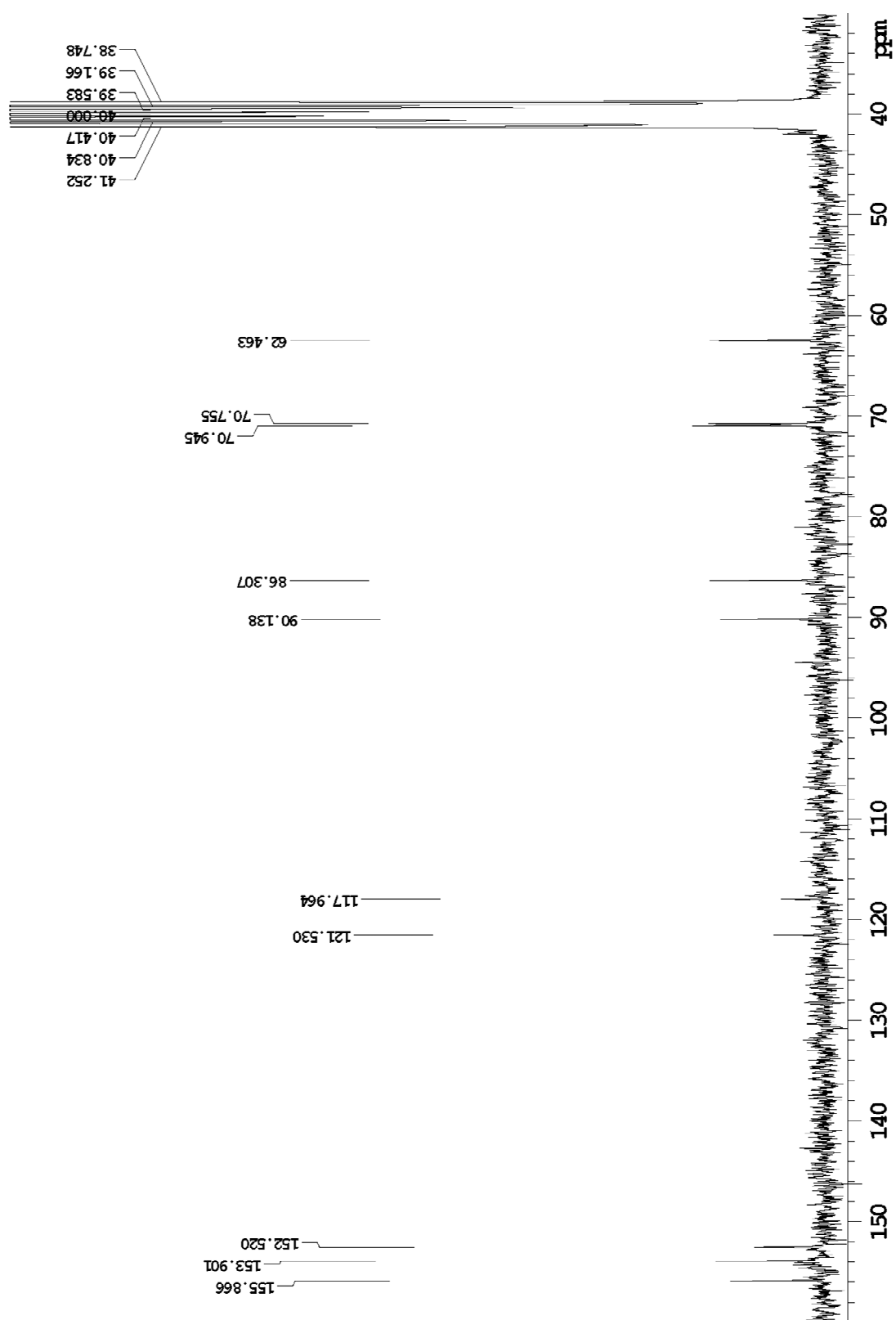
FT size 4096 x 2048

Total time 0 min 0 sec

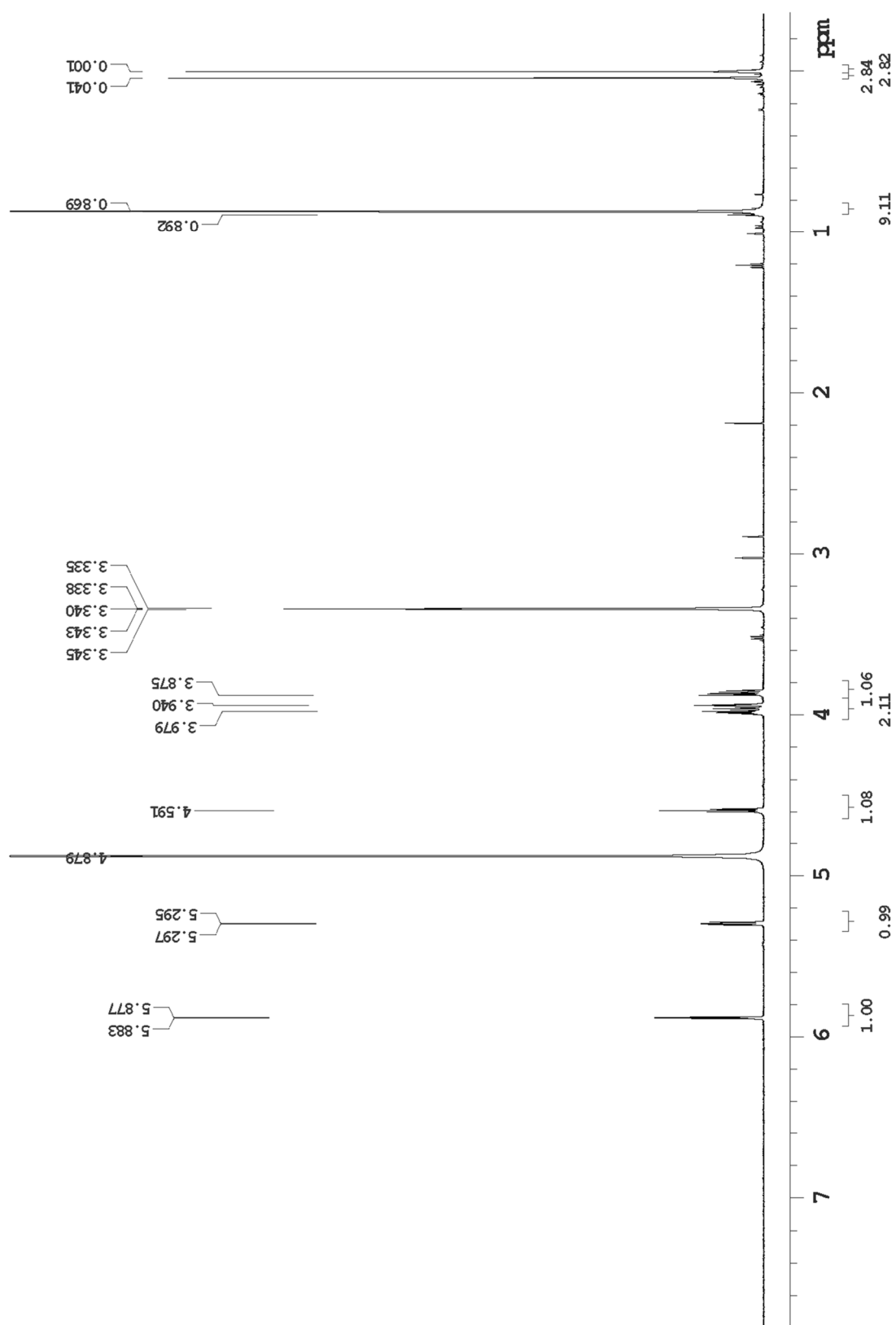




