

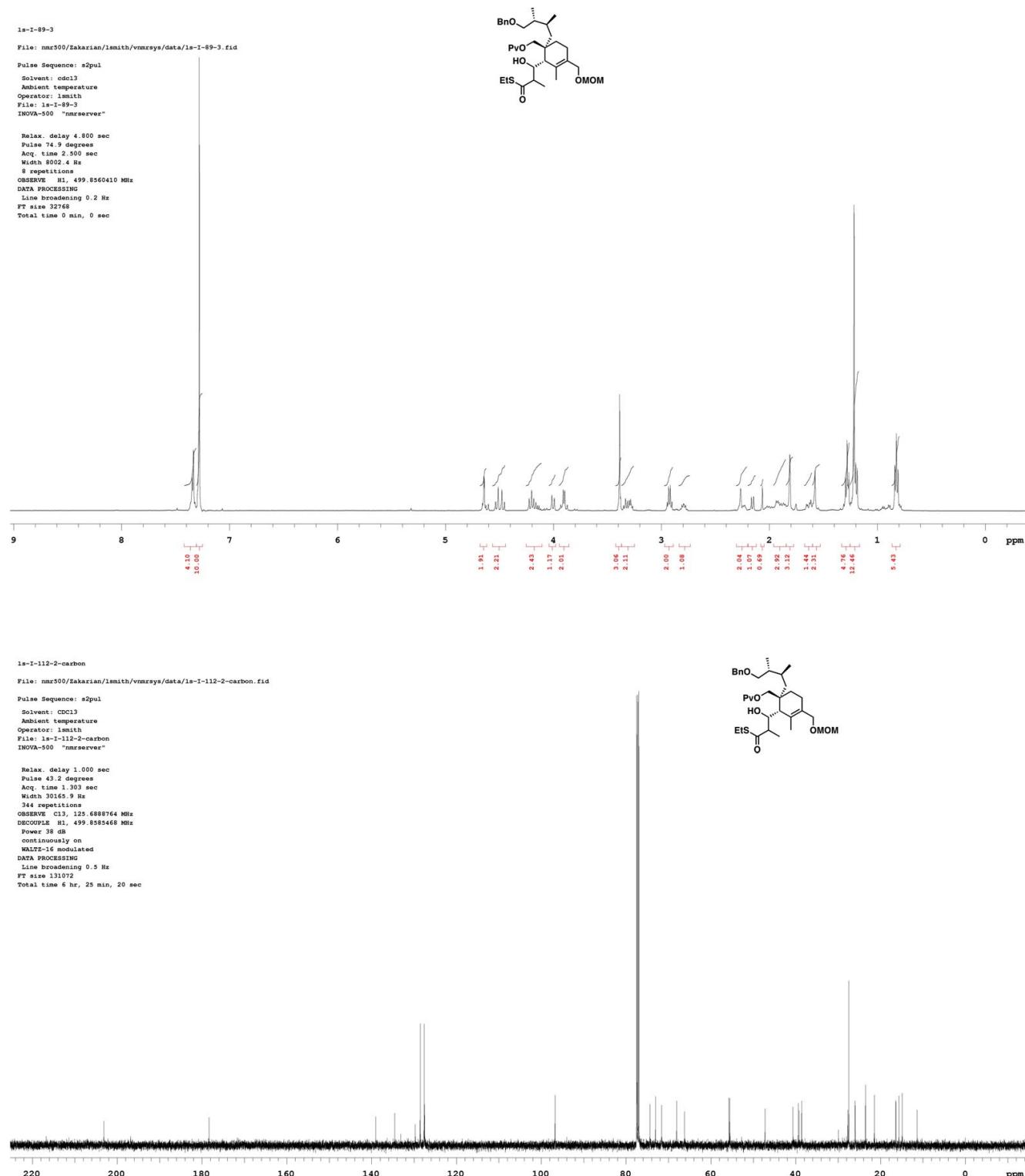
Studies Toward the Synthesis of Spirolide C: Exploration into the Formation of the 23-Membered All-Carbon Macrocyclic Framework

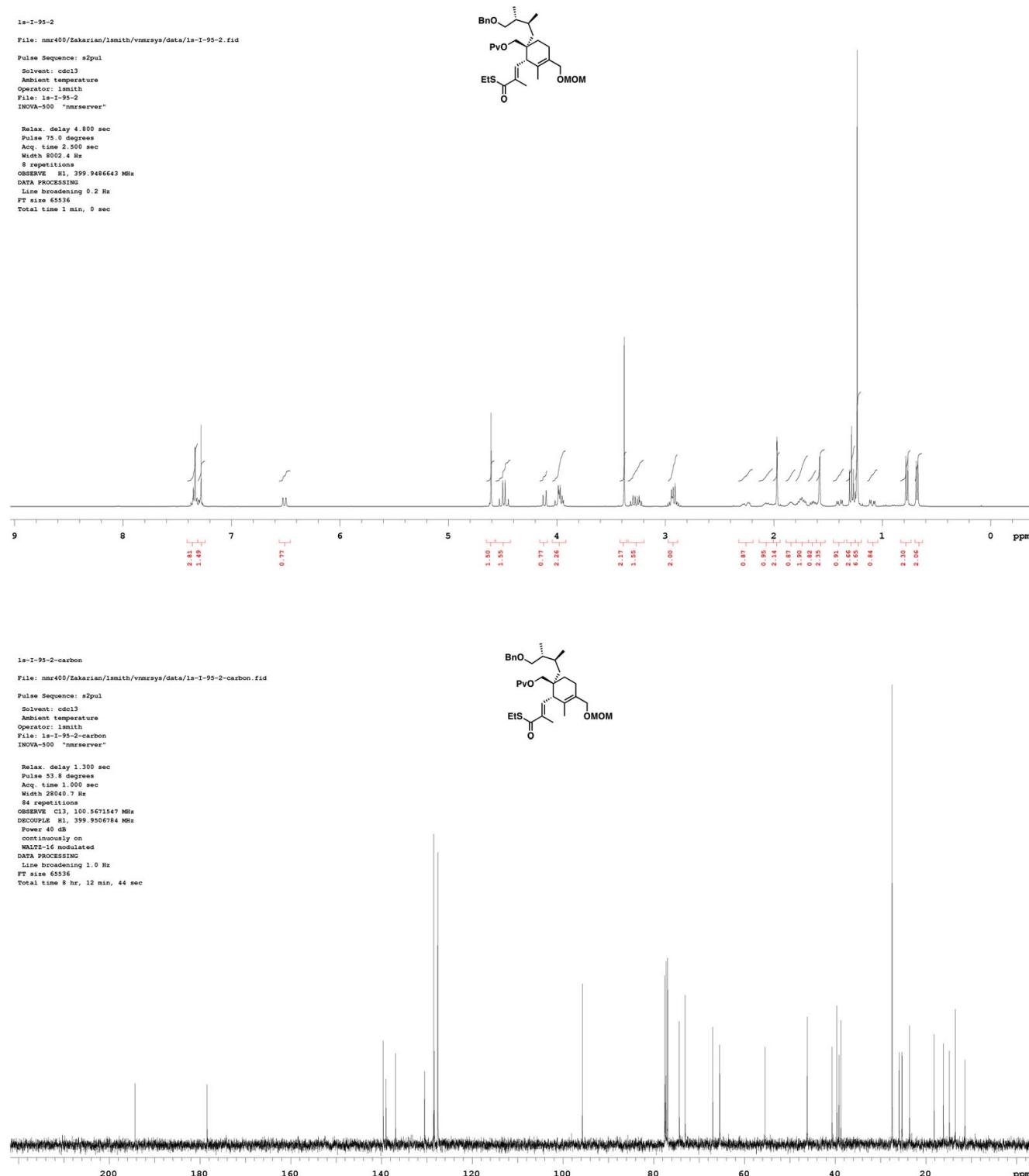
