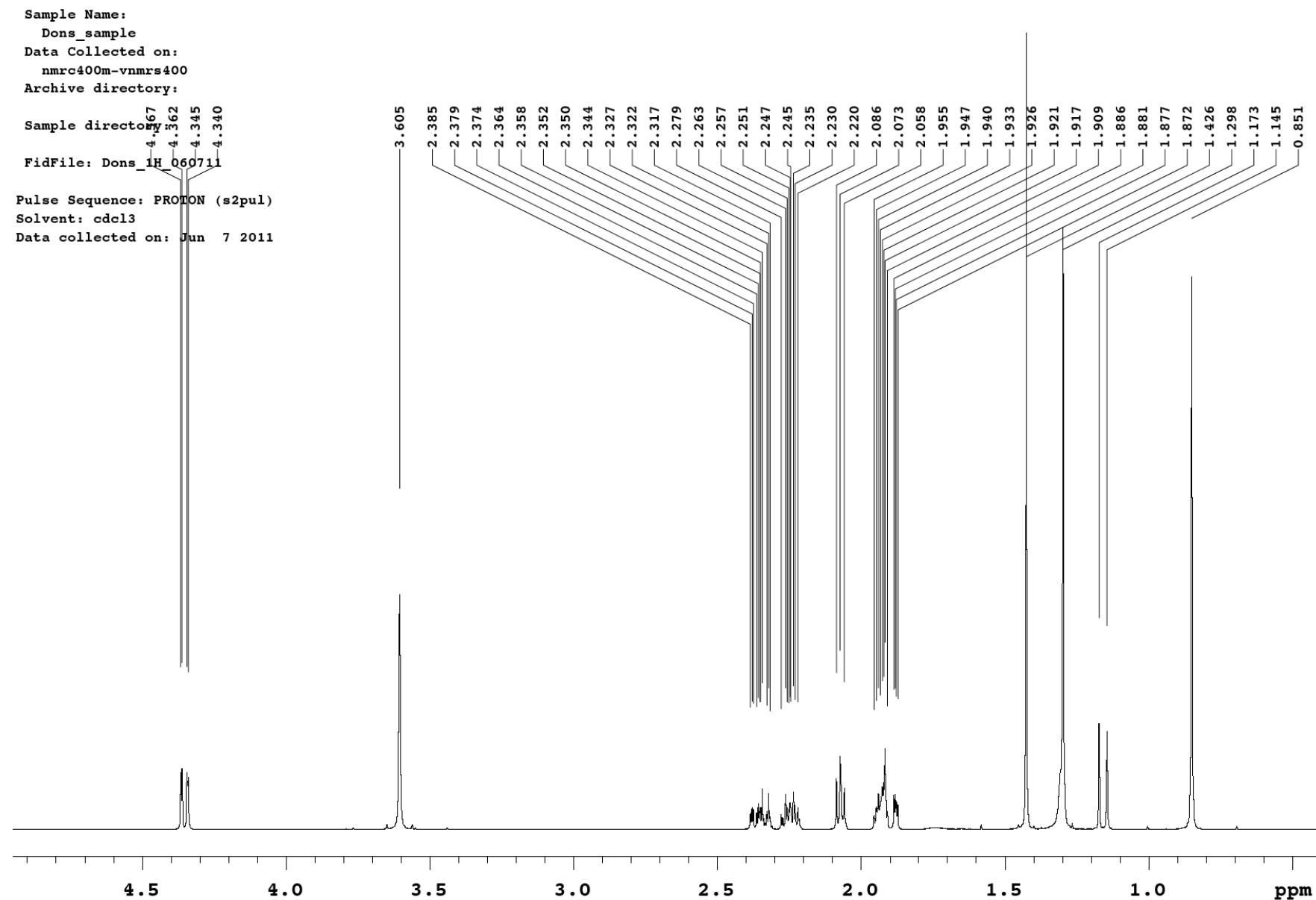
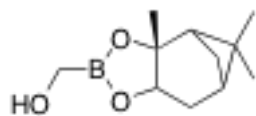
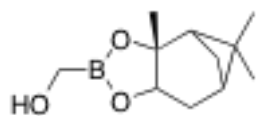
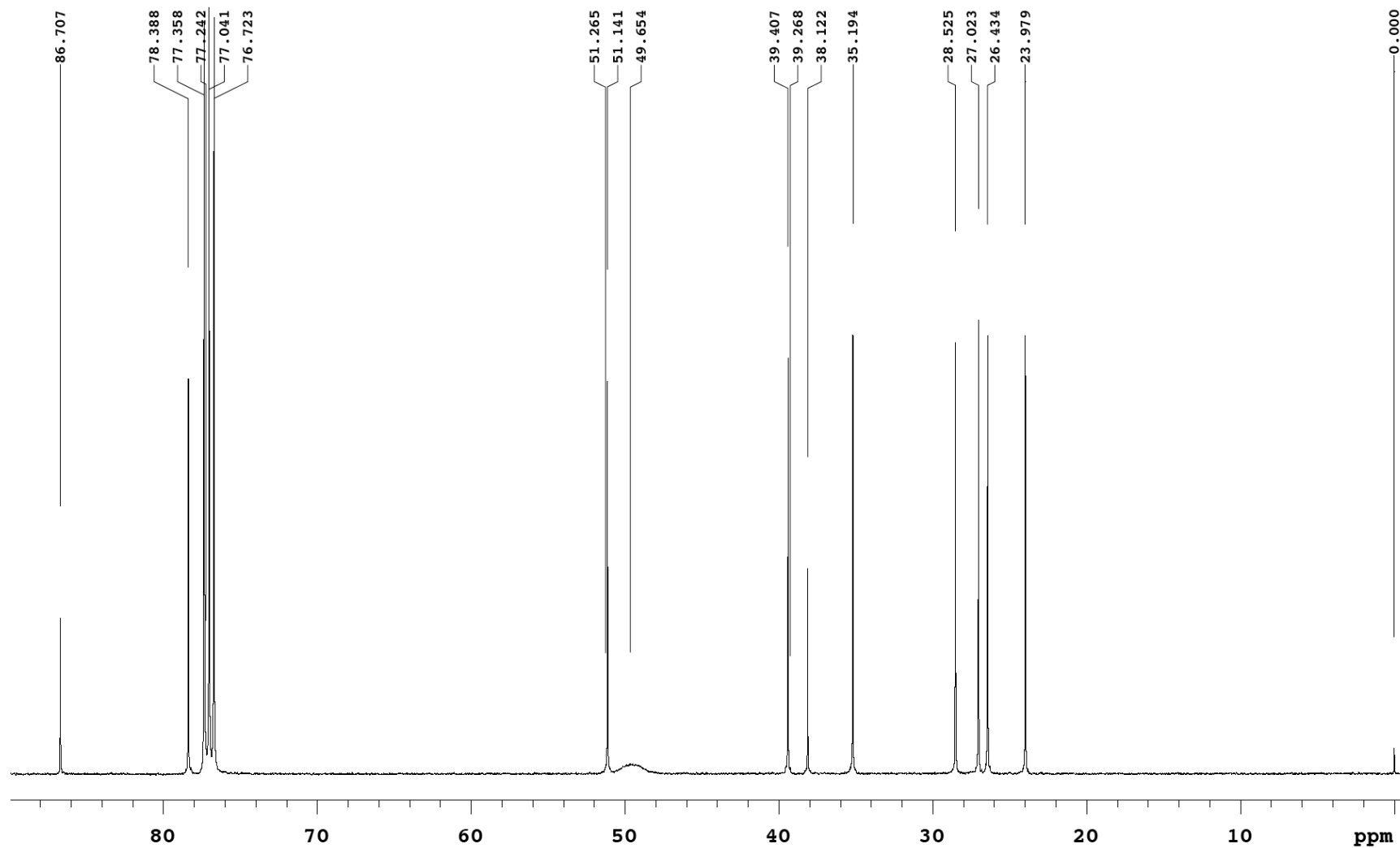