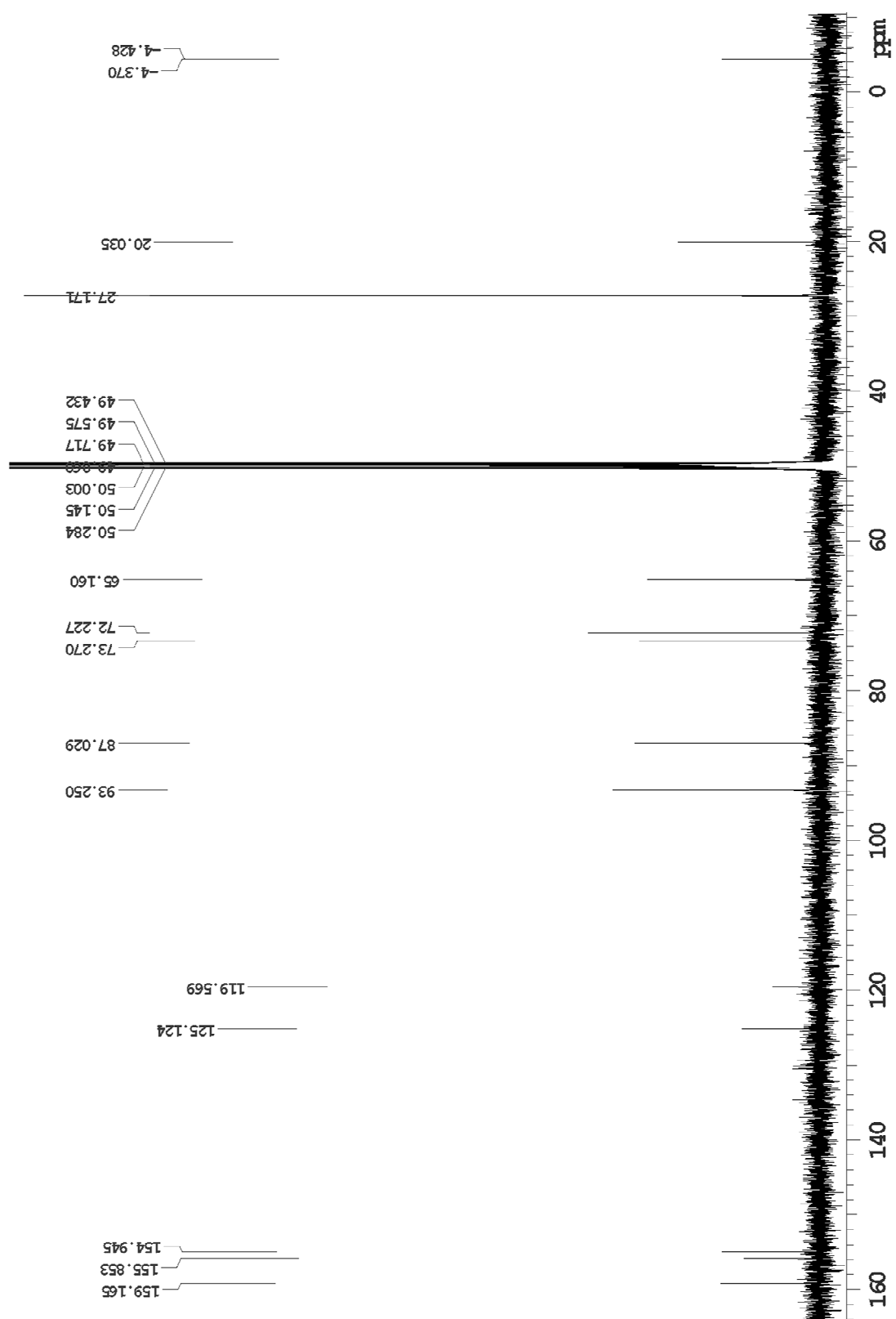
<sup>1</sup>H-NMR (dms0-d<sub>6</sub>, 600 MHz) of **9**