Craig E. Stivala, Zhenhua Gu, Lindsay Smith, and Armen Zakarian

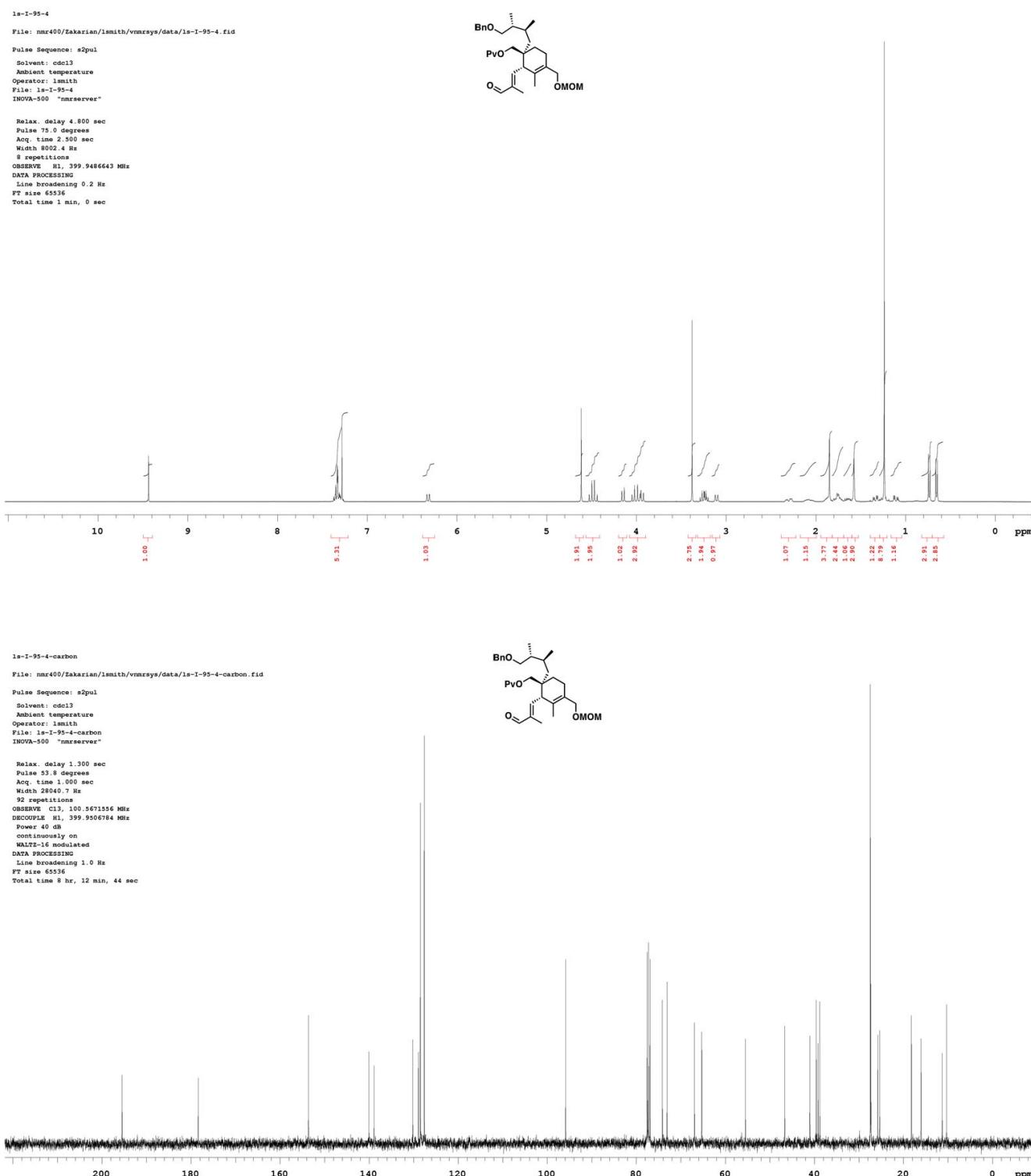
Department of Chemistry and Biochemistry, University of California, Santa Barbara, California 93106,
United States

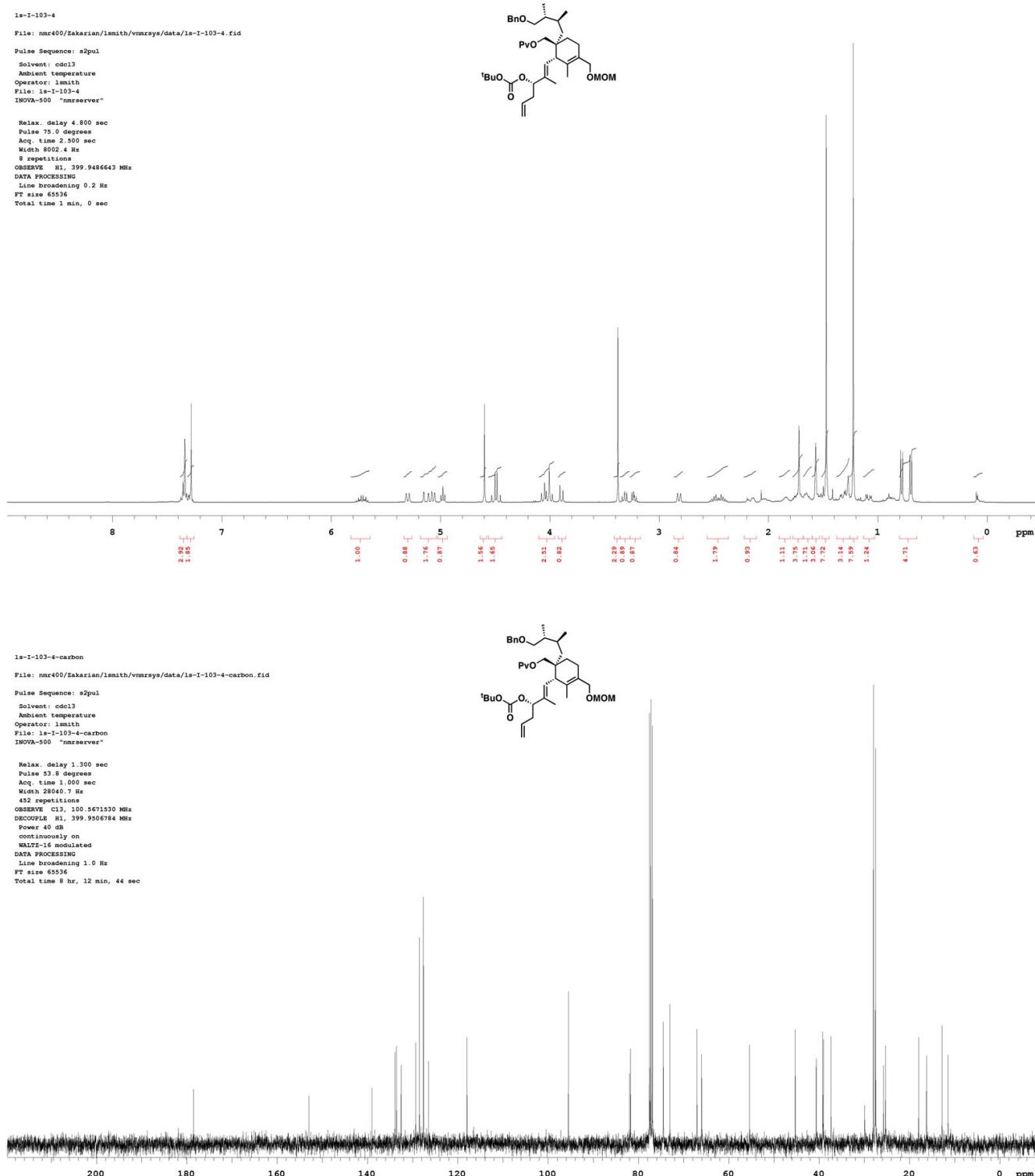