**Accessory Publication: Donald S. Matteson, Preparation and Properties of (Hydroxymethyl)boronic Acid and its Pinanediol Ester**  
Selected  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra





**12** **12:** 400-MHz <sup>1</sup>H-NMR (CDCl<sub>3</sub>)



**12** **100-MHz** <sup>13</sup>C-NMR (CDCl<sub>3</sub>)

STANDARD 1H OBSERVE - profile

Sample Name:

HOCH2BODimer44mg

Data Collected on:

nmrc400m-vnmrs400

Archive directory:

/home/walkup/vnmrsys/data/matteson

Sample directory:

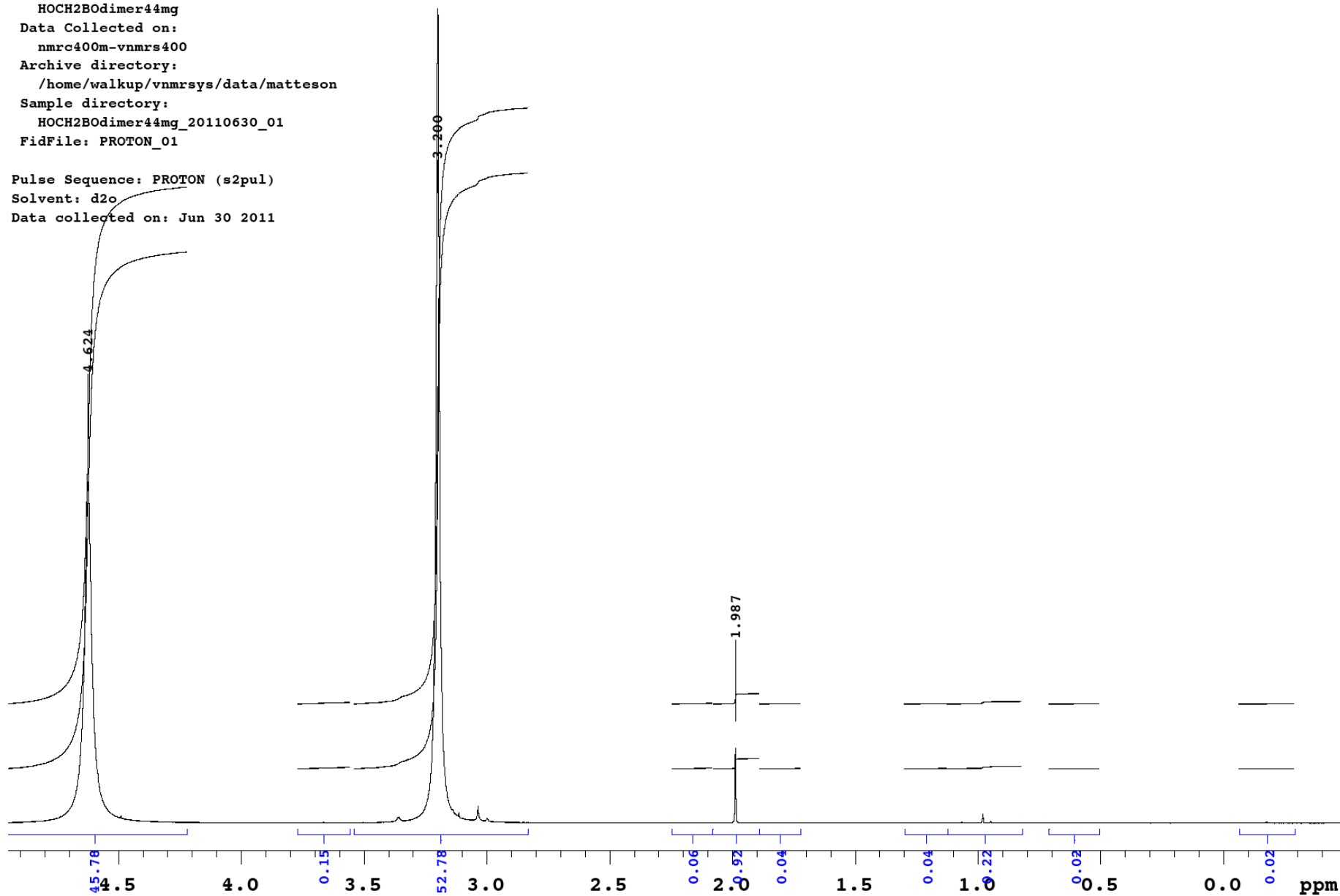
HOCH2BODimer44mg\_20110630\_01

FidFile: PROTON\_01

Pulse Sequence: PROTON (s2pul)

Solvent: d2o

Data collected on: Jun 30 2011



400-MHz <sup>1</sup>H-NMR (D<sub>2</sub>O)

HOCH2BODimer44mg

Sample Name:

HOCH2BODimer44mg

Data Collected on:

nmrc400m-vnmrs400

Archive directory:

/home/walkup/vnmrsys/data/matteson

Sample directory:

HOCH2BODimer44mg\_20110630\_01

FidFile: CARBON\_01

Pulse Sequence: CARBON (s2pul)

Solvent: d2o

Data collected on: Jun 30 2011

Temp. 22.0 C / 295.1 K

Operator: matteson

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.285 sec

Width 25510.2 Hz

2048 repetitions

OBSERVE C13, 100.5163269 MHz

DECOUPLE H1, 399.7484569 MHz

Power 36 dB

continuously on

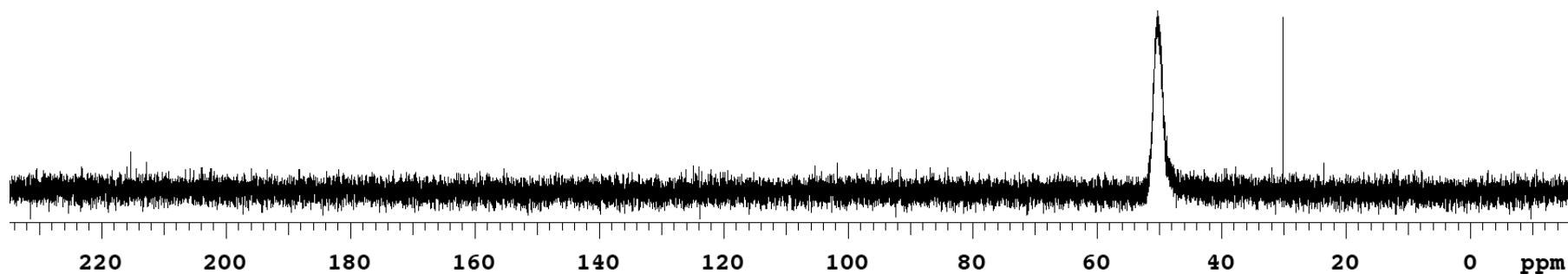
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