$^{13}\text{C}\{^1\text{H}\}$  NMR (dms0-d<sub>6</sub>, 75 MHz) of **9**

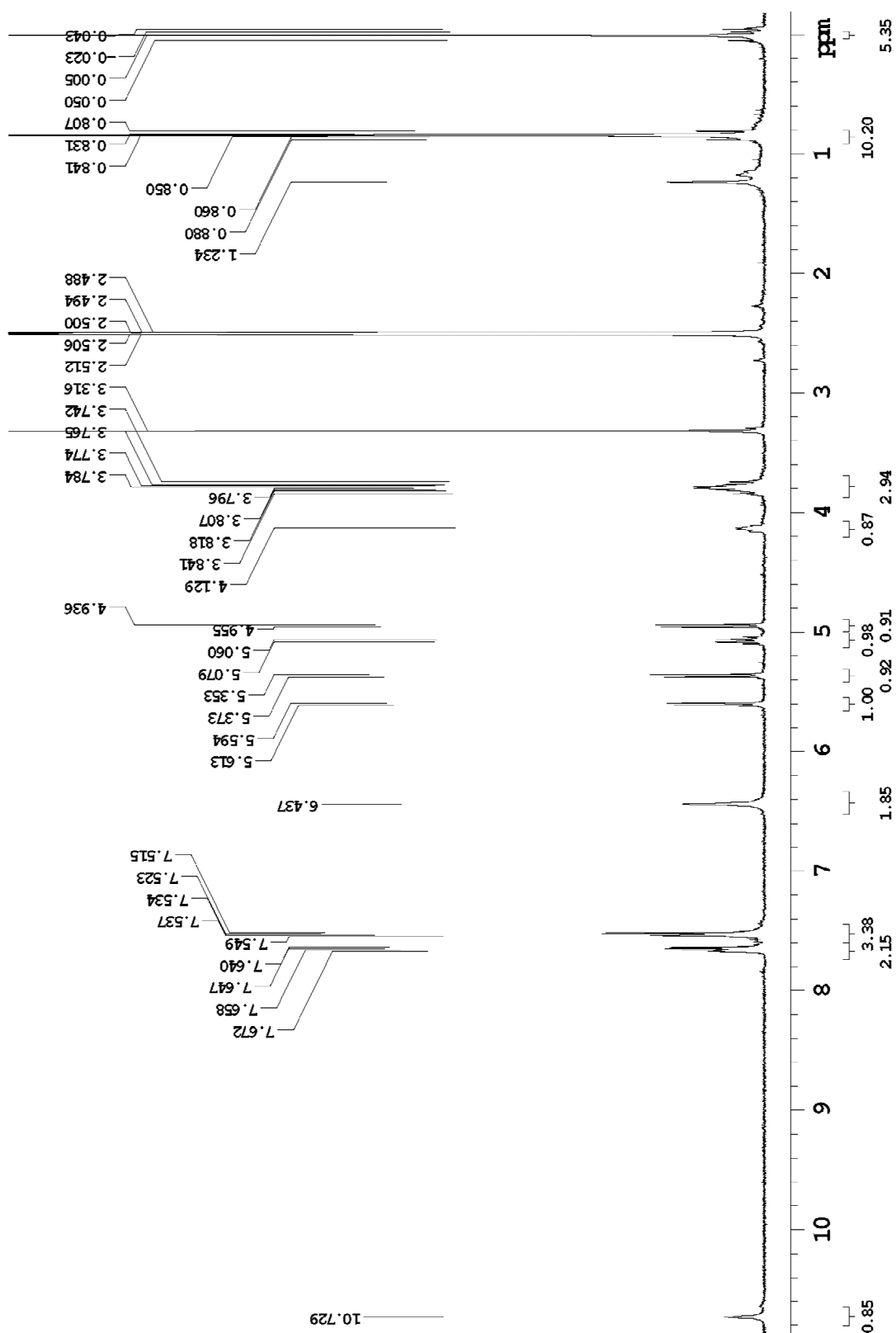


<sup>1</sup>H-NMR (CD<sub>3</sub>OD, 