SUPPORTING INFORMATION 2

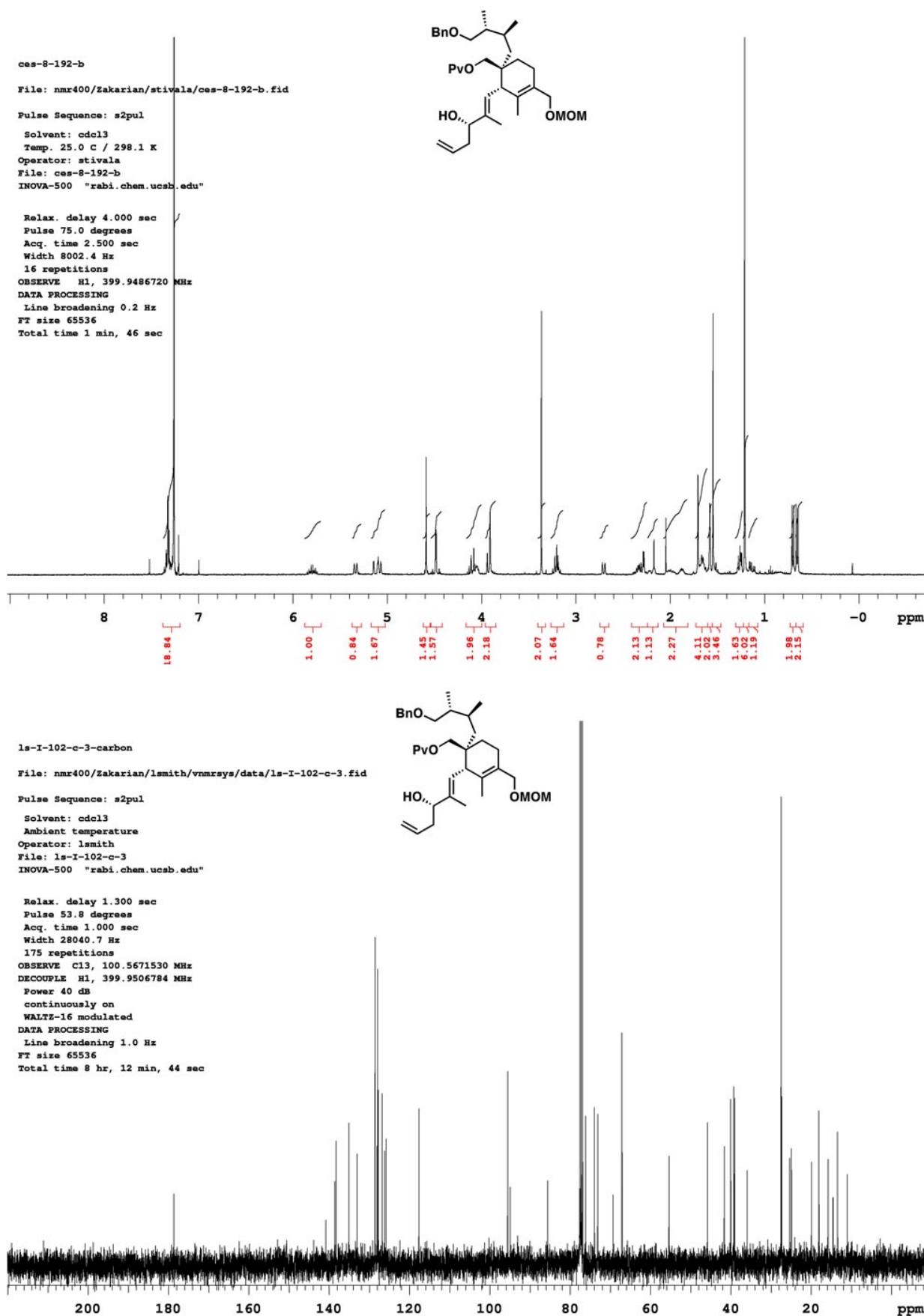
Copies of Spectra

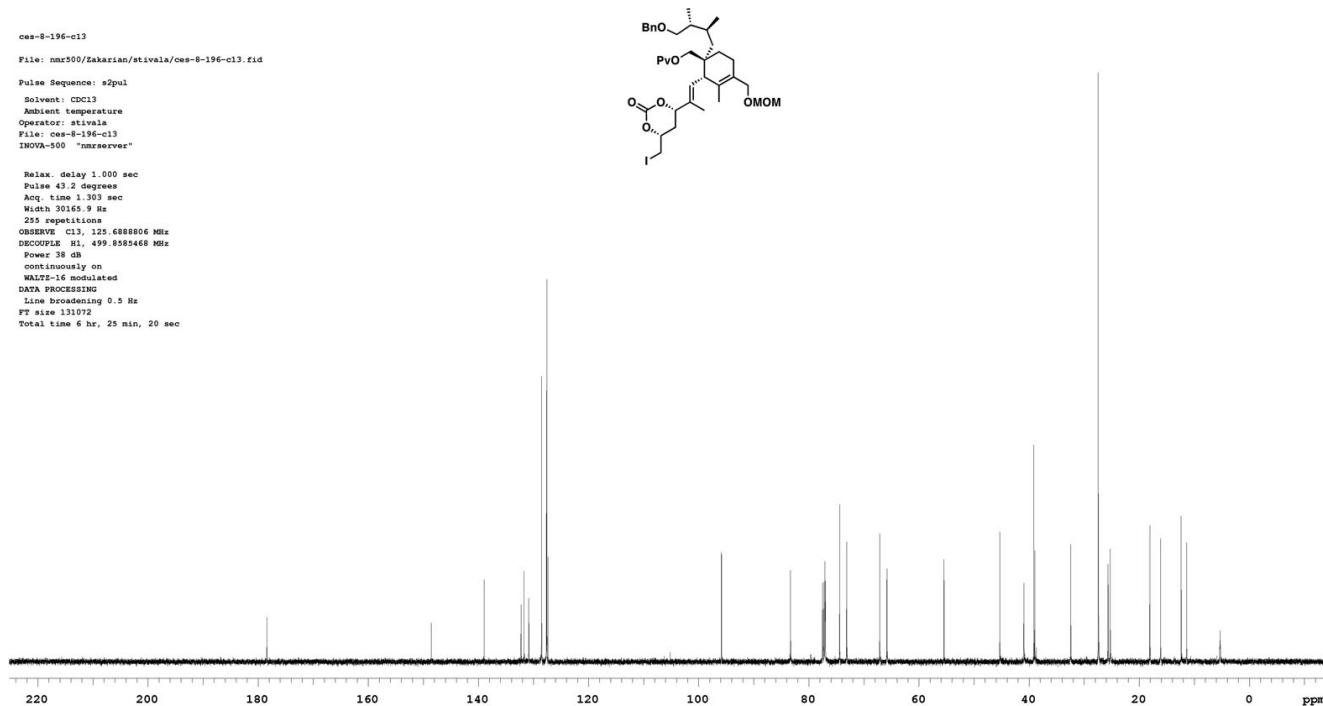
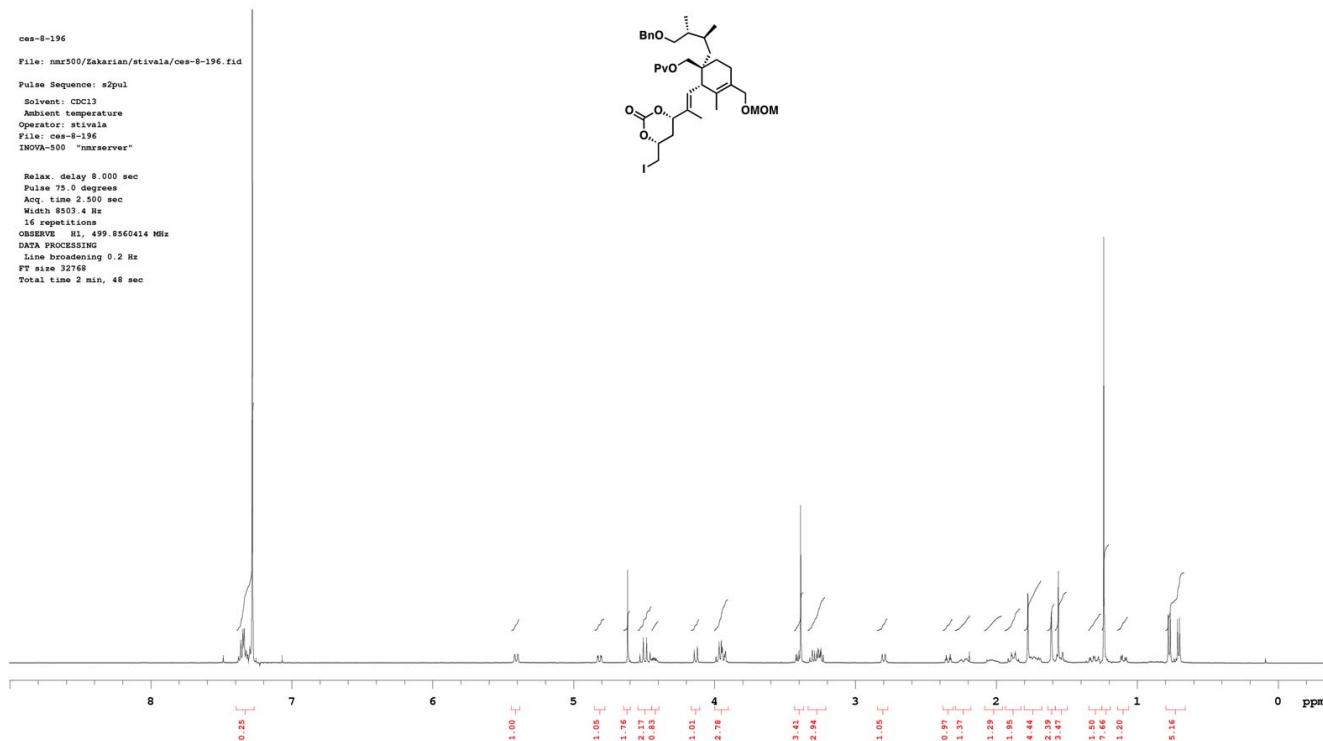




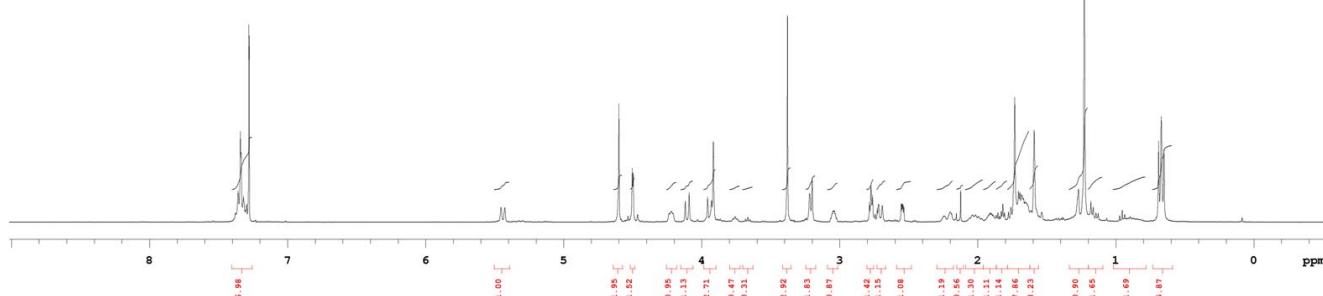
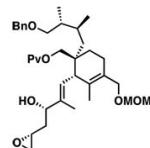