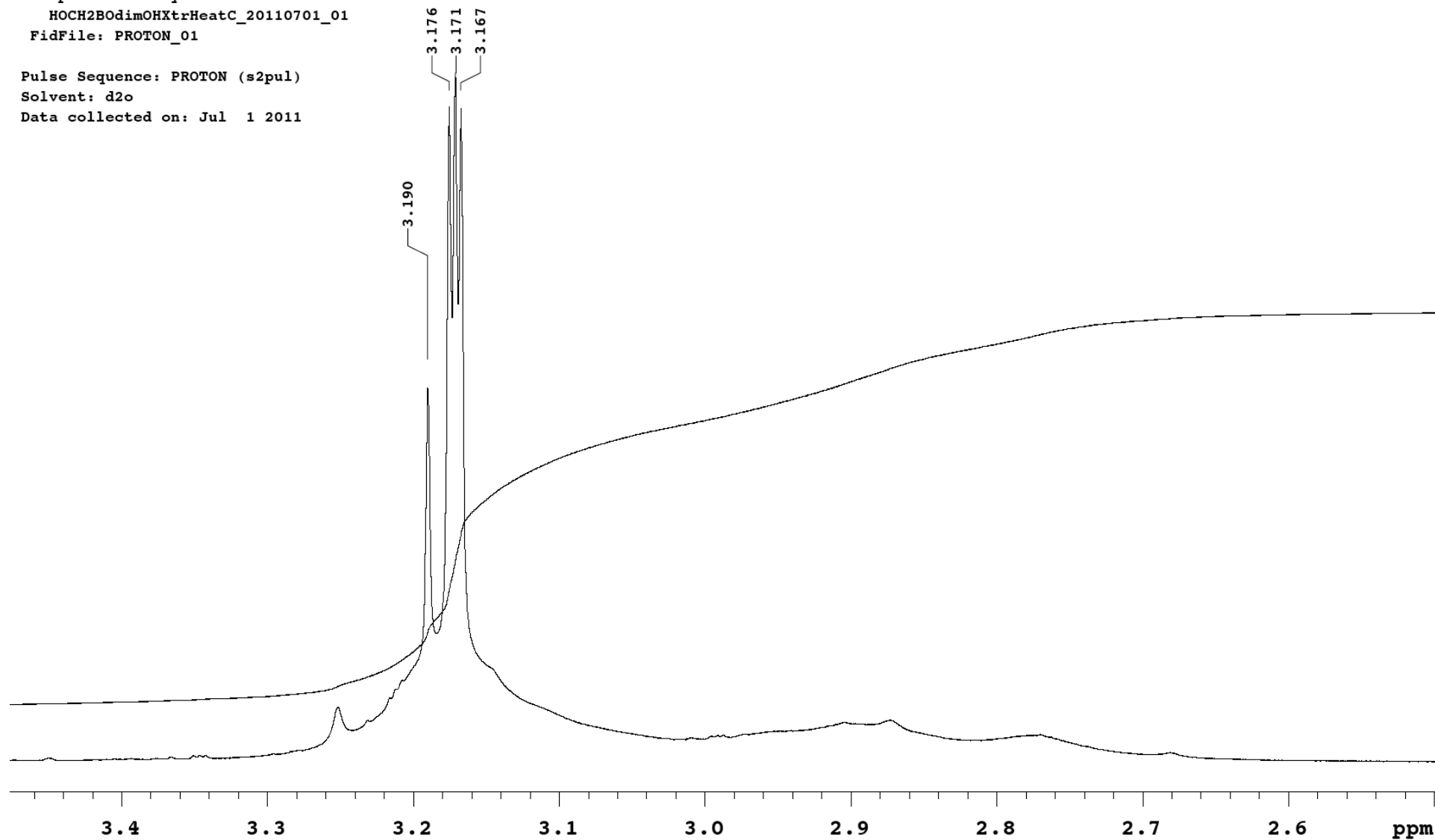
FT size 65536

Total time 1 hr, 17 min



Sample Name:  
HOCH2BODimOHXtrHeatC  
Data Collected on:  
nmrc400m-vnmrs400  
Archive directory:  
/home/walkup/vnmrsys/data/matteson  