600 MHz) of **10**

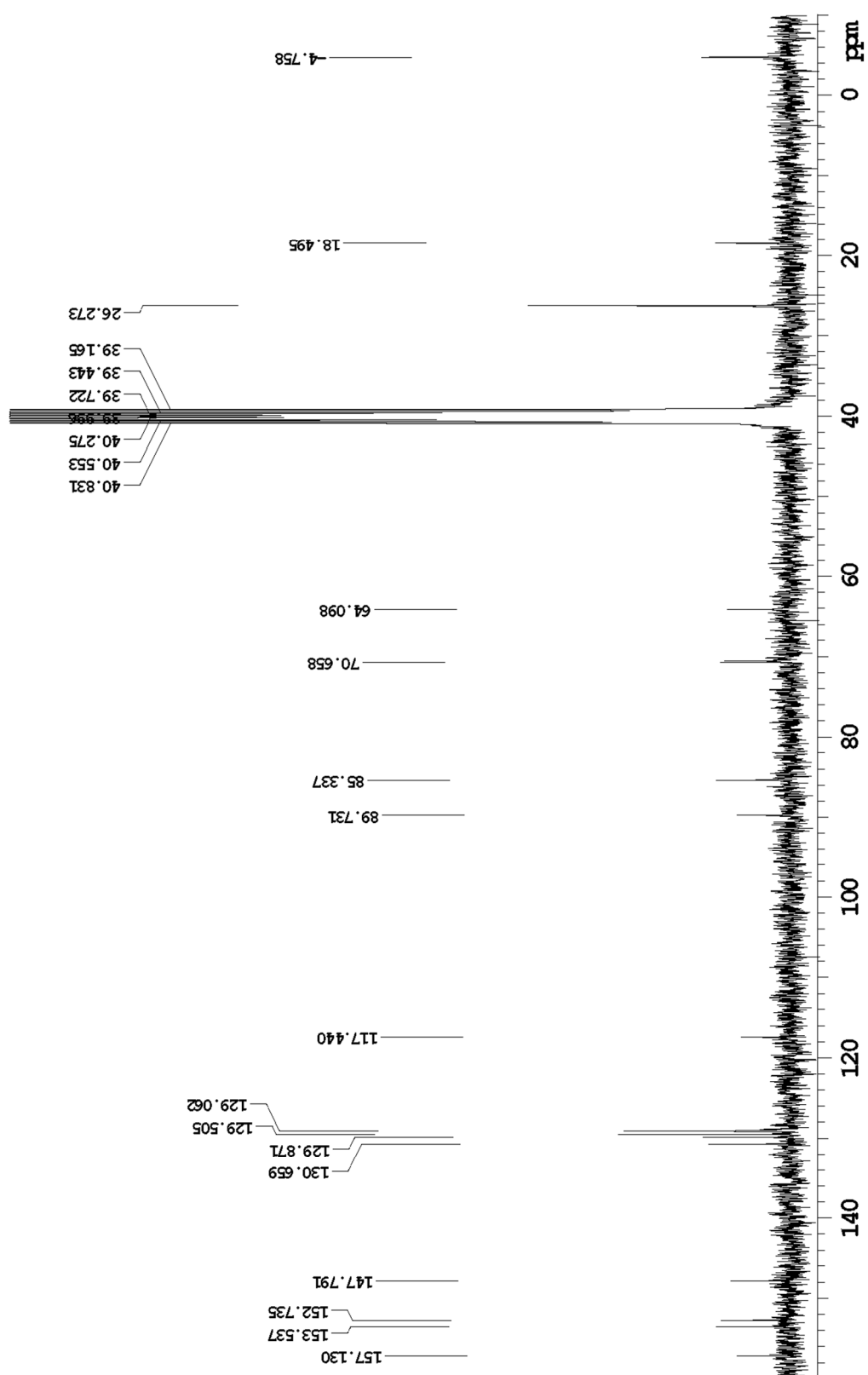


$^{13}\text{C}\{^1\text{H}\}$  NMR (CD<sub>3</sub>OD, 151 MHz) of **10**

