

Supporting Information for

**Macrocyclic Trichothecenes from *Myrothecium roridum* Strain M10 with Motility Inhibitory and Zoosporicidal Activities against *Phytophthora nicotianae***

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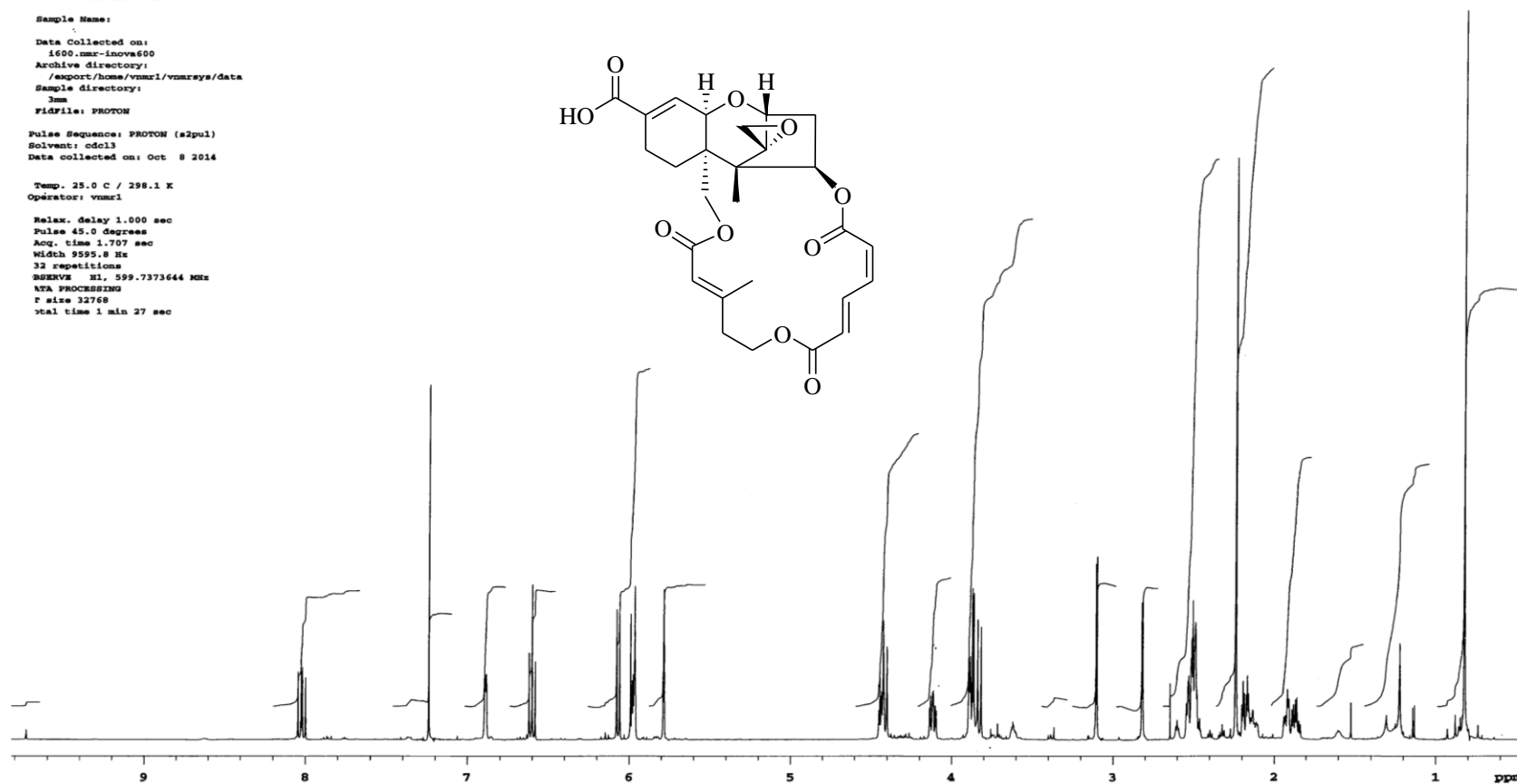
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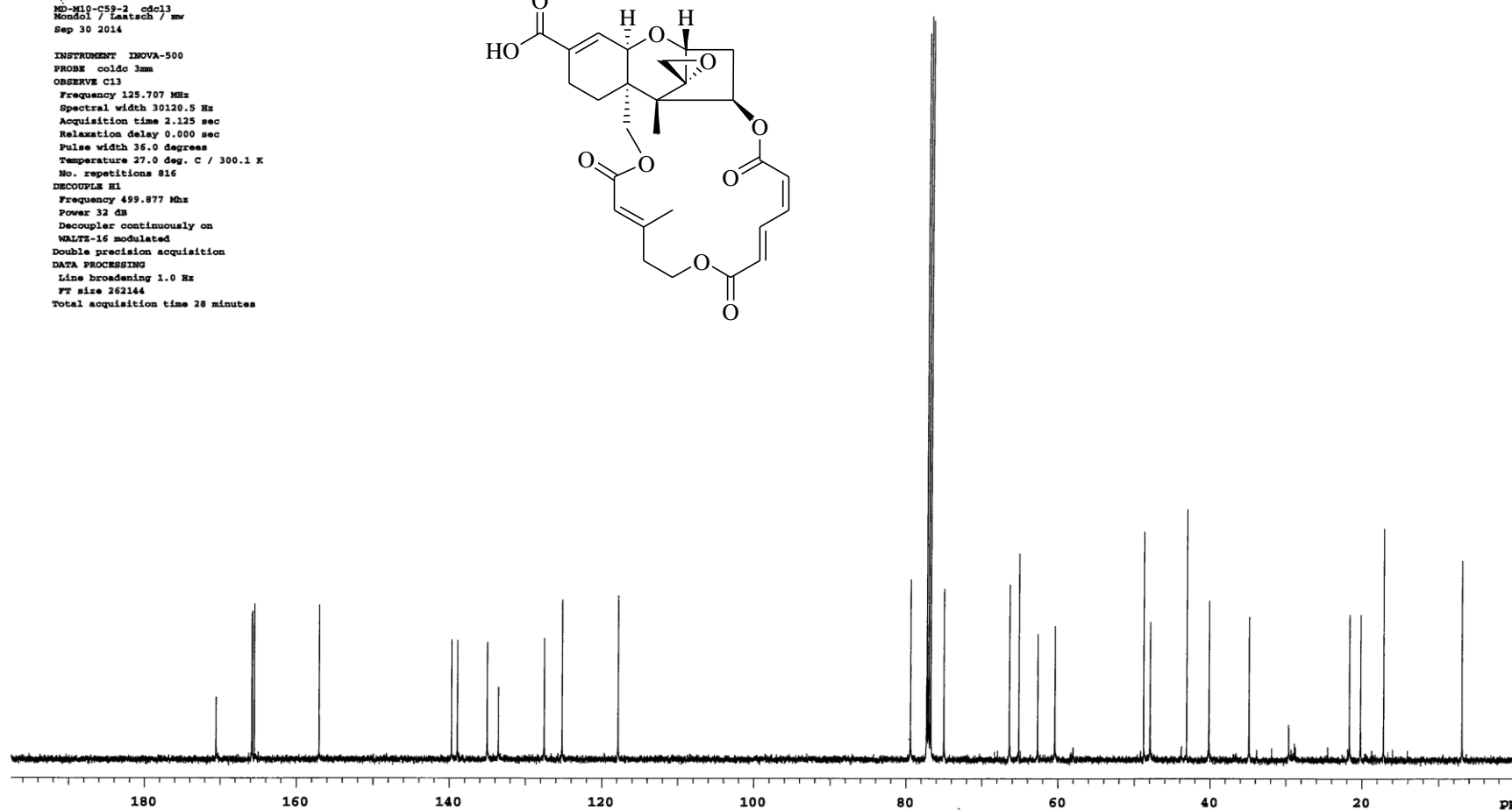
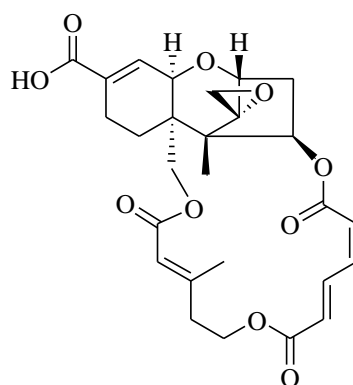
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MD-M10-C59-2 cdcl3  
 Mondol / Laatsch / CS  
 Sample Name:  
 Data Collected on:  
 1600.mmr-inova600  
 Archive directory:  
 /export/home/vnmr1/vnmrsys/data  
 Sample directory:  
 3mm  
 Fidfile: PROTON  
 Pulse Sequence: PROTON (s2pul)  
 Solvent: cdcl3  
 Data collected on: Oct 8 2014  
 Temp. 25.0 C / 298.1 K  
 Operator: vnmr1  
 Relax. delay 1.000 sec  
 Pulse 45.0 degrees  
 Acq. time 1.707 sec  
 Width 9595.8 Hz  
 32 repetitions  
 SFOVX H1, 599.7373644 MHz  
 NTA PROCESSING  
 F size 32768  
 Total time 1 min 27 sec