Sample directory:  
HOCH2BODimOHXtrHeatC\_20110701\_01  
FidFile: PROTON\_01

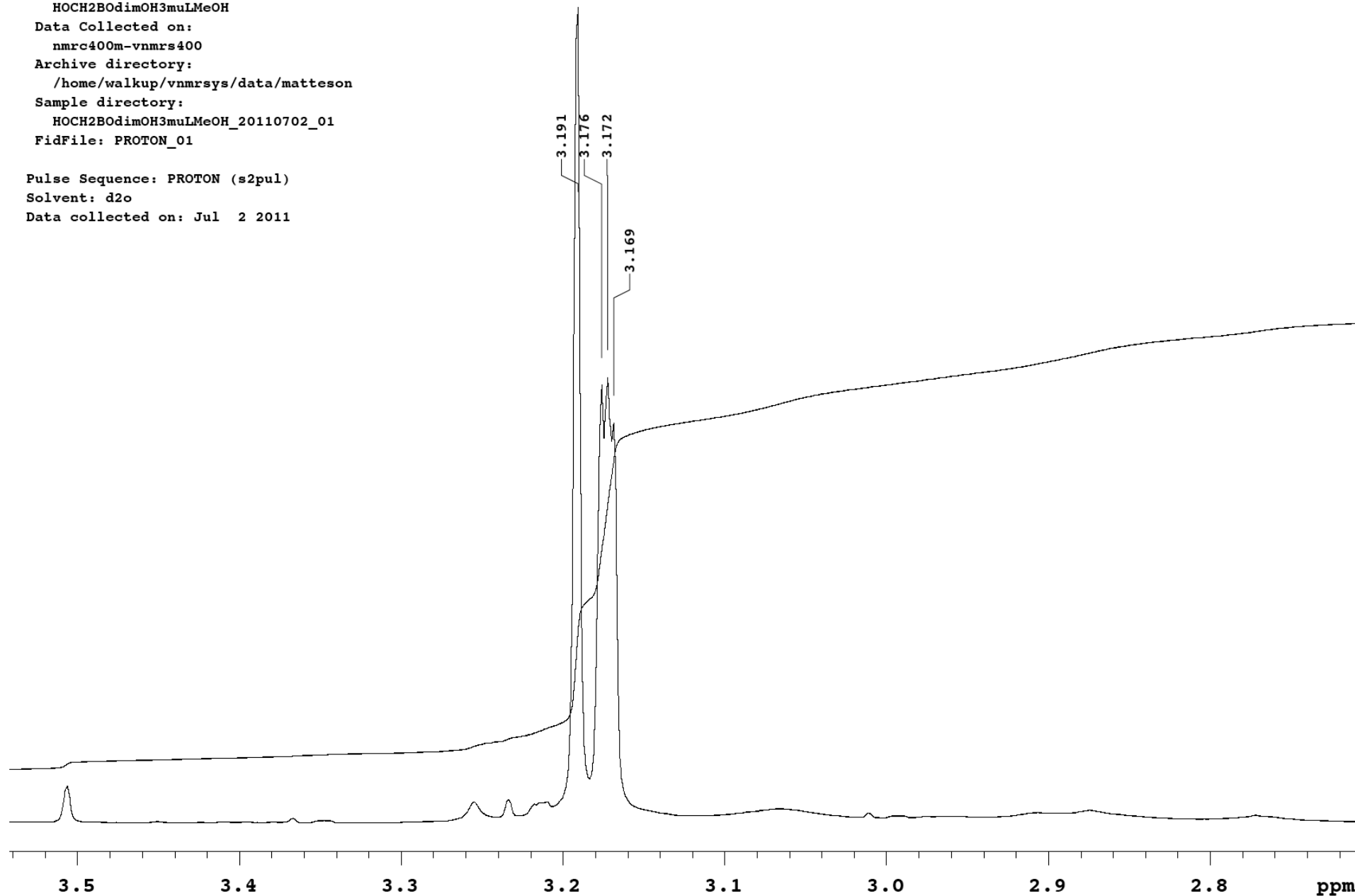
Pulse Sequence: PROTON (s2pul)  
Solvent: d2o  
Data collected on: Jul 1 2011