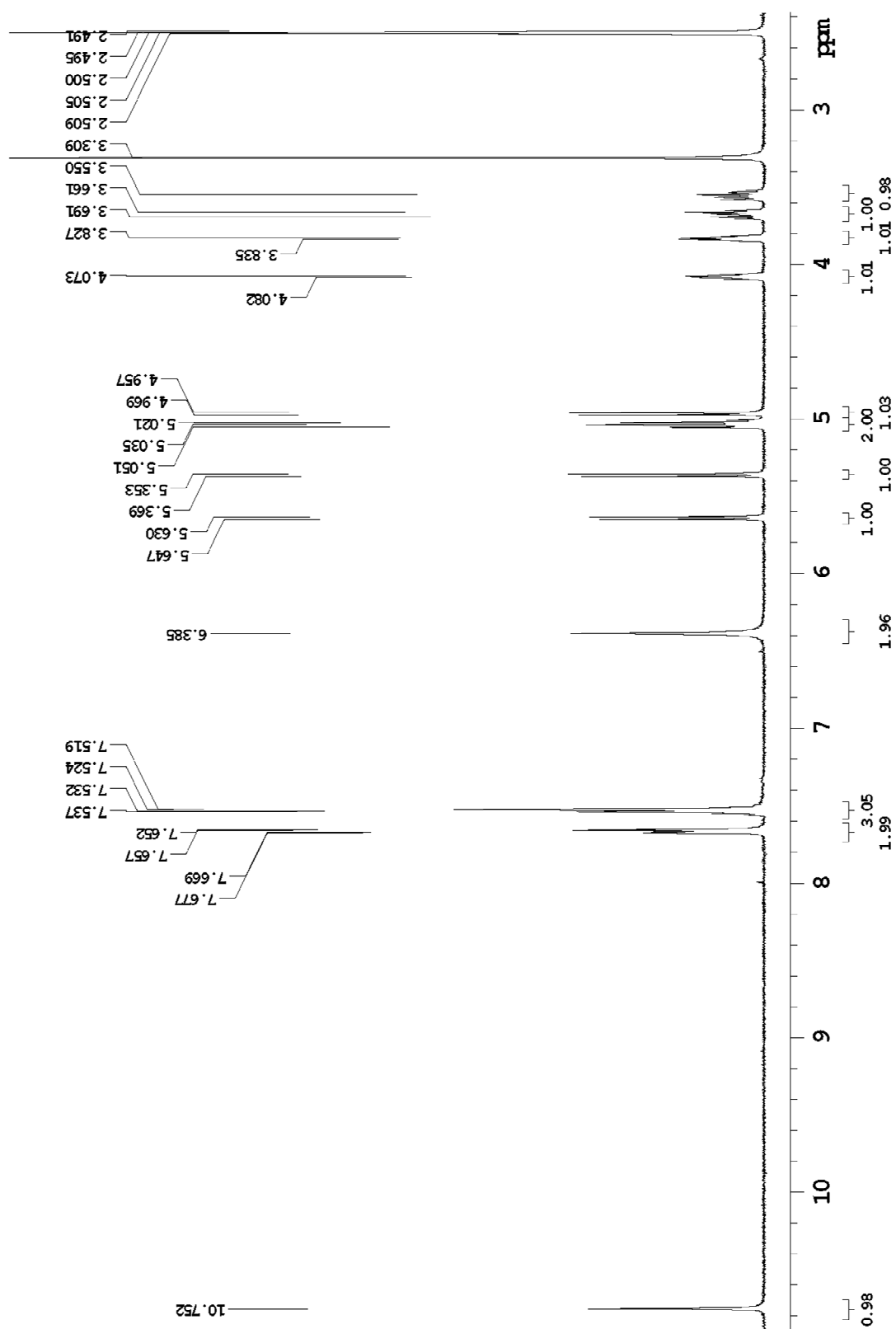




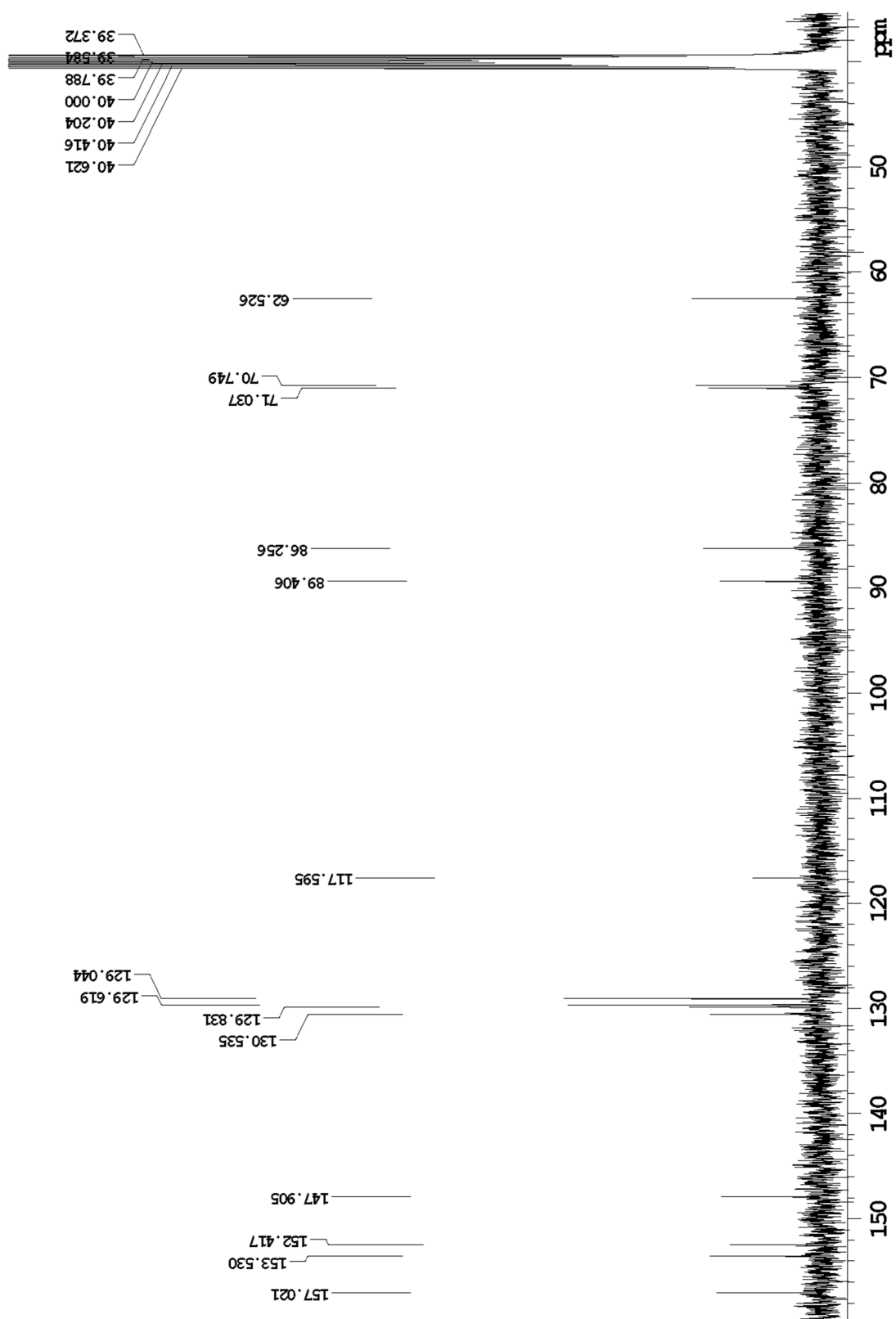
<sup>1</sup>H-NMR (dms0-d<sub>6</sub>, 600 MHz) of **11**