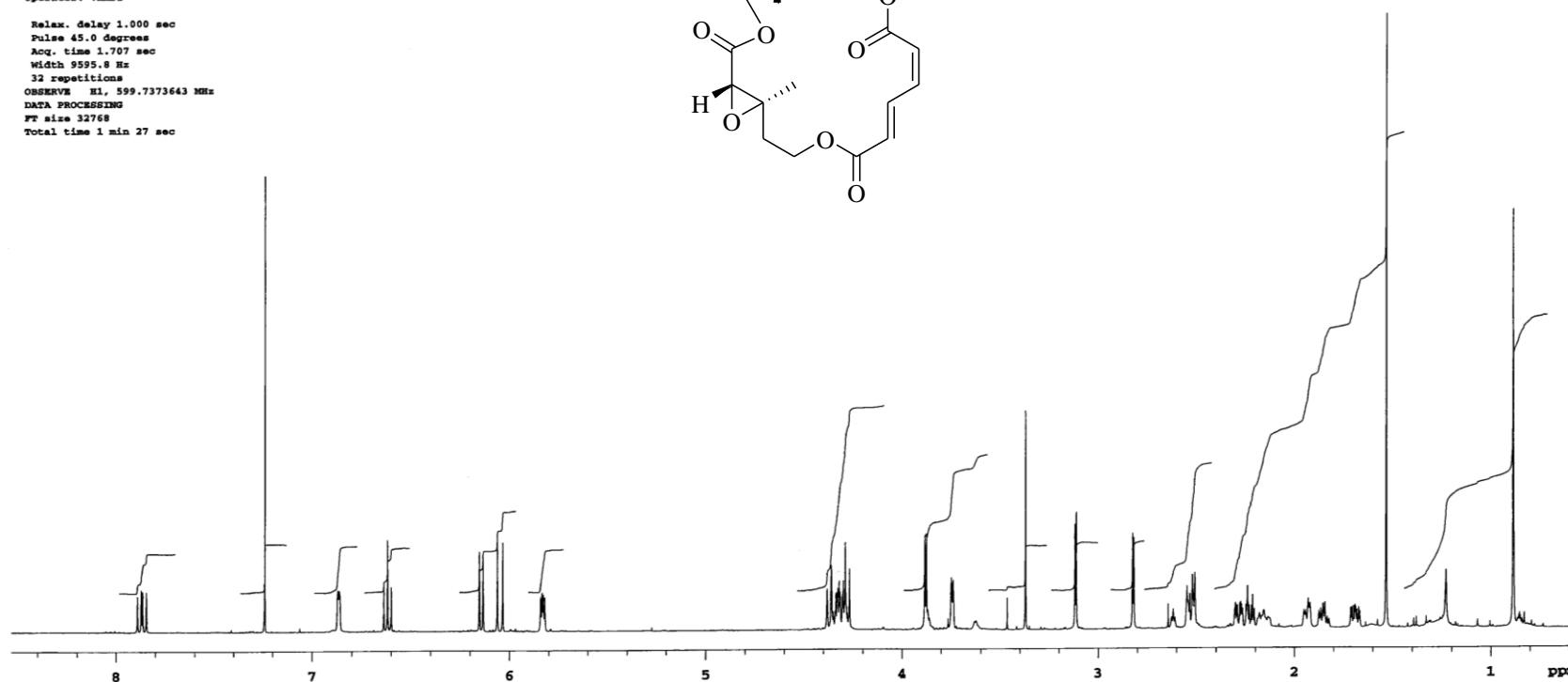
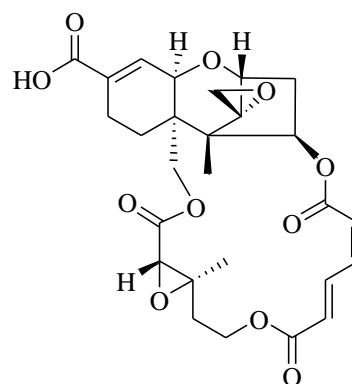
**Figure S1.**  $^1\text{H}$  NMR spectrum of **1** recorded in  $\text{CDCl}_3$  at 600 MHz.

MD-M10-C59-2 cdcl3  
 Mondol / Leatsch / mw  
 Sep 30 2014  
 INSTRUMENT INOVA-500  
 PROBE coldc 3mm  
 OBSERVE C13  
 Frequency 125.707 MHz  
 Spectral width 30120.5 Hz  
 Acquisition time 2.125 sec  
 Relaxation delay 0.000 sec  
 Pulse width 16.0 degrees  
 Temperature 27.0 deg. C / 300.1 K  
 No. repetitions 816  
 DECOUPLE H1  
 Frequency 499.877 MHz  
 Power 32 dB  
 Decoupler continuously on  
 WALTZ-16 modulated  
 Double precision acquisition  
 DATA PROCESSING  
 Line broadening 1.0 Hz  
 FT size 262144  
 Total acquisition time 28 minutes



**Figure S2.** <sup>13</sup>C NMR spectrum of **1** recorded in CDCl<sub>3</sub> at 125 MHz.

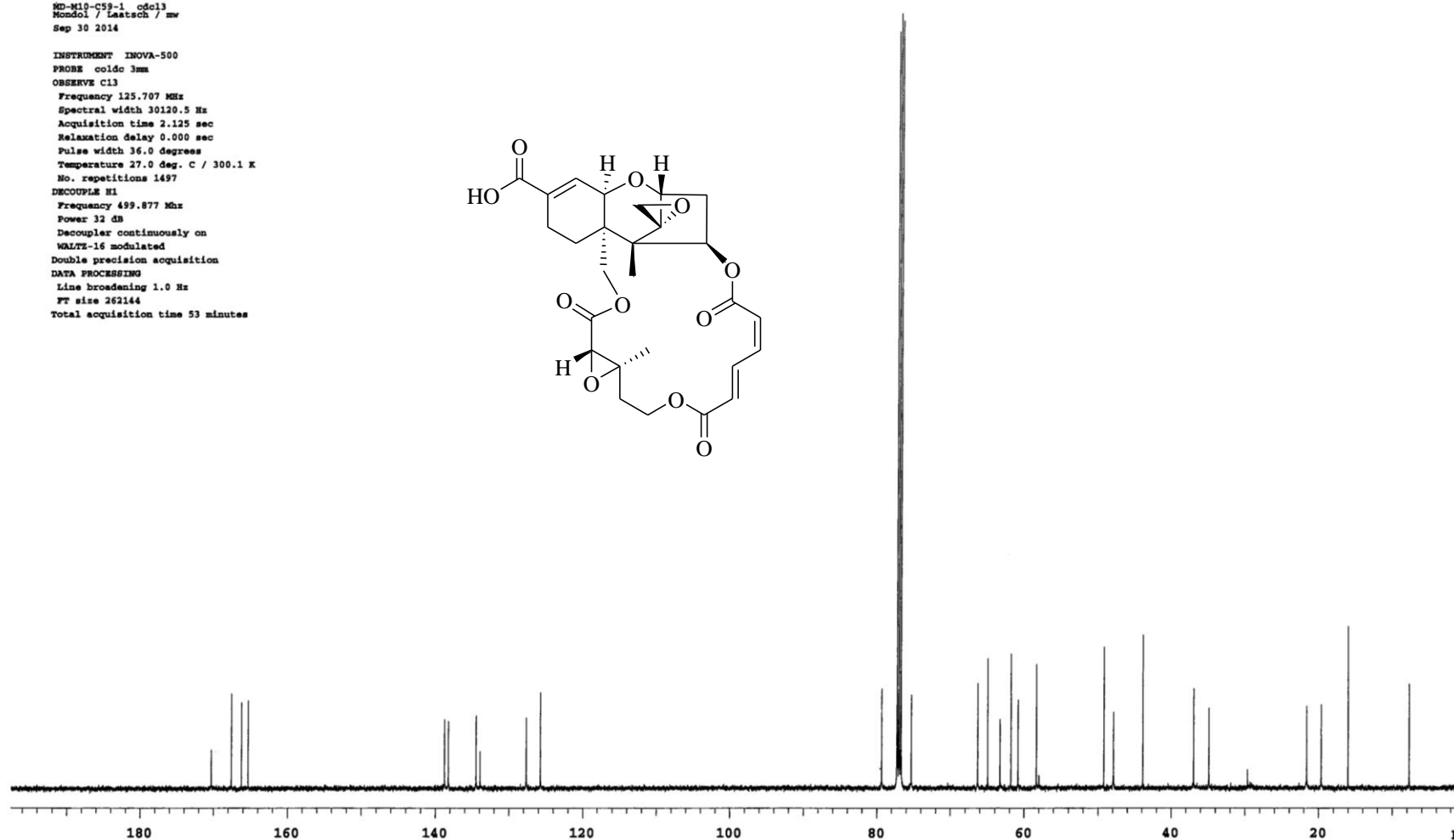
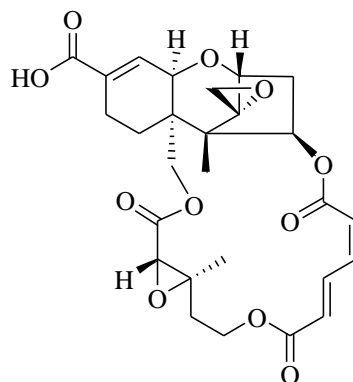
MD-M10-C59-1 cdcl3  
Mondol / Laatsch / CS  
  