Detail of the 400-MHz  $^1\text{H}$ -NMR spectrum of DCH<sub>2</sub>OD from **10-d** after 4h in boiling water bath (no added methanol)

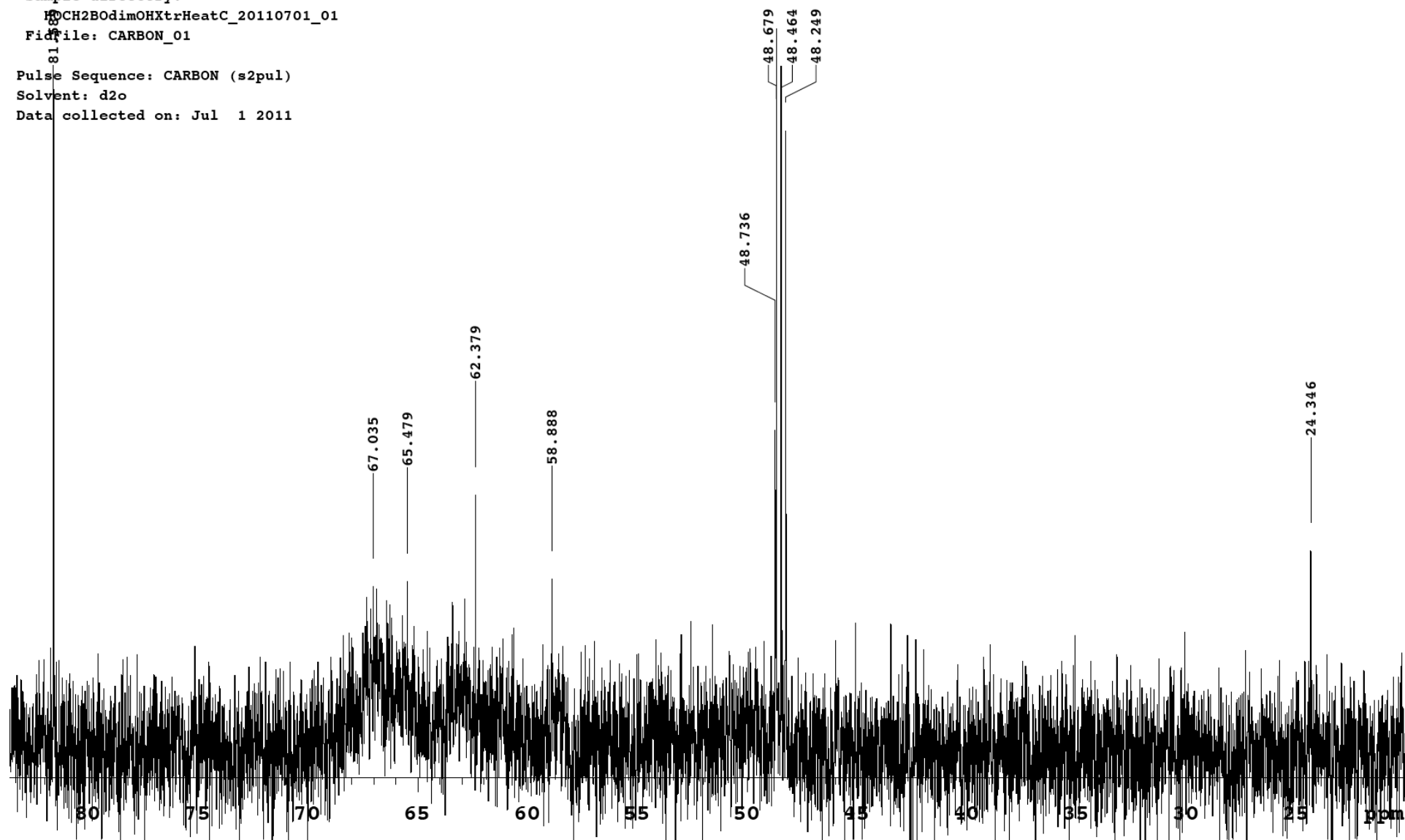
Sample Name:  
HOCH2BODimOH3muLMeOH  
Data Collected on:  
nmrc400m-vnmrs400  
Archive directory:  
/home/walkup/vnmrsys/data/matteson  
Sample directory:  
HOCH2BODimOH3muLMeOH\_20110702\_01  
FidFile: PROTON\_01