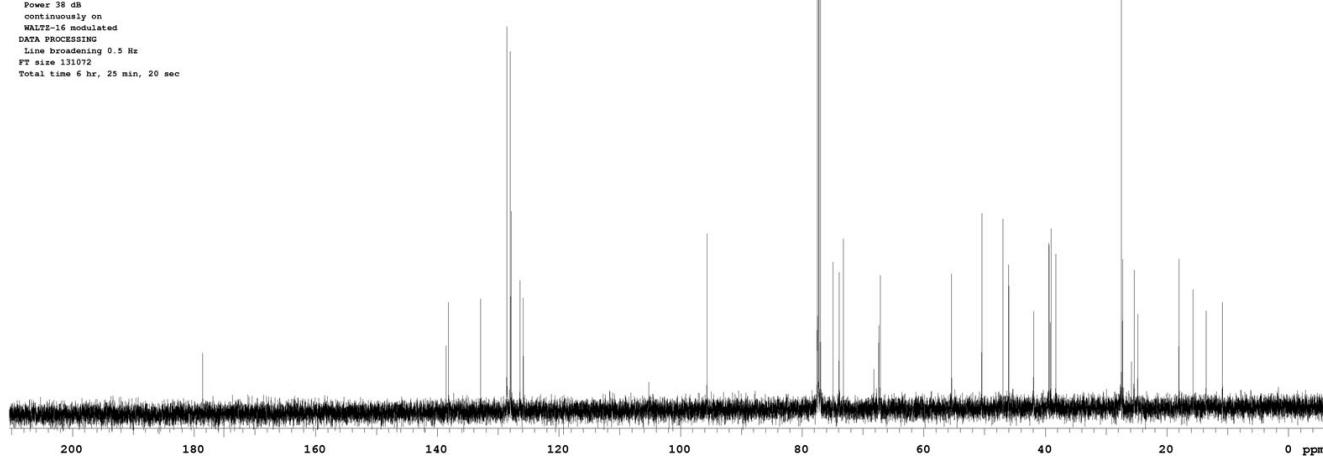
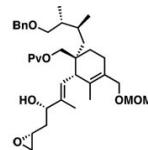


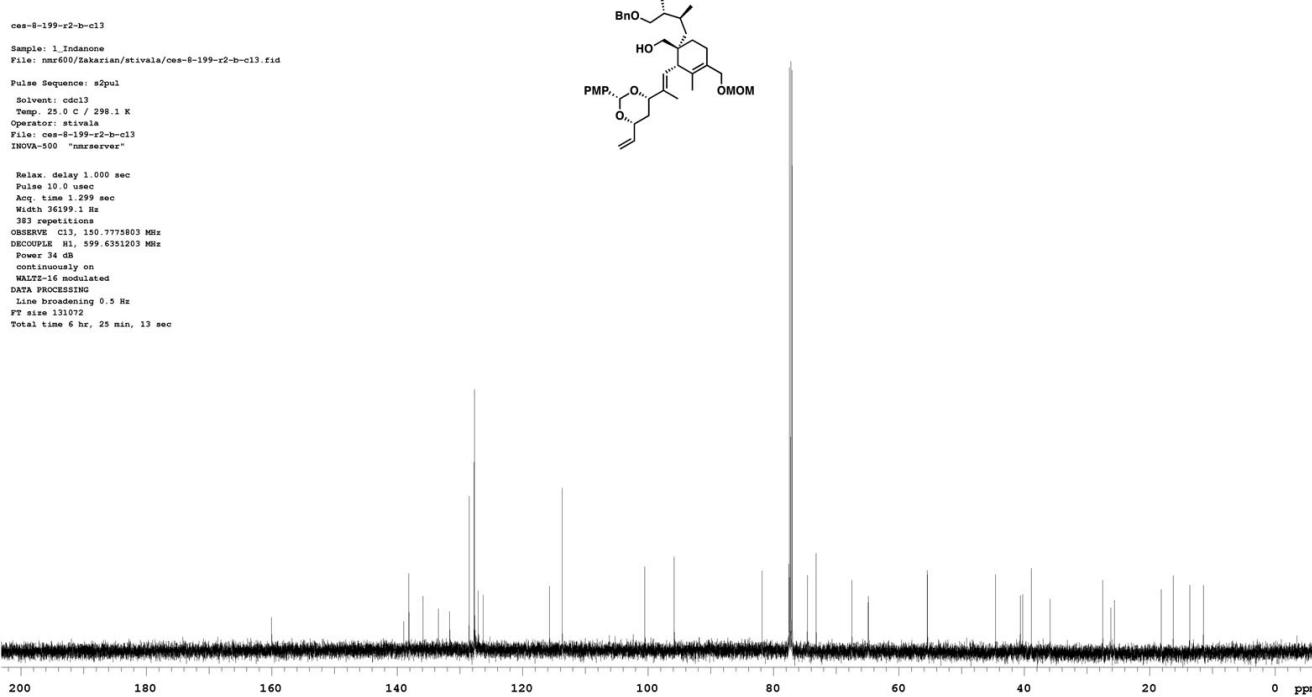
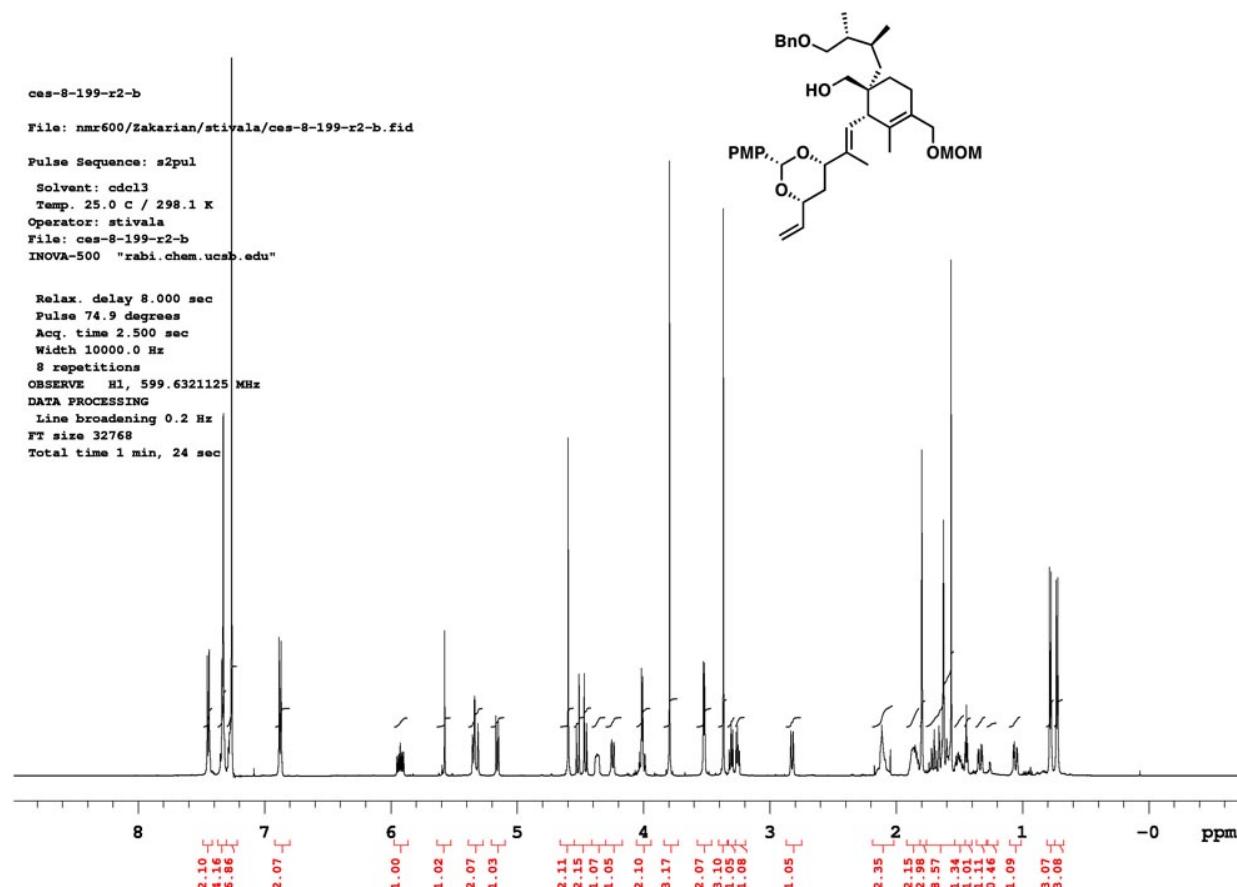


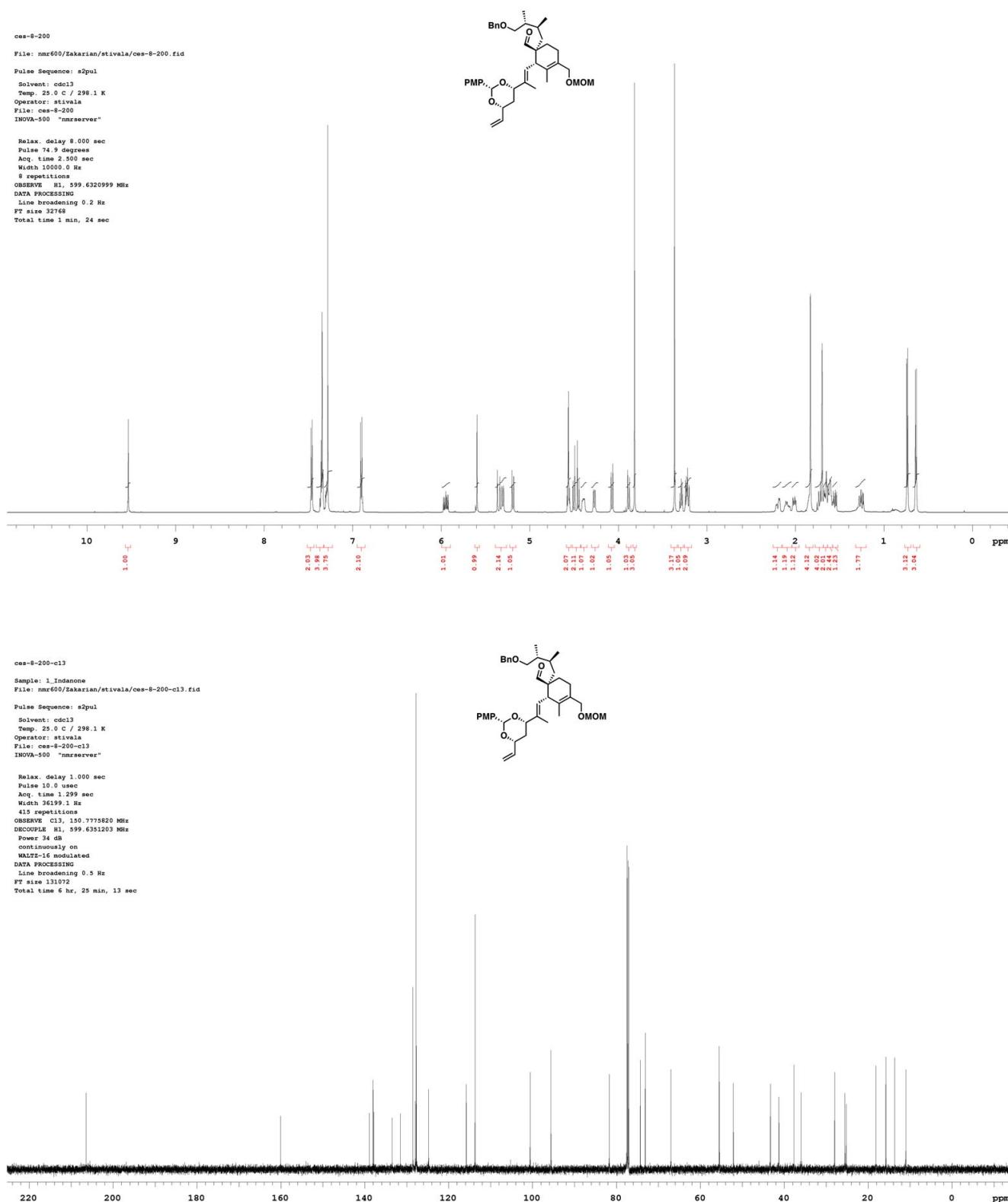
ces-8-197