Sample Name:  
  
Data collected on:  
1600.nmr-inova600  
Archive directory:  
  
Sample directory:  
  
Fidfile: PROTON  
  
Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Oct 10 2014  
  
Temp. 25.0 C / 298.1 K  
Operator: vmmr1  
  
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.707 sec  
Width 9595.8 Hz  
32 repetitions  
OBSERVE H1, 599.7373643 MHz  
DATA PROCESSING  
FT size 32768  
Total time 1 min 27 sec



**Figure S3.** <sup>1</sup>H NMR spectrum of **6** recorded in CDCl<sub>3</sub> at 600 MHz.

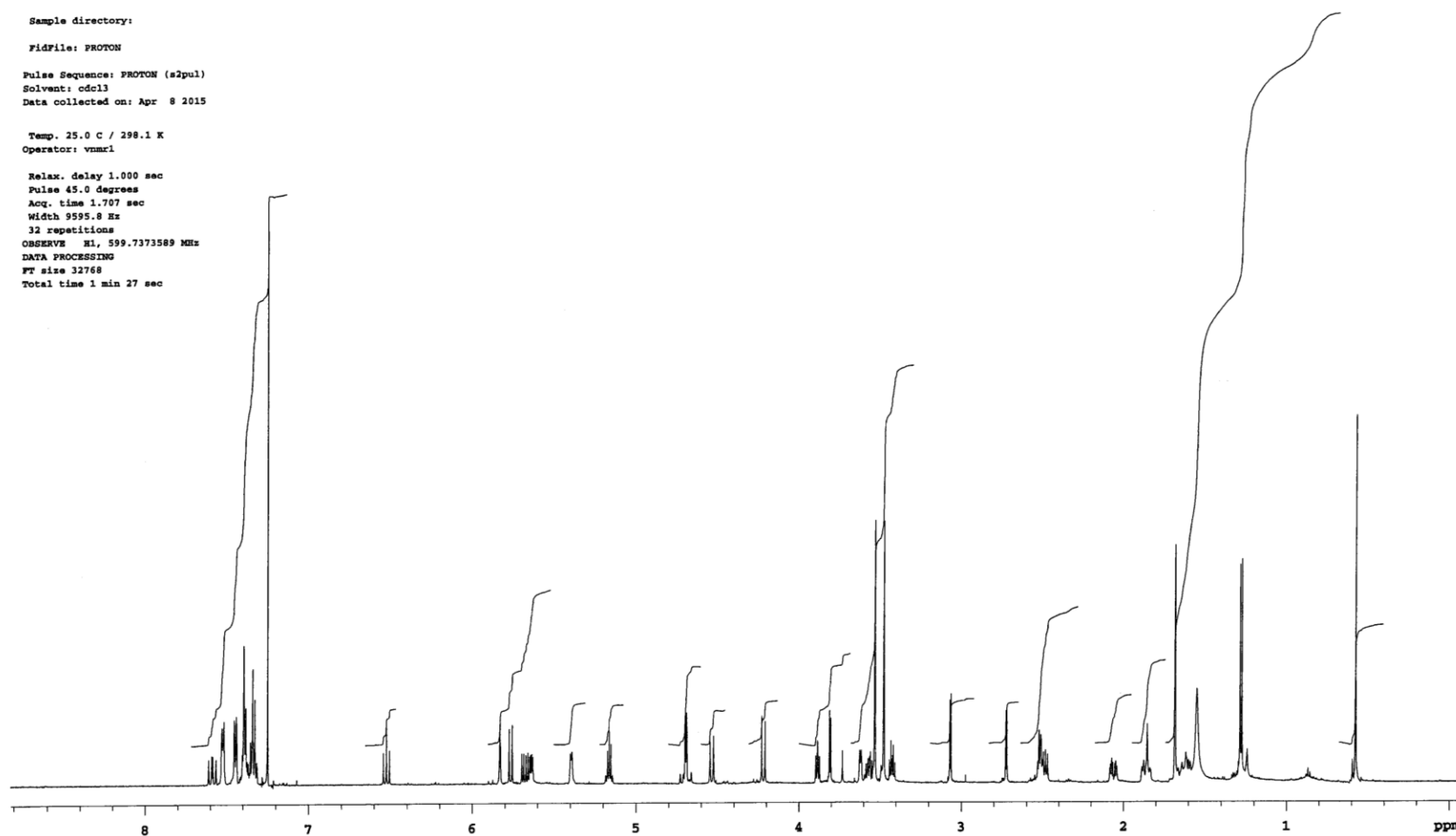
NO-M10-C59-1 cdcl3  
Mondol / Laatsch / mw  
Sep 30 2014

INSTRUMENT INOVA-500  
PROBE coldc 3mm  
OBSERVE C13  
Frequency 125.707 MHz  
Spectral width 30120.5 Hz  
Acquisition time 2.125 sec  
Relaxation delay 0.000 sec  
Pulse width 36.0 degrees  
Temperature 27.0 deg. C / 300.1 K  
No. repetitions 1497  
DECOUPLE H1  
Frequency 499.877 MHz  
Power 32 dB  
Decoupler continuously on  
WALTZ-16 modulated  
Double precision acquisition  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 262144  
Total acquisition time 53 minutes