Pulse Sequence: PROTON (s2pul)  
Solvent: d2o  
Data collected on: Jul 2 2011



Detail of the 400-MHz  $^1\text{H}$ -NMR spectrum of  $\text{DCH}_2\text{OD}$  from **10-d** after 6h in boiling water bath and addition of 3  $\mu\text{L}$  of methanol

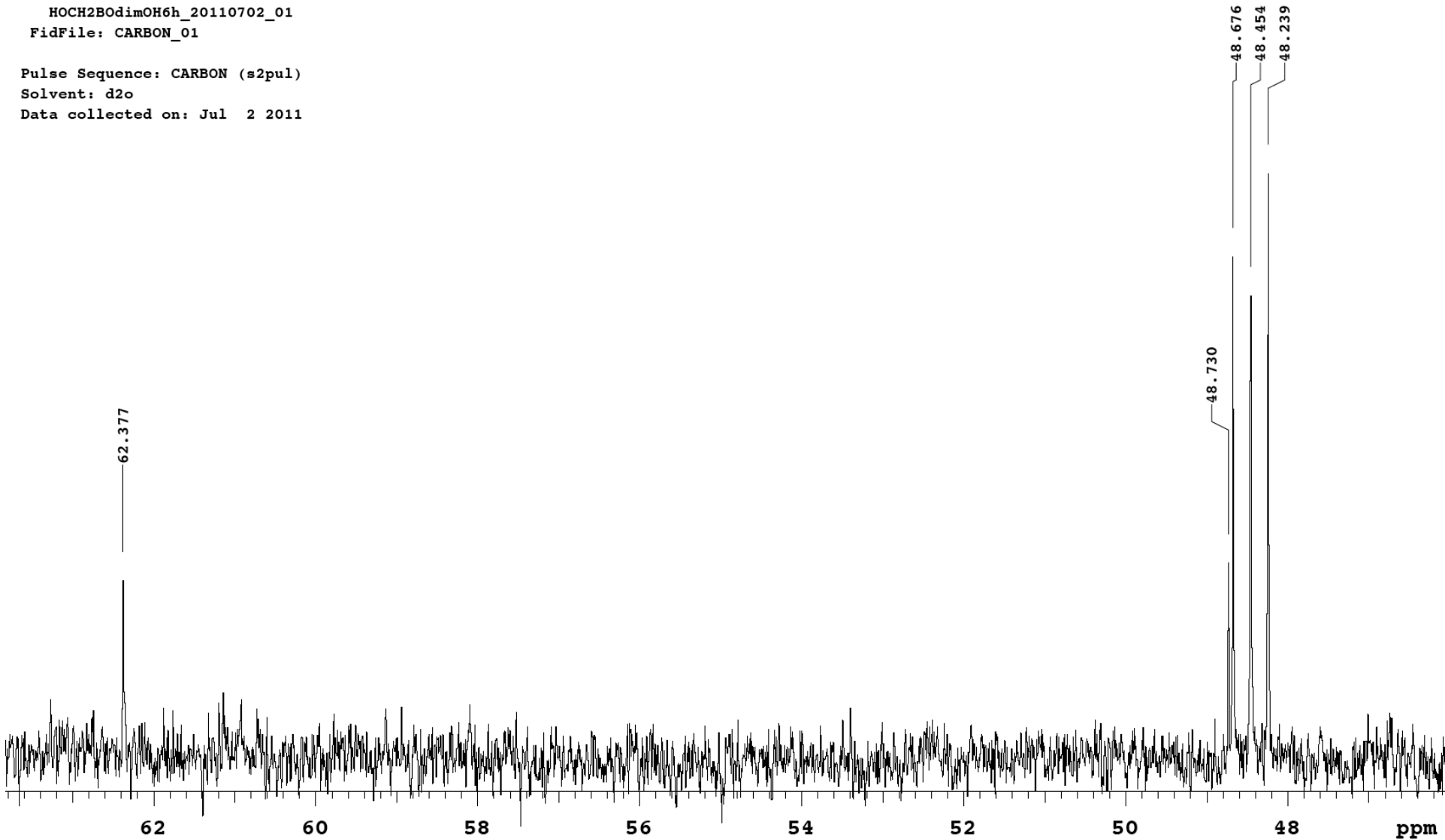
Sample Name:  
HOCH2BODimOHXtrHeatC  
Data Collected on:  
nmrc400m-vnmrs400  
Archive directory:  
/home/walkup/vnmrsys/data/matteson  
Sample directory:  
HOCH2BODimOHXtrHeatC\_20110701\_01  
FidFile: CARBON\_01  
Pulse Sequence: CARBON (s2pul)  
Solvent: d2o  
Data collected on: Jul 1 2011