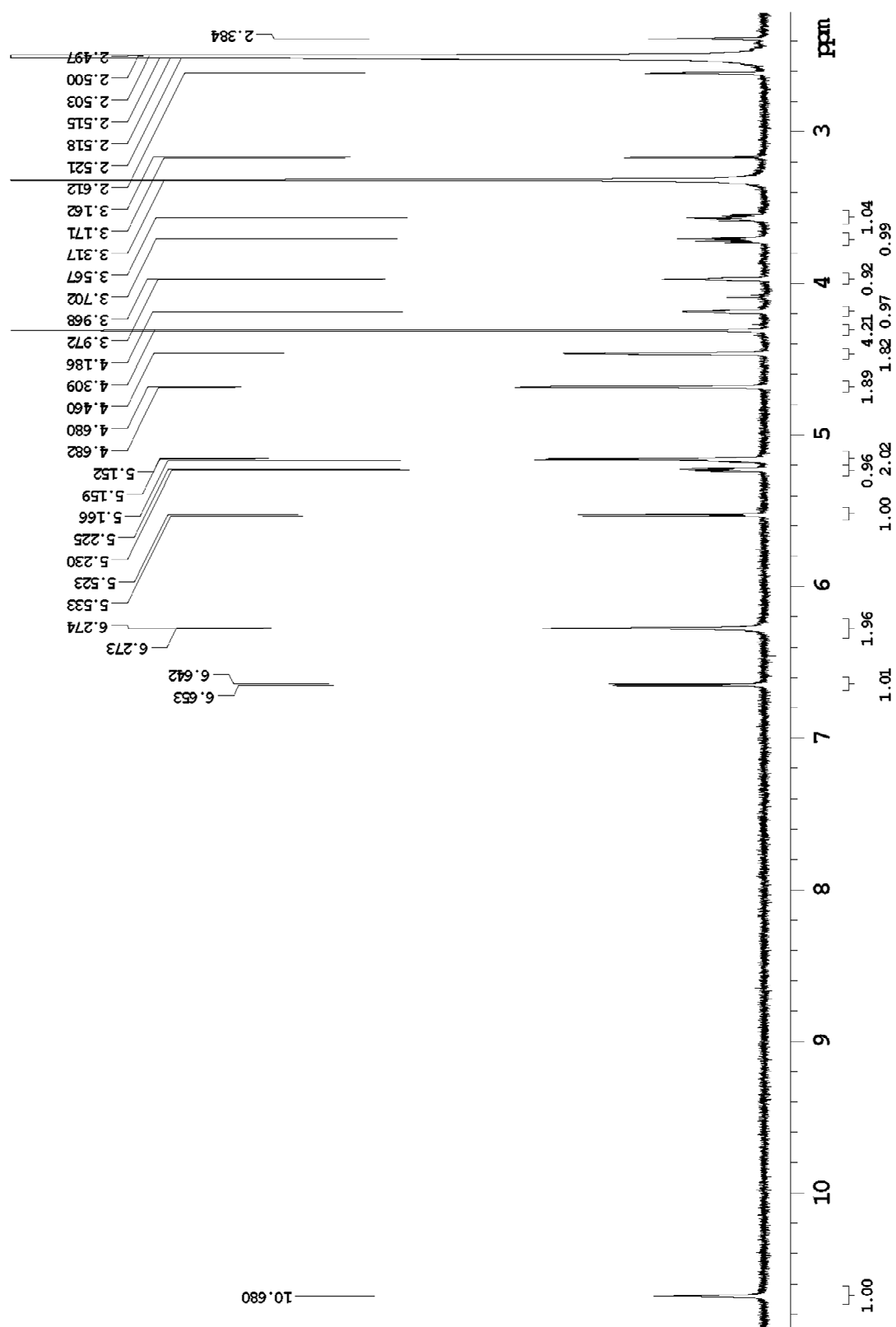
$^{13}\text{C}\{^1\text{H}\}$  NMR (dms0-d<sub>6</sub>, 75 MHz) of **11**