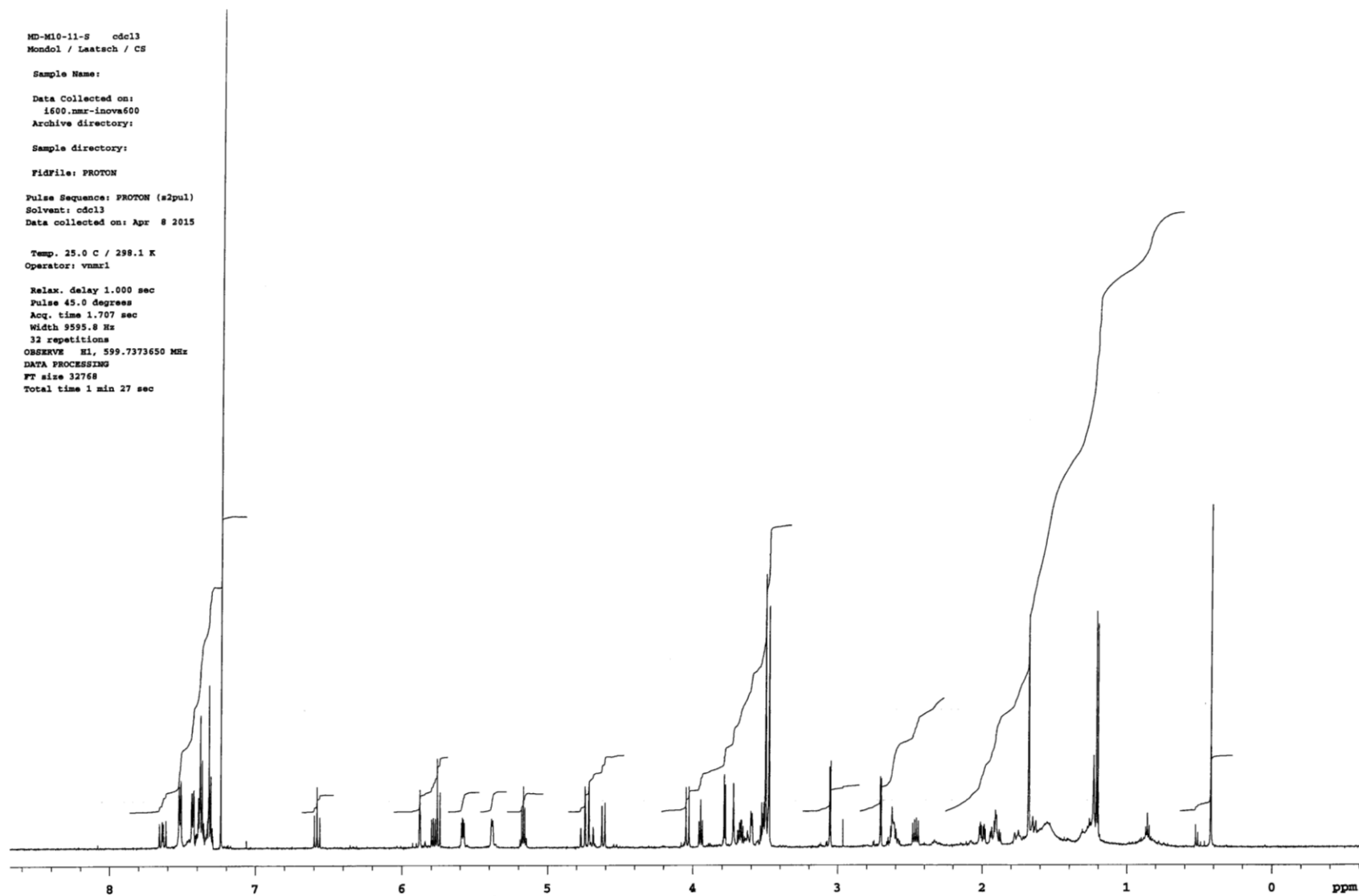


**Figure S4.**  $^{13}\text{C}$  NMR spectrum of **6** recorded in  $\text{CDCl}_3$  at 125 MHz.

MD-M10-11-R cdcl3  
Mondol / Laatsch / CS  
  
Sample Name:  
  
Data Collected on:  
1600.nmr-inova600  
Archive directory:  
  
Sample directory:  
  
FidFile: PROTON  
  
Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Apr 8 2015  
  
Temp. 25.0 C / 298.1 K  
Operator: vnmr1  
  
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.707 sec  
Width 9595.8 Hz  
32 repetitions  
OBSERVE H1, 599.7373589 MHz  
DATA PROCESSING  
FT size 32768  
Total time 1 min 27 sec