100-MHz  $^{13}\text{C}$ -NMR spectrum of **10-d** and derived  $\text{DCH}_2\text{OD}$  after 4h in boiling water bath (no added methanol)

Sample Name:  
HOCH2BODimOH6h  
Data Collected on:  
nmrc400m-vnmrs400  
Archive directory:  
/home/walkup/vnmrsys/data/matteson  
Sample directory:  
HOCH2BODimOH6h\_20110702\_01  
FidFile: CARBON\_01

Pulse Sequence: CARBON (s2pul)  
Solvent: d2o  
Data collected on: Jul 2 2011



Detail of the 100-MHz <sup>13</sup>C-NMR spectrum of DCH<sub>2</sub>OD from **10-d** after 6h in boiling water bath (no added methanol)

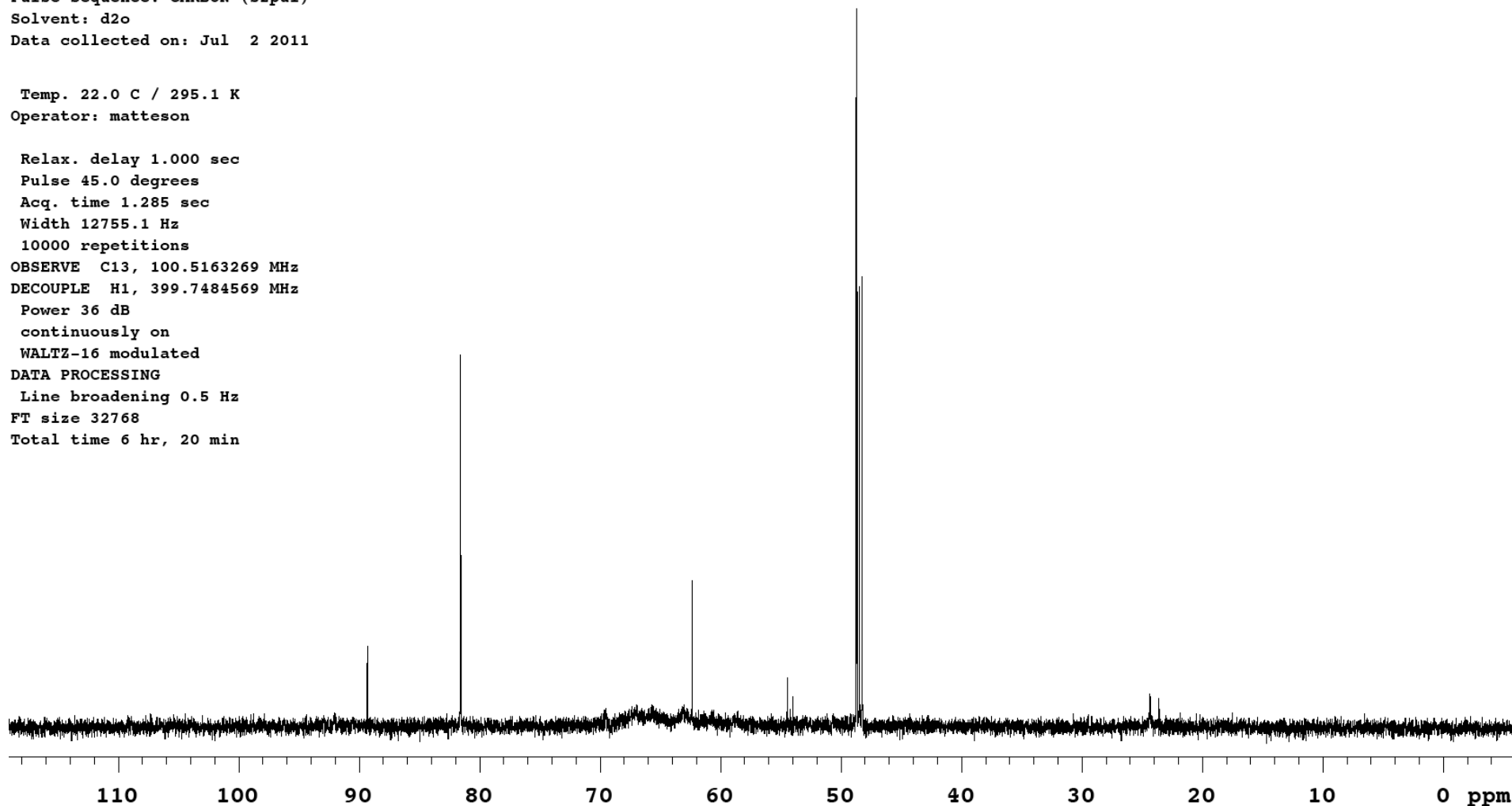


Sample Name:  
HOCH2BODimOH3muLMeOH  
Data Collected on:  
nmrc400m-vnmrs400  
Archive directory:  
/home/walkup/vnmrsys/data/matteson  
Sample directory:  
HOCH2BODimOH3muLMeOH\_20110702\_01  
FidFile: CARBON\_01