 File: nmr400/Zakarian/stivala/ces-8-197.fid
 Pulse Sequence: s2pul
 Solvent: CDCl₃
 Temp: 29.0 °C / 298.1 K
 Operator: stivala
 File: ces-8-197
 INOVA-500 "nmrserver"
 Relax. delay 4.000 sec
 Pulse 75.0 degrees
 Acq. time 2.500 sec
 Width: 30165.9 Hz
 4 repetitions
 OBSERVE H1, 399.9486645 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 6 min, 20 sec

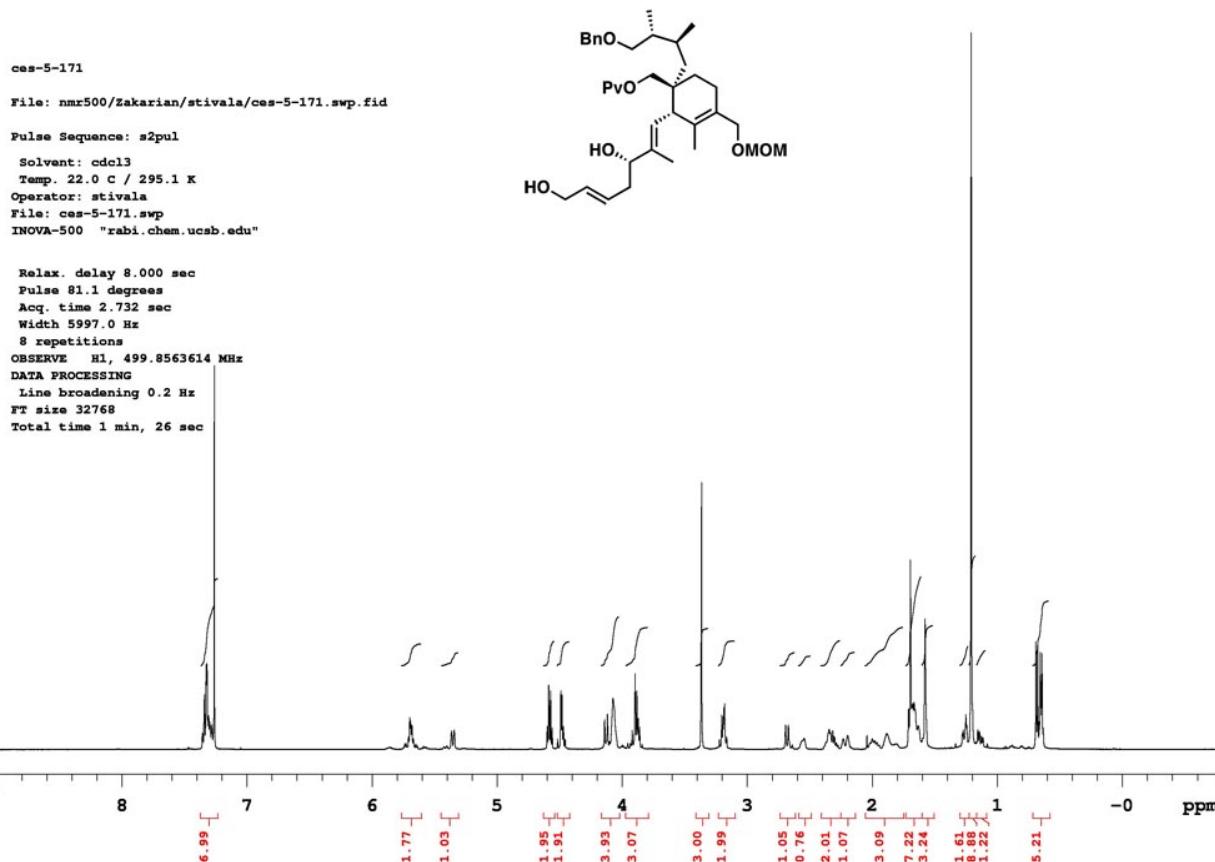
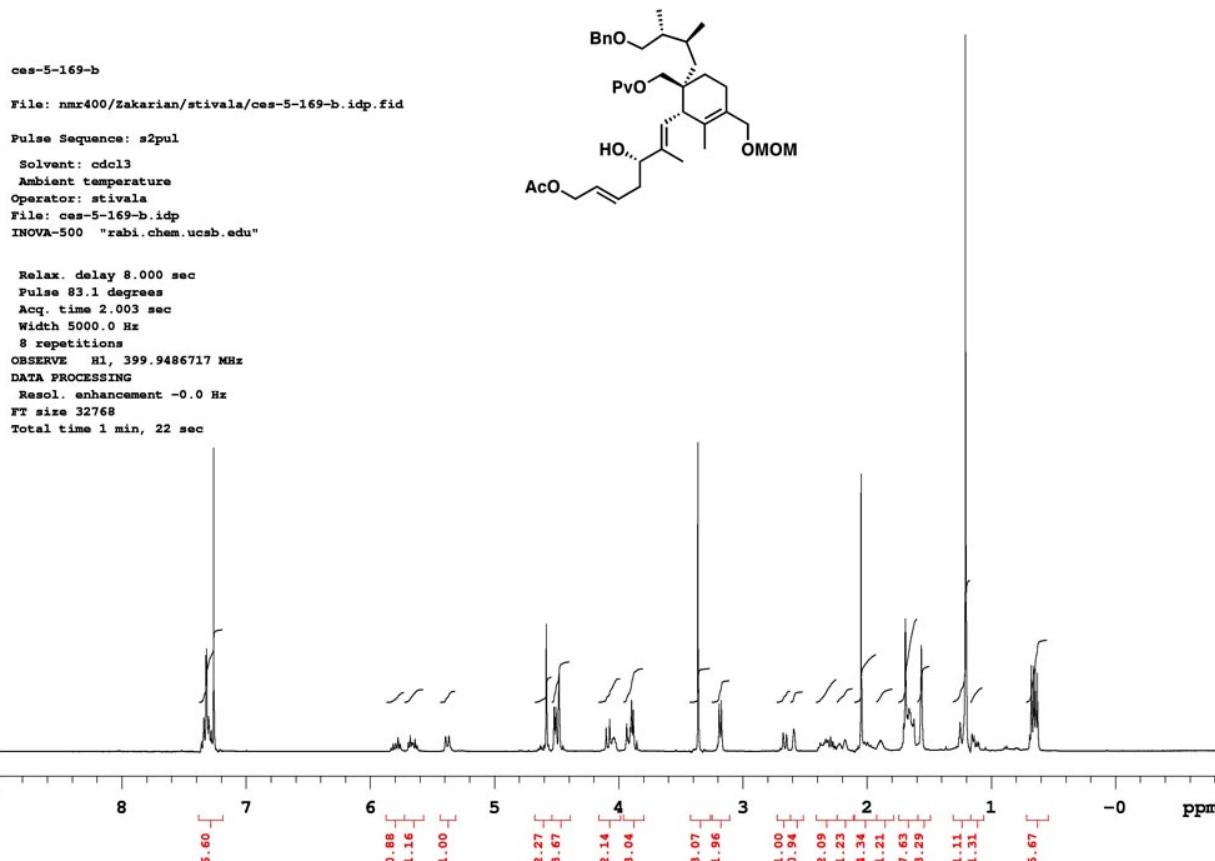


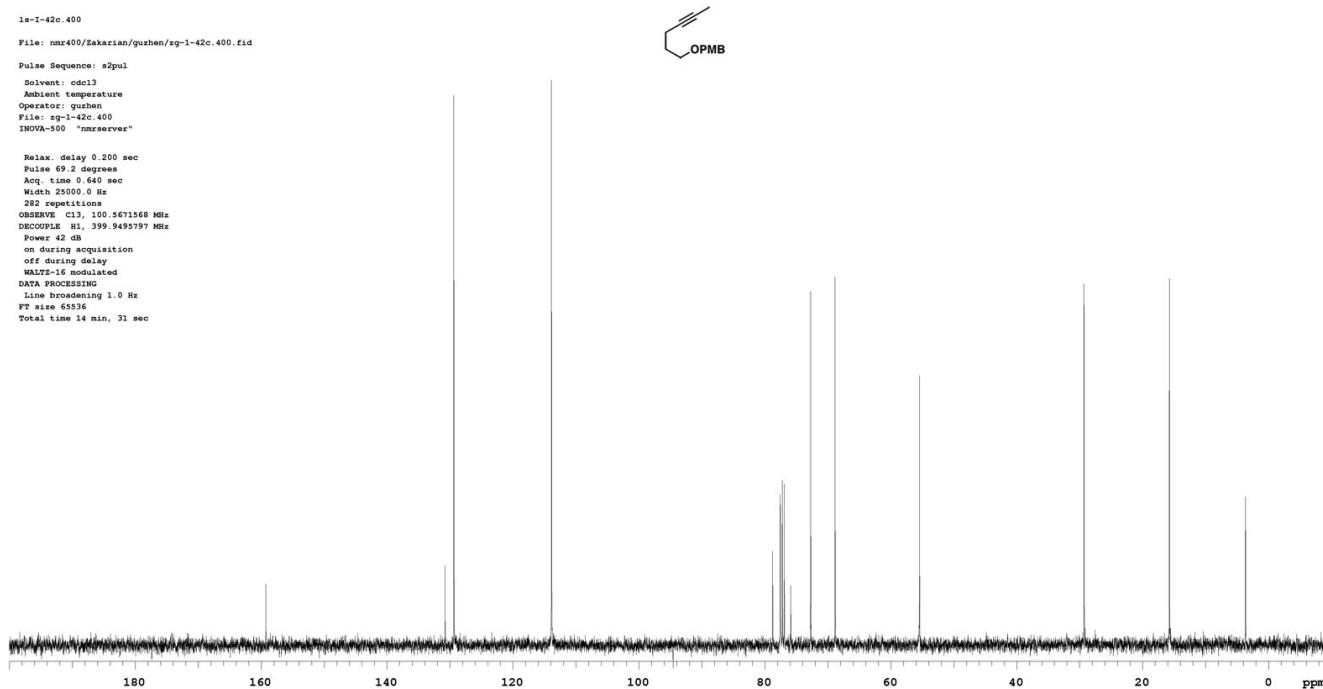