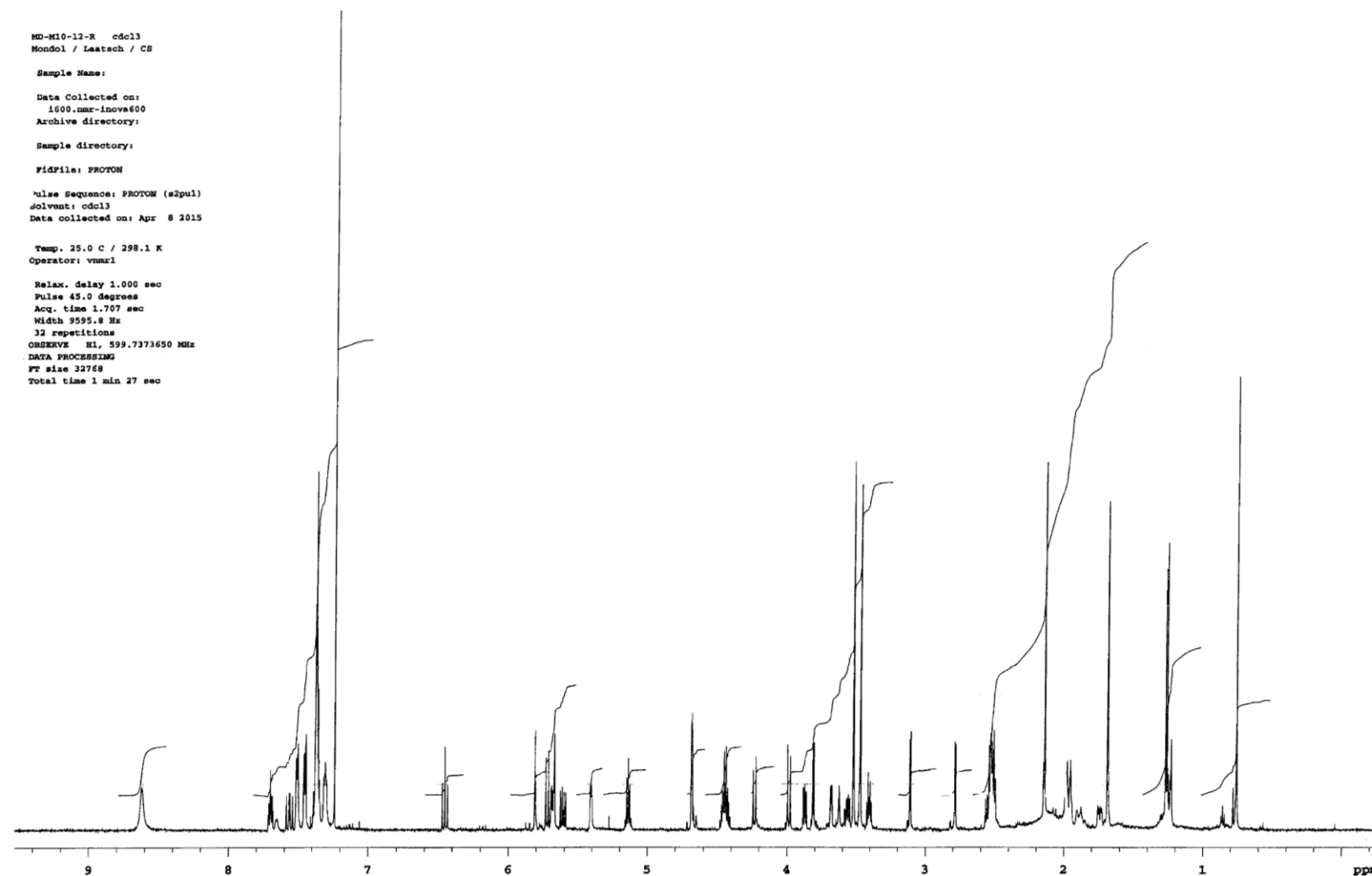


**Figure S5.** <sup>1</sup>H NMR spectrum of **10a** recorded in CDCl<sub>3</sub> at 600 MHz.



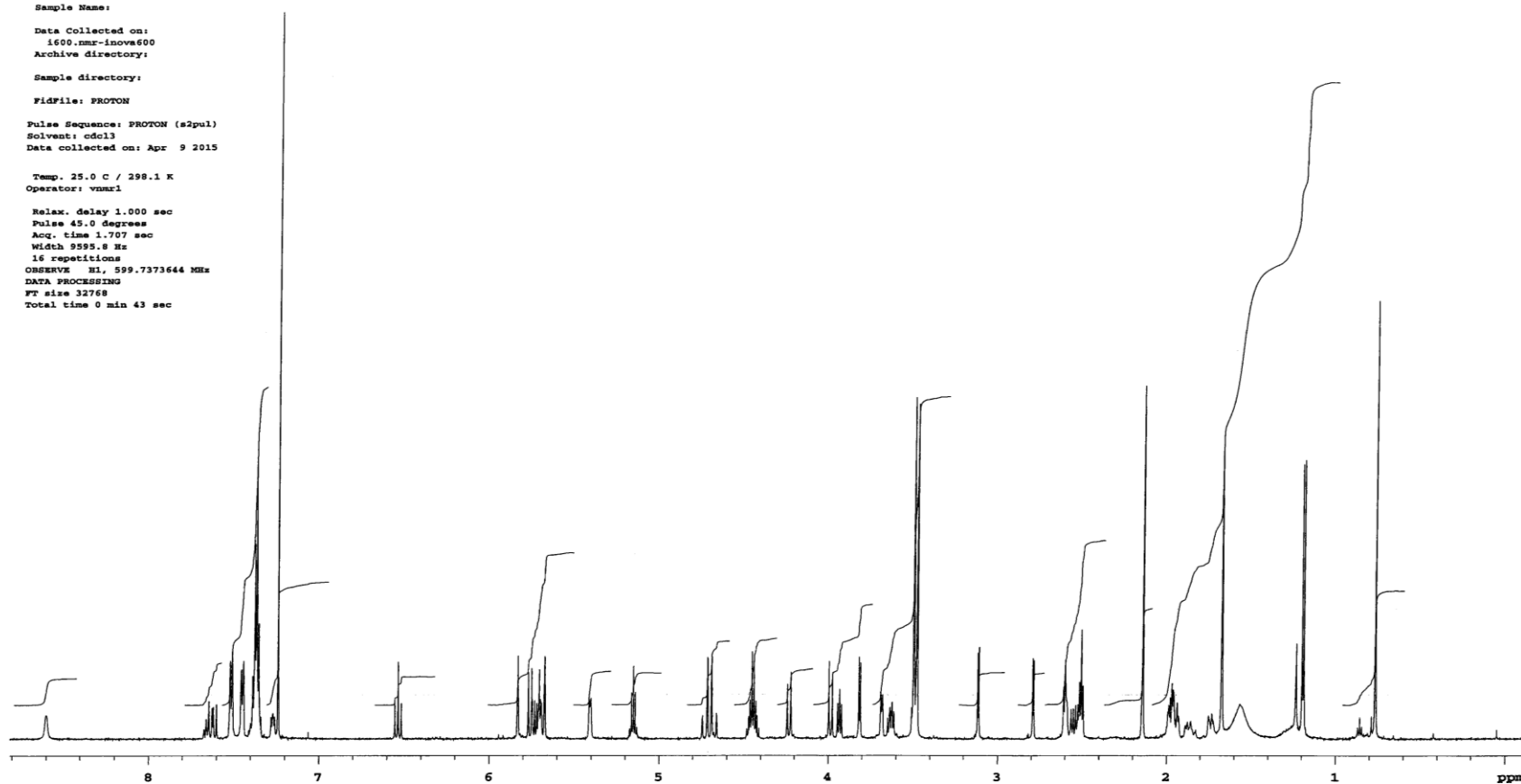
**Figure S6.** <sup>1</sup>H NMR spectrum of **10b** recorded in CDCl<sub>3</sub> at 600 MHz.





**Figure S7.**  $^1\text{H}$  NMR spectrum of **11a** recorded in  $\text{CDCl}_3$  at 600 MHz.

MD-M10-12-S cdc13  
Mondol / Laatsch / mw  
Sample Name:  
Data Collected on:  
1600.mw-inova600  
Archive directory:  
Sample directory:  
FidFile: PROTON  
Pulse Sequence: PROTON (s2pul)  
Solvent: cdc13  
Data collected on: Apr 9 2015  
Temp. 25.0 C / 298.1 K  
Operator: vnmr1  
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.707 sec  
Width 9595.8 Hz  
16 repetitions  
OBSERVE H1, 599.7373644 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min 43 sec

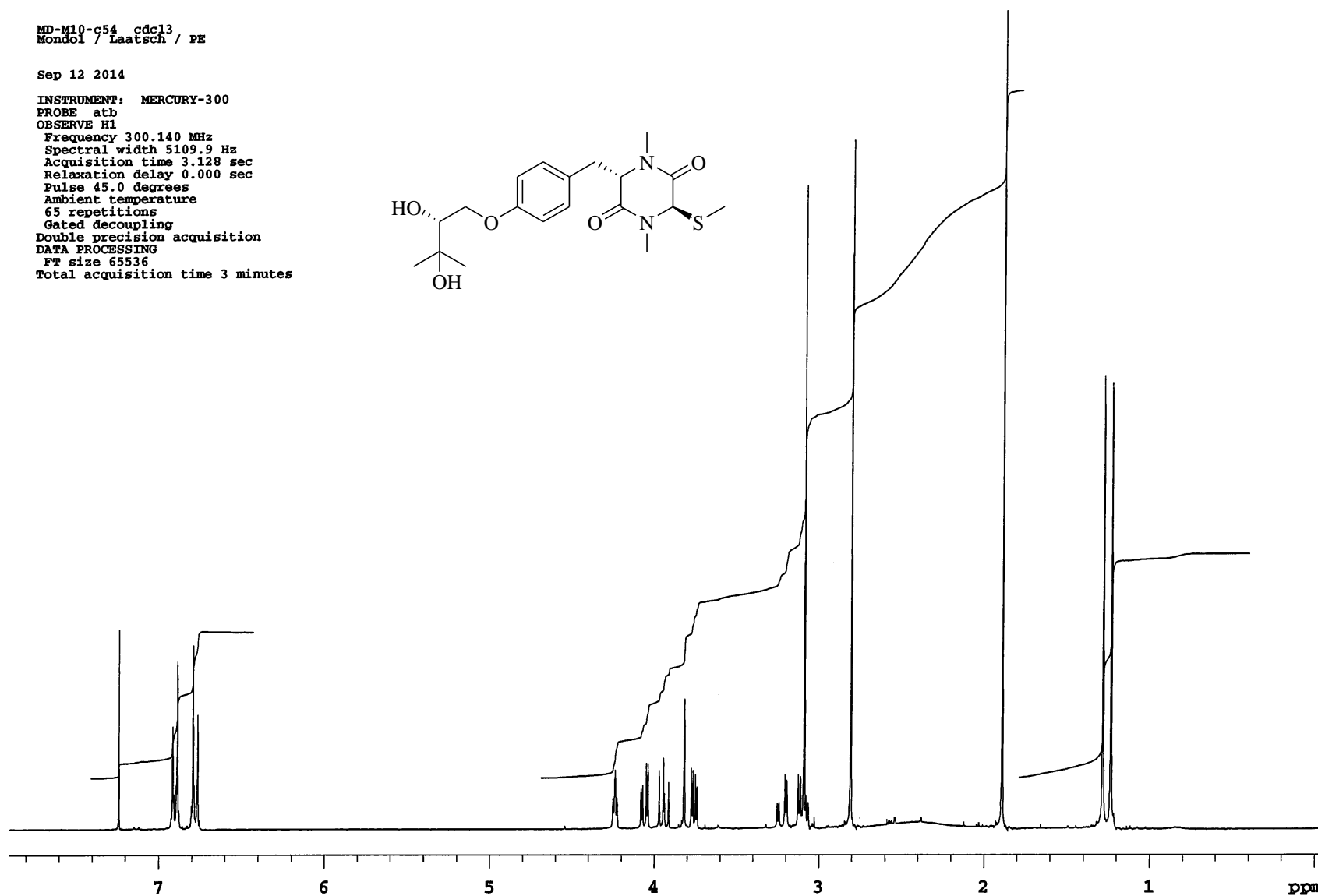
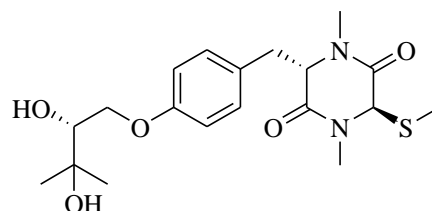