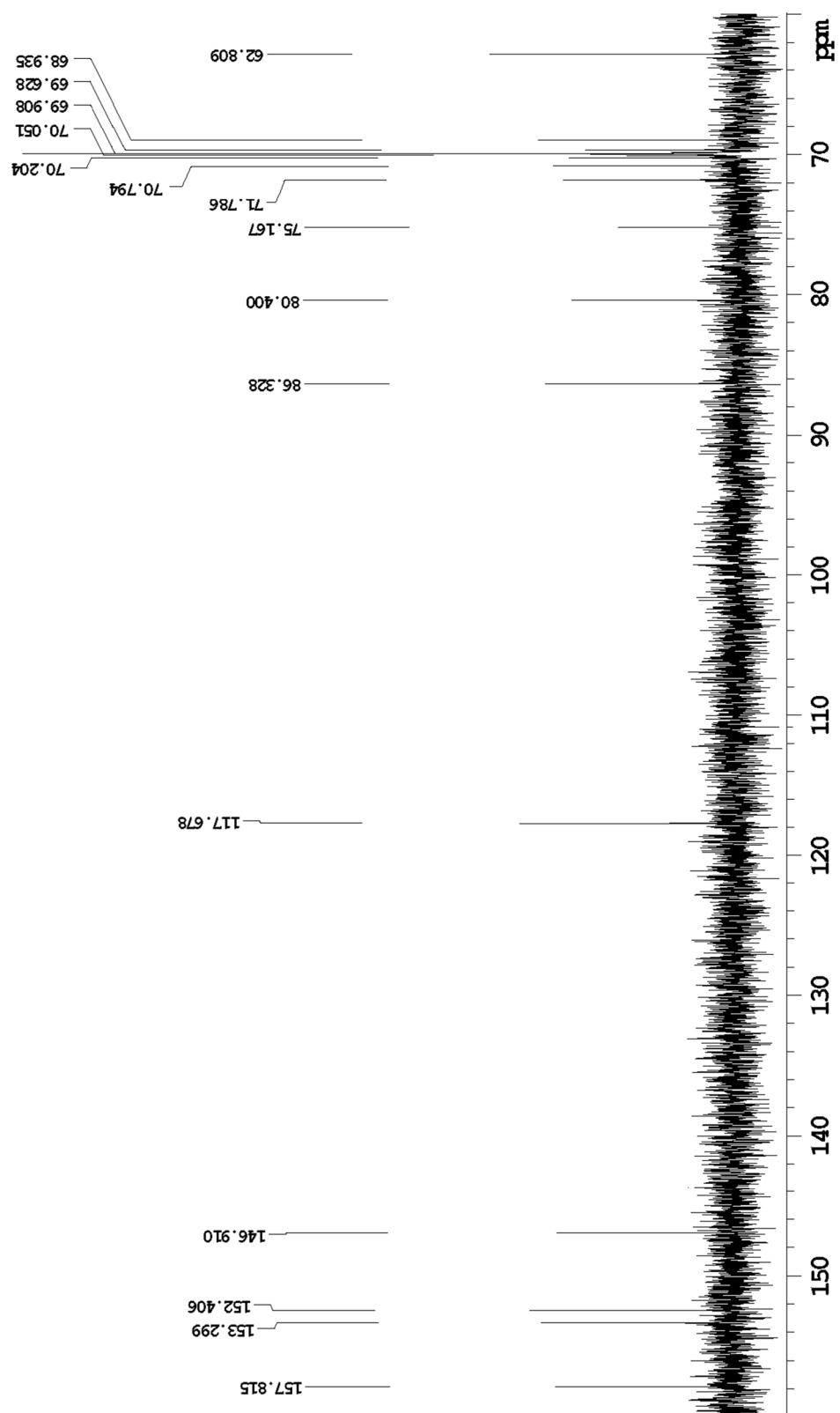
<sup>1</sup>H-NMR (dms0-d<sub>6</sub>, 400 MHz) of **12**



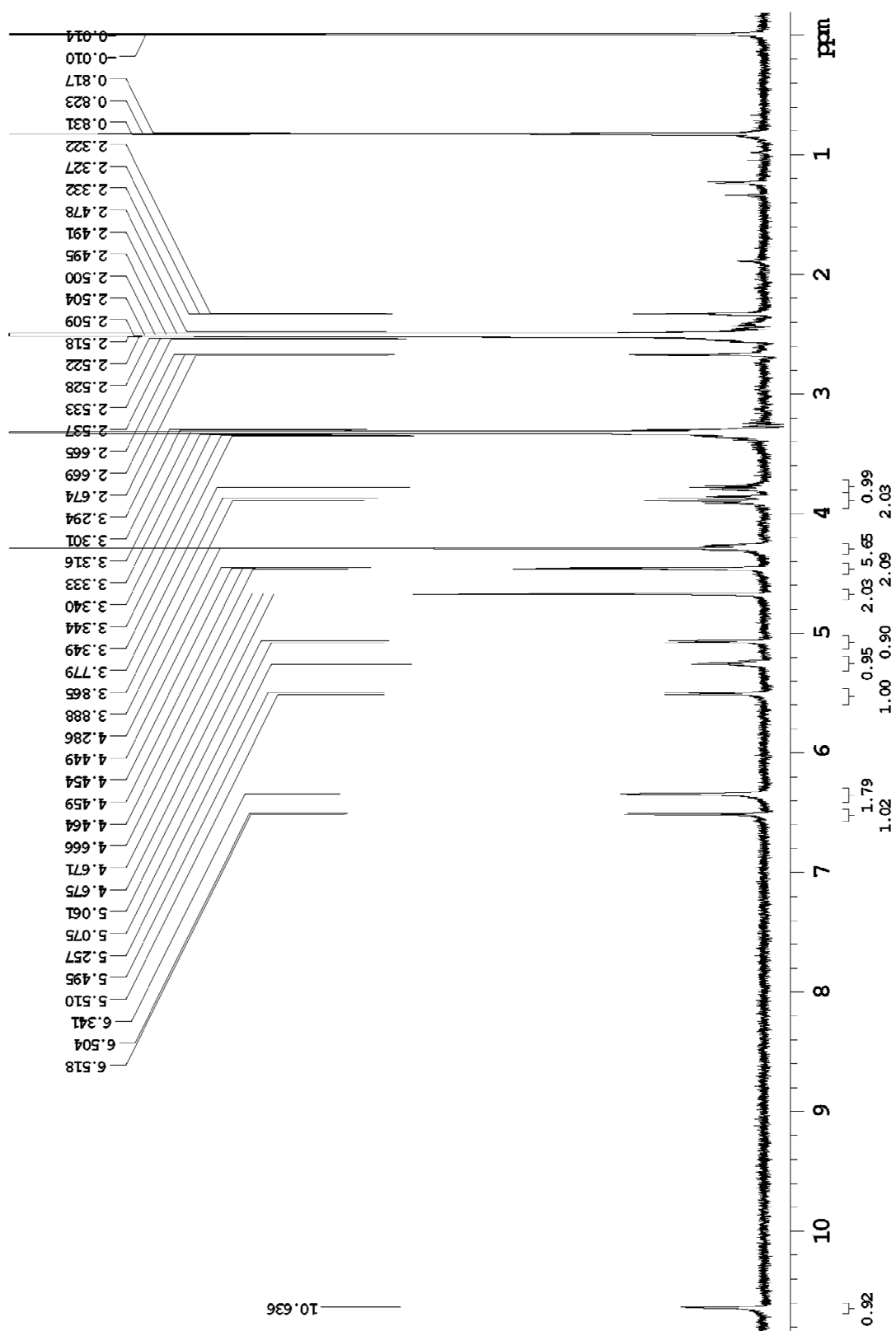
$^{13}\text{C}\{^1\text{H}\}$  NMR (dms0-d<sub>6</sub>, 101 MHz) of **12**