Pulse Sequence: CARBON (s2pul)  
Solvent: d2o  
Data collected on: Jul 2 2011

Temp. 22.0 C / 295.1 K  
Operator: matteson

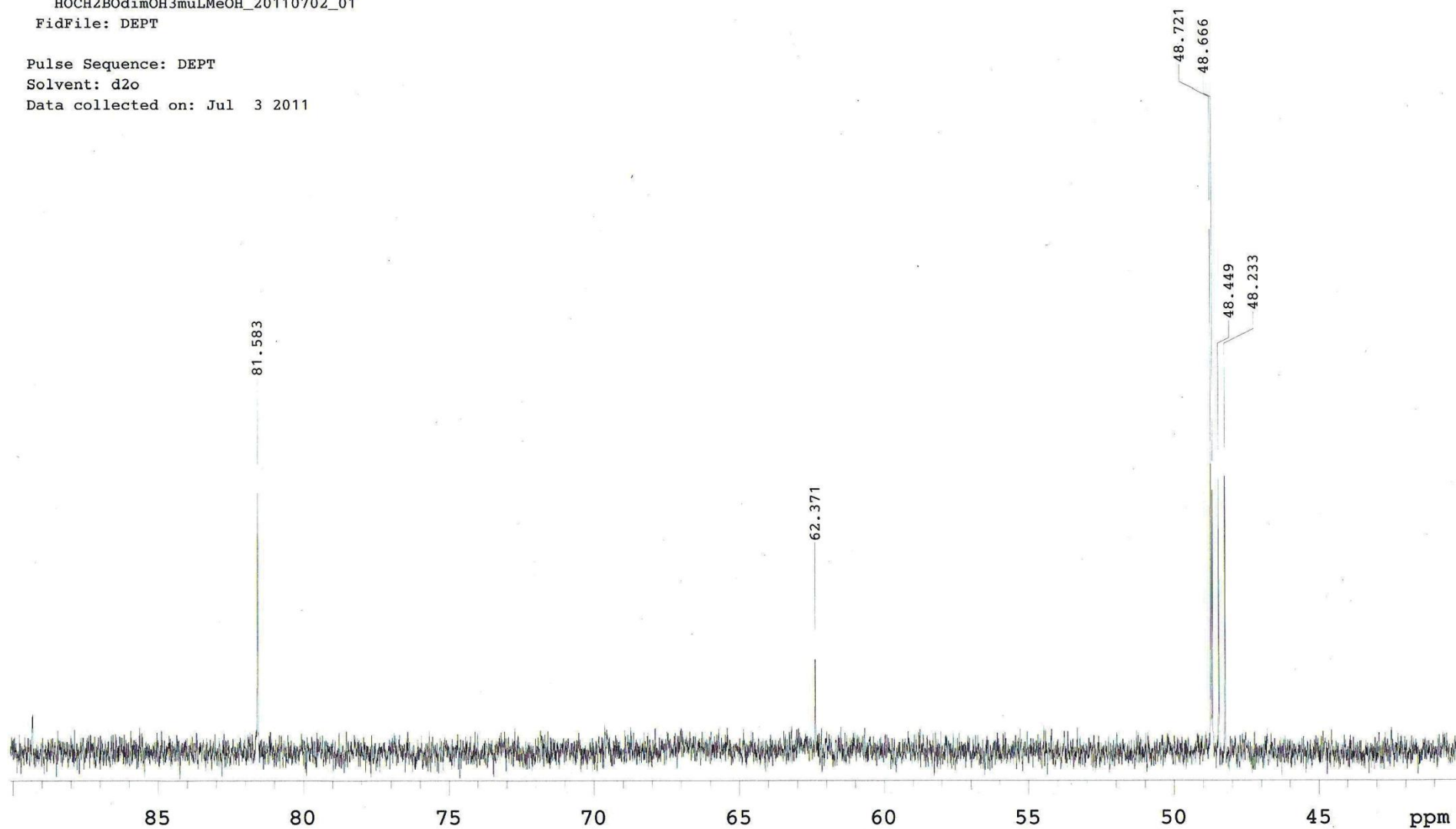
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.285 sec  
Width 12755.1 Hz  
10000 repetitions  
OBSERVE C13, 100.5163269 MHz  
DECOUPLE H1, 399.7484569 MHz  
Power 36 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 0.5 Hz  
FT size 32768  
Total time 6 hr, 20 min



100-MHz <sup>13</sup>C-NMR spectrum of **10-d** and derived DCH<sub>2</sub>OD after 6h in boiling water bath and addition of 3 μL of methanol

Sample Name:  
HOCH2BODimOH3muLMeOH  
Data Collected on:  
nmrc400m-vnmrs400  
Archive directory:  
/home/walkup/vnmrsys/data/matteson  
Sample directory:  
HOCH2BODimOH3muLMeOH\_20110702\_01  
FidFile: DEPT

Pulse Sequence: DEPT  
Solvent: d2o  
Data collected on: Jul 3 2011



Expanded image of 100-MHz  $^{13}\text{C}$ -NMR spectrum of **10-d** and derived  $\text{DCH}_2\text{OD}$  after 6h in boiling water bath and addition of 3  $\mu\text{L}$  of methanol