**Figure S8.**  $^1\text{H}$  NMR spectrum of **11b** recorded in  $\text{CDCl}_3$  at 600 MHz.

MD-M10-c54 cdc13  
Mondol / Laatsch / PE

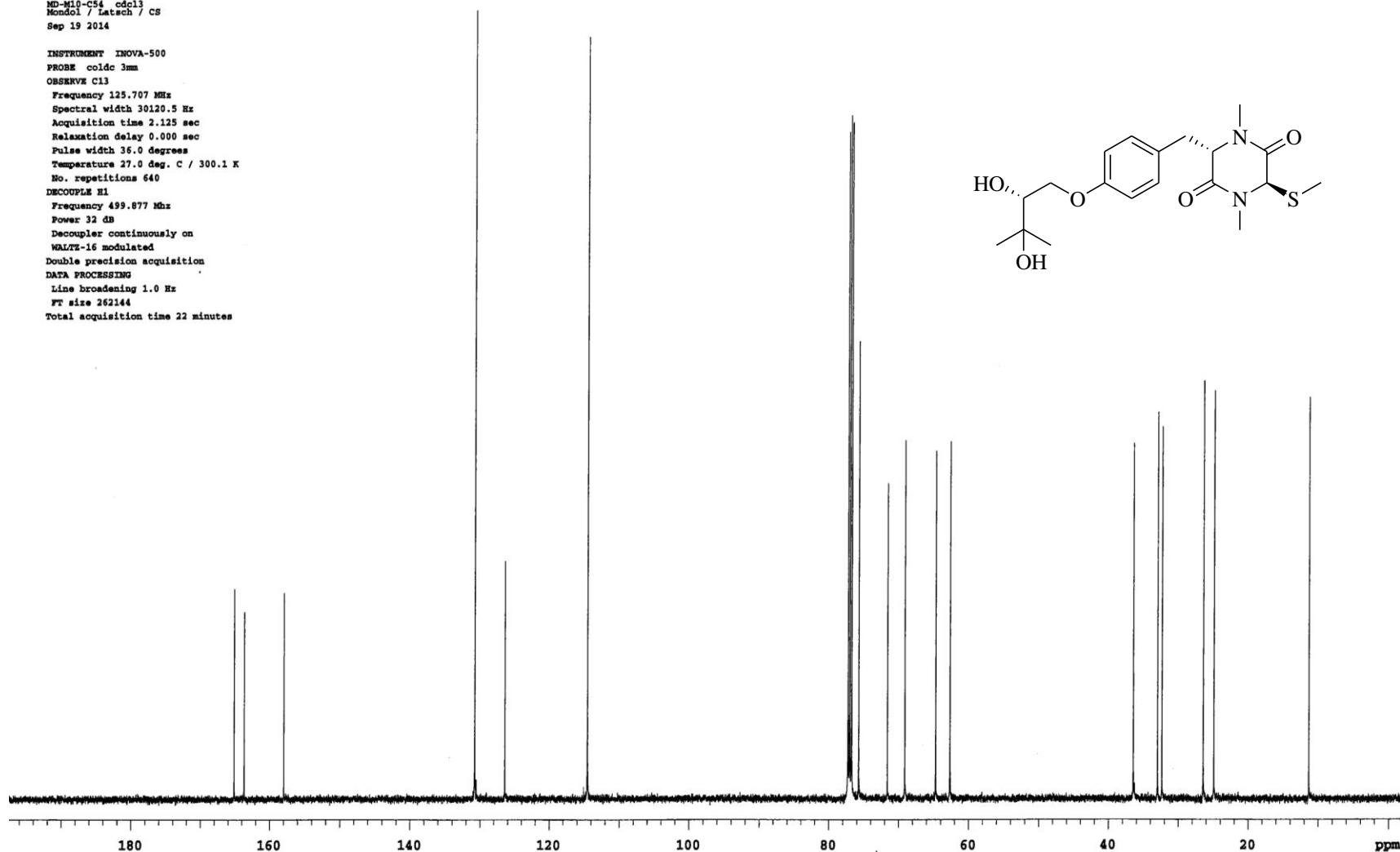
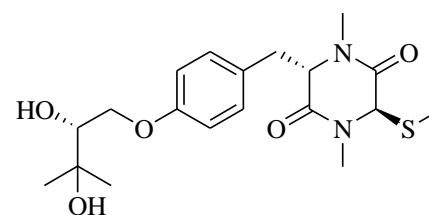
Sep 12 2014

INSTRUMENT: MERCURY-300  
PROBE atb  
OBSERVE H1  
Frequency 300.140 MHz  
Spectral width 5109.9 Hz  
Acquisition time 3.128 sec  
Relaxation delay 0.000 sec  
Pulse 45.0 degrees  
Ambient temperature  
65 repetitions  
Gated decoupling  
Double precision acquisition  
DATA PROCESSING  
FT size 65536  
Total acquisition time 3 minutes



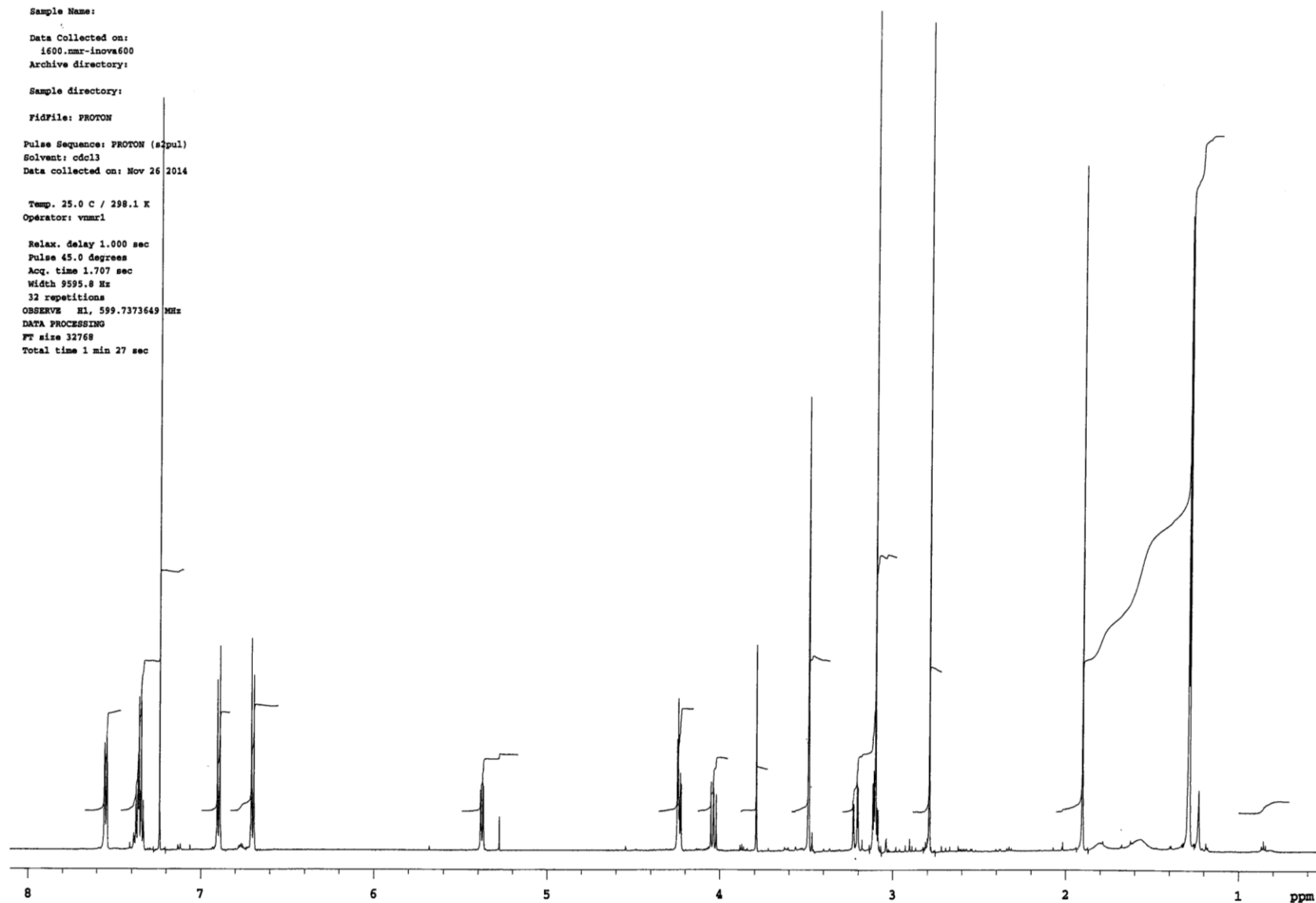
**Figure S9.** <sup>1</sup>H NMR spectrum of **12** in CDCl<sub>3</sub> at 300 MHz.