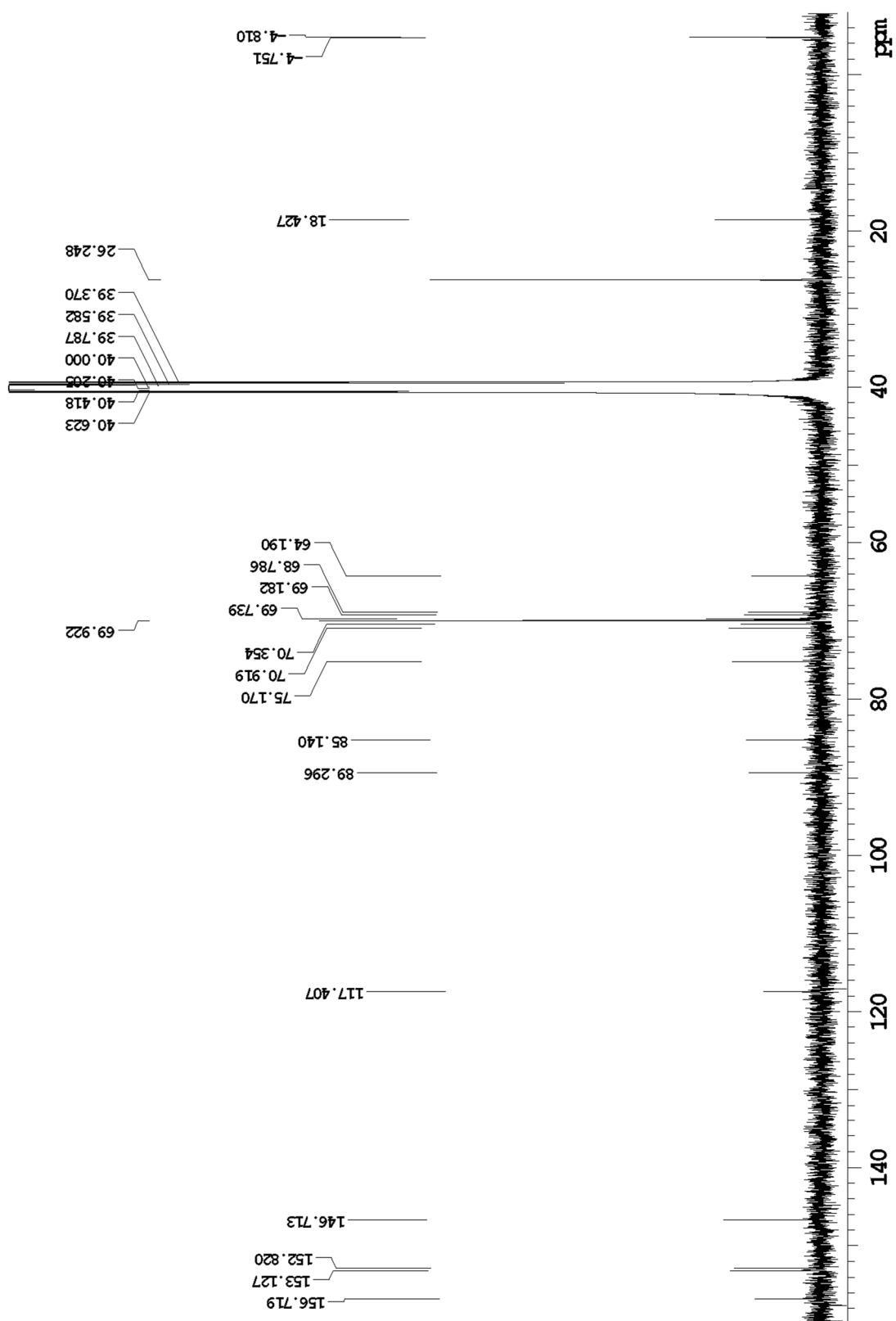
<sup>1</sup>H-NMR (dms0-d<sub>6</sub>, 600 MHz) of **13**



$^{13}\text{C}\{^1\text{H}\}$  NMR (dmsso- $\text{d}_6$ , 151 MHz) of **13**



<sup>1</sup>H-NMR (dms0-d<sub>6</sub>, 400 MHz) of **14**



$^{13}\text{C}\{^1\text{H}\}$  NMR (dms0-d<sub>6</sub>, 101 MHz) of **14**