MD-M10-C54 cdcl3  
 Mondol / Latsch / CS  
 Sep 19 2014  
  
 INSTRUMENT INOVA-500  
 PROBE coldc 3mm  
 OBSERVE C13  
 Frequency 125.707 MHz  
 Spectral width 30120.5 Hz  
 Acquisition time 2.125 sec  
 Relaxation delay 0.000 sec  
 Pulse width 36.0 degrees  
 Temperature 27.0 deg. C / 300.1 K  
 No. repetitions 640  
 DECOUPLE H1  
 Frequency 499.877 MHz  
 Power 32 dB  
 Decoupler continuously on  
 HALTZ-16 modulated  
 Double precision acquisition  
 DATA PROCESSING  
 Line broadening 1.0 Hz  
 FT size 262144  
 Total acquisition time 22 minutes

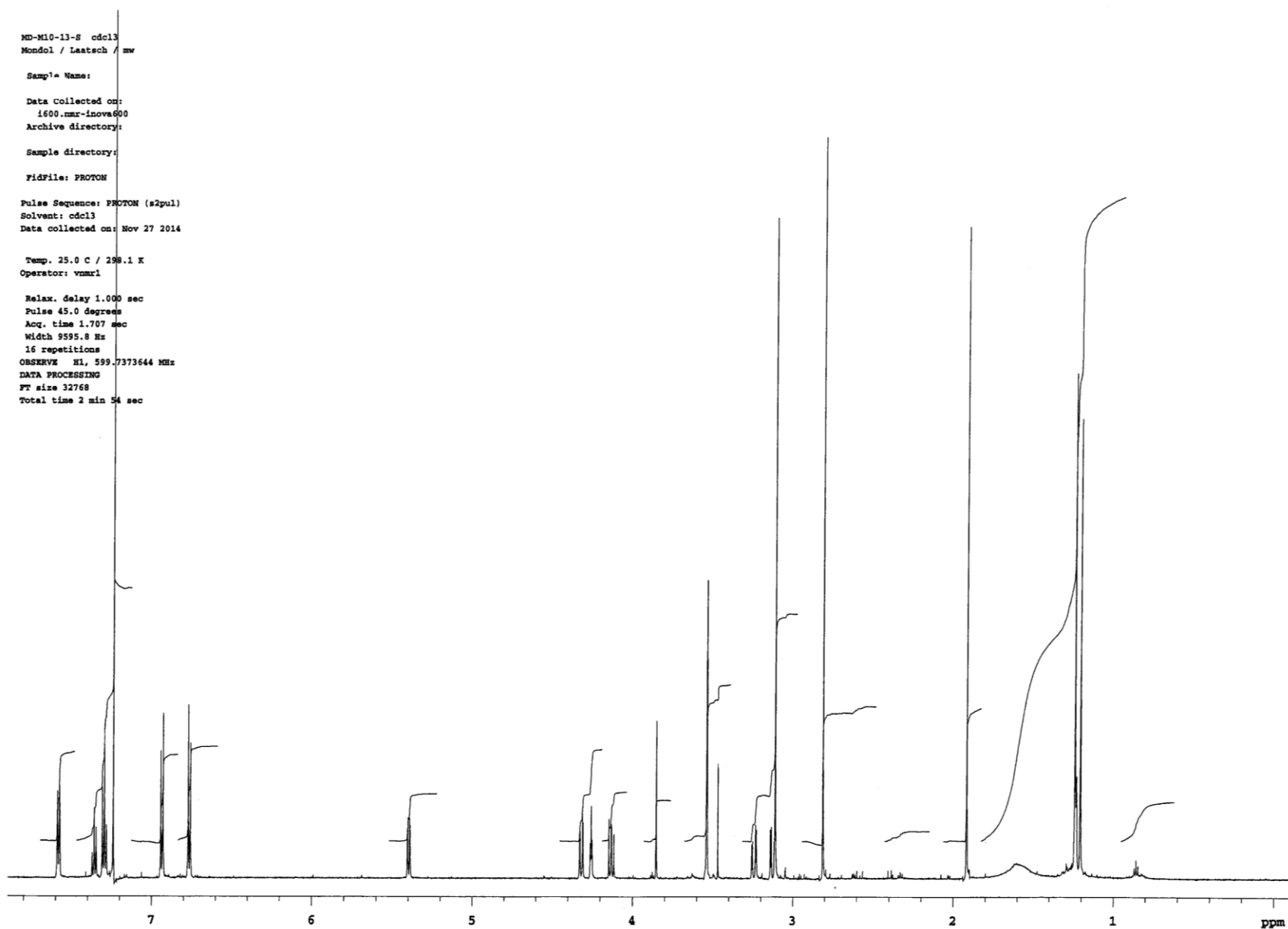


**Figure S10.**  $^{13}\text{C}$  NMR spectrum of **12** recorded in  $\text{CDCl}_3$  at 125 MHz.

M10-13R cdcl3  
Mondol / Laatsch / CS  
  
Sample Name:  
  
Data Collected on:  
1600.mmr-inova600  
Archive directory:  
  
Sample directory:  
  
FidFile: PROTON  
  
Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Nov 26 2014  
  
Temp. 25.0 C / 298.1 K  
Operator: vmr1  
  
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.707 sec  
Width 9595.8 Hz  
32 repetitions  
OBSERVE H1, 599.7373649 MHz  
DATA PROCESSING  
F1 size 32768  
Total time 1 min 27 sec



**Figure S11.** <sup>1</sup>H NMR spectrum of **12a** recorded in CDCl<sub>3</sub> at 600 MHz.



**Figure S12.**  $^1\text{H}$  NMR spectrum of **12b** recorded in  $\text{CDCl}_3$  at 600 MHz.

MD-M10-C32-2 cd3od  
Mondol / Laatsch / mw

Sample Name:

Data Collected on:  
1600.nmr-inova600

Archive directory:

Sample directory:

Fidfile: PROTON

Pulse Sequence: PROTON (s2pul)  
Solvent: cd3od  
Data collected on: Oct 23 2014

Temp. 25.0 C / 298.1 K  
Operator: vnmr1

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.707 sec  
Width 9595.8 Hz  
32 repetitions  
OBSERVE H1 599.7397189 MHz  
DATA PROCESSING  
F2 size 32768  
Total time 1 min 27 sec

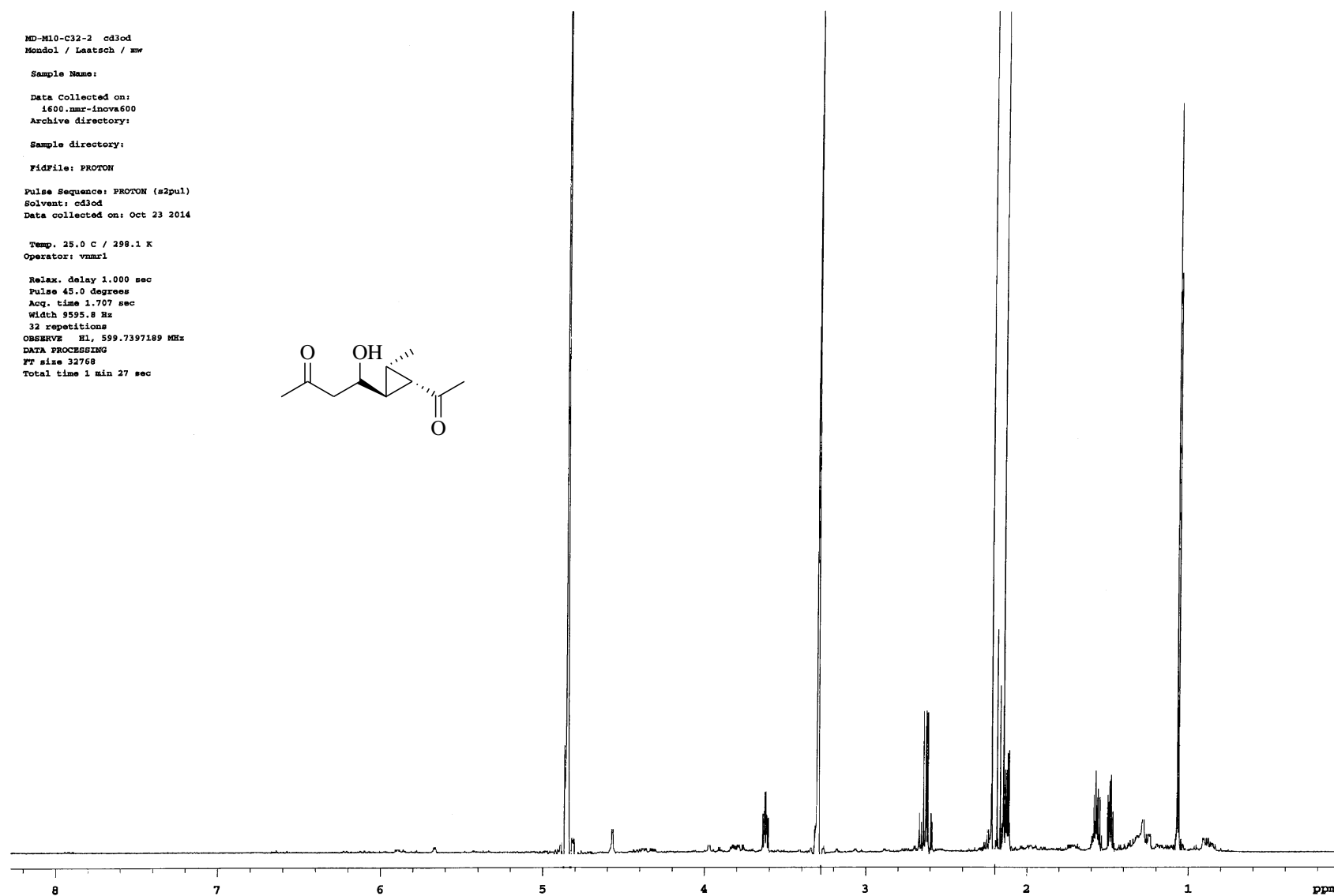
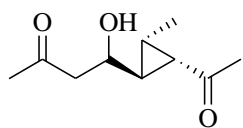
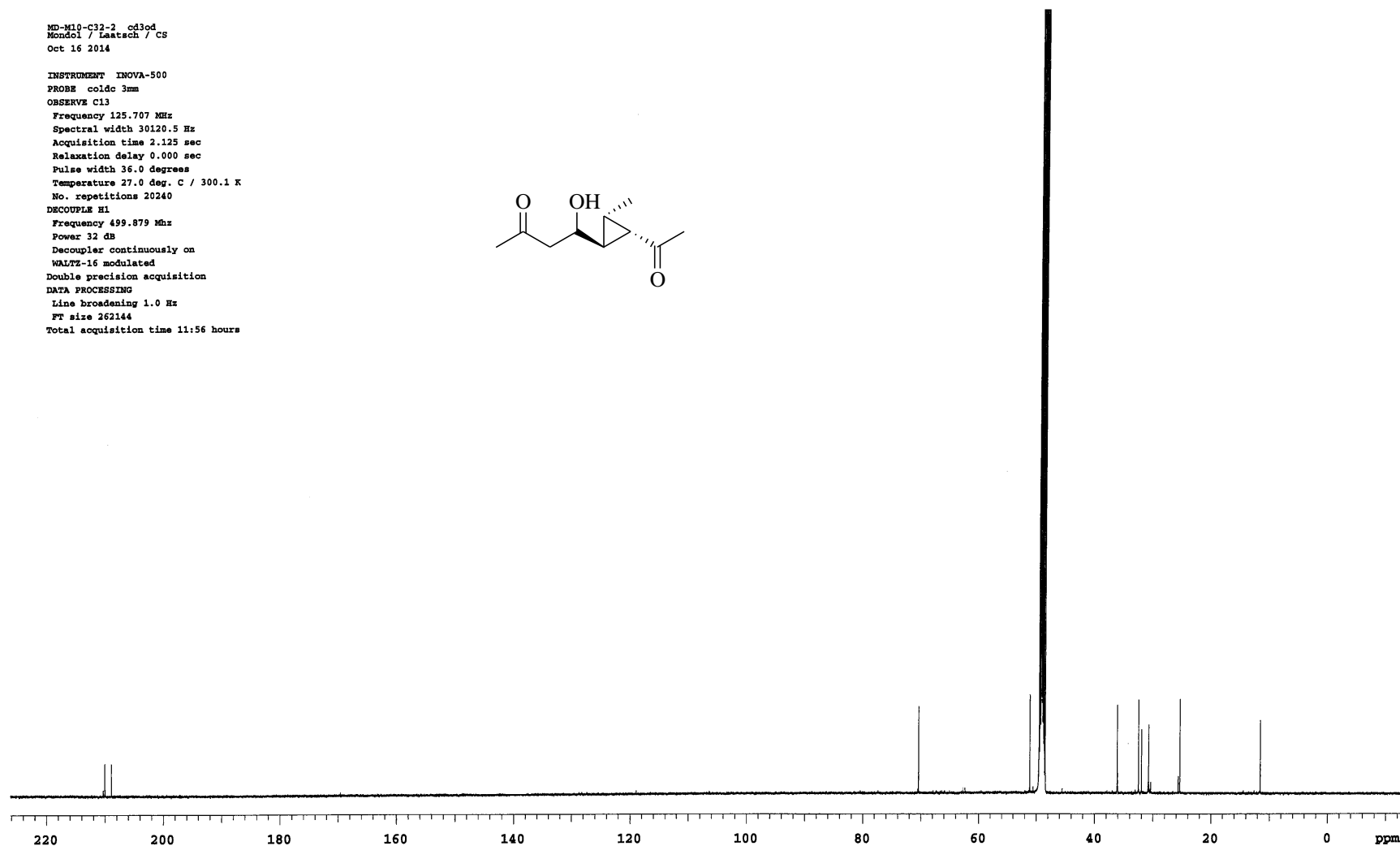
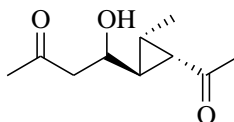


Figure S13.  $^1\text{H}$  NMR spectrum of **14** recorded in  $\text{CD}_3\text{OD}_3$  at 600 MHz.

MD-M10-C32-2 cd3od  
Mondol / Laatsch / CS  
Oct 16 2014

INSTRUMENT INOVA-500  
PROBE coldc 3mm  
OBSERVE C13  
Frequency 125.707 MHz  
Spectral width 30120.5 Hz  
Acquisition time 2.125 sec  
Relaxation delay 0.000 sec  
Pulse width 36.0 degrees  
Temperature 27.0 deg. C / 300.1 K  
No. repetitions 20240  
DECOUPLE H1  
Frequency 499.879 MHz  
Power 32 dB  
Decoupler continuously on  
WALTZ-16 modulated  
Double precision acquisition  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 262144  
Total acquisition time 11:56 hours



**Figure S14.**  $^{13}\text{C}$  NMR spectrum of **14** recorded in  $\text{CD}_3\text{OD}$  at 125 MHz



**Table S1. Selected  $^1\text{H}$  Chemical Shifts for Verrucarins J (2) Isomers, and Verrucarins Y (1) for Determination of the  $\Delta^2$  Configuration**

C No.	2'-(Z)-2	2'-(E)-2	1
15	5.05 ( $\text{H}_a$ )	4.44 ( $\text{H}_a$ )	4.42 ( $\text{H}_a$ )
	3.65 ( $\text{H}_b$ )	3.97 ( $\text{H}_b$ )	3.83 ( $\text{H}_b$ )
4'	4.15 ( $\text{H}_a$ )	2.50 ( $\text{H}_2$ )	2.52 ( $\text{H}_2$ )
	2.30 ( $\text{H}_b$ )		
5'	4.66–4.46 ( $\text{H}_2$ )	4.47 ( $\text{H}_a$ )	4.43 ( $\text{H}_a$ )
		4.15 ( $\text{H}_b$ )	4.12 ( $\text{H}_b$ )

#### Calculation of ORD data to determine the absolute configuration of bilain D (**12**)

The absolute configuration of C-15 in bilain D (**12**) was determined as (15*R*) by Mosher's method. As the diketopiperazine ring is *trans*-configured according to NOESY data, the absolute configuration of **12** must be (3*S*,6*R*,15*R*) or (3*R*,6*S*,15*R*). For both diastereomers in question, the ORD data were calculated according to procedures described previously.<sup>1</sup> For the (3*S*,6*R*,15*R*)-isomer, an  $[\alpha]_{\text{D}}^{20}$  value of +118.6° resulted, while the (3*R*,6*S*,15*R*)-isomer afforded  $[\alpha]_{\text{D}}^{20} = -9.7^\circ$ . The experimental value of  $[\alpha]_{\text{D}}^{20} = -24^\circ$  indicated an absolute (3*R*,6*S*,15*R*)-configuration for **12**.

<sup>1</sup> Mondol, M. A. M.; Farhouse, J.; Islam, M. T.; Schüffler, A.; Laatsch, H. A new lactone from *Chaetomium globosum* strain M65 that inhibits the motility of zoospores. *Nat. Prod. Commun.* Submitted June 27, **2015**.