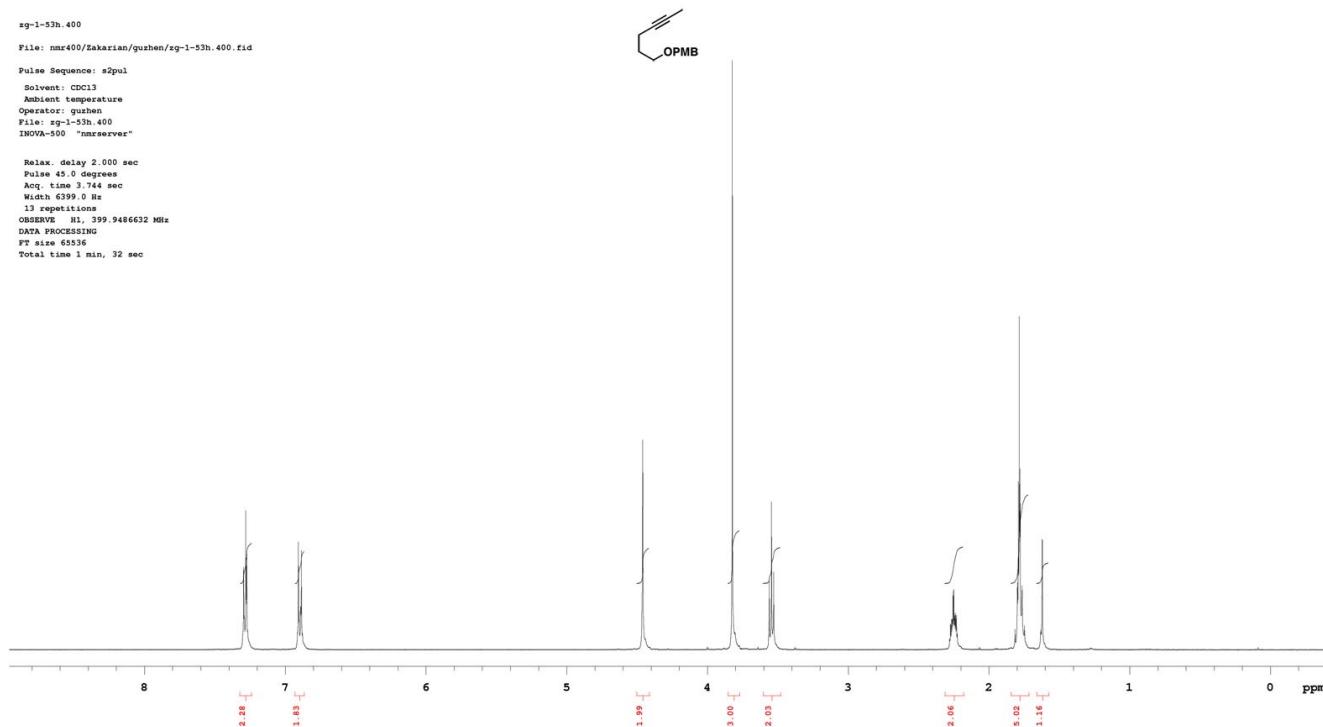
ces-8-197-c13
 File: nmr500/Zakarian/stivala/ces-8-197-c13.fid
 Pulse Sequence: s2pul
 Solvent: CDCl₃
 Ambient temperature
 Operator: stivala
 File: ces-8-197-c13
 INOVA-500 "nmrserver"
 Relax. delay 1.000 sec
 Pulse 43.2 degrees
 Acq. time 1.303 sec
 Width 30165.9 Hz
 135 repetitions
 OBSERVE C13, 131.0688760 MHz
 DQFCOSY, 131.0688760, 139.5585468 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072
 Total time 6 hr, 25 min, 20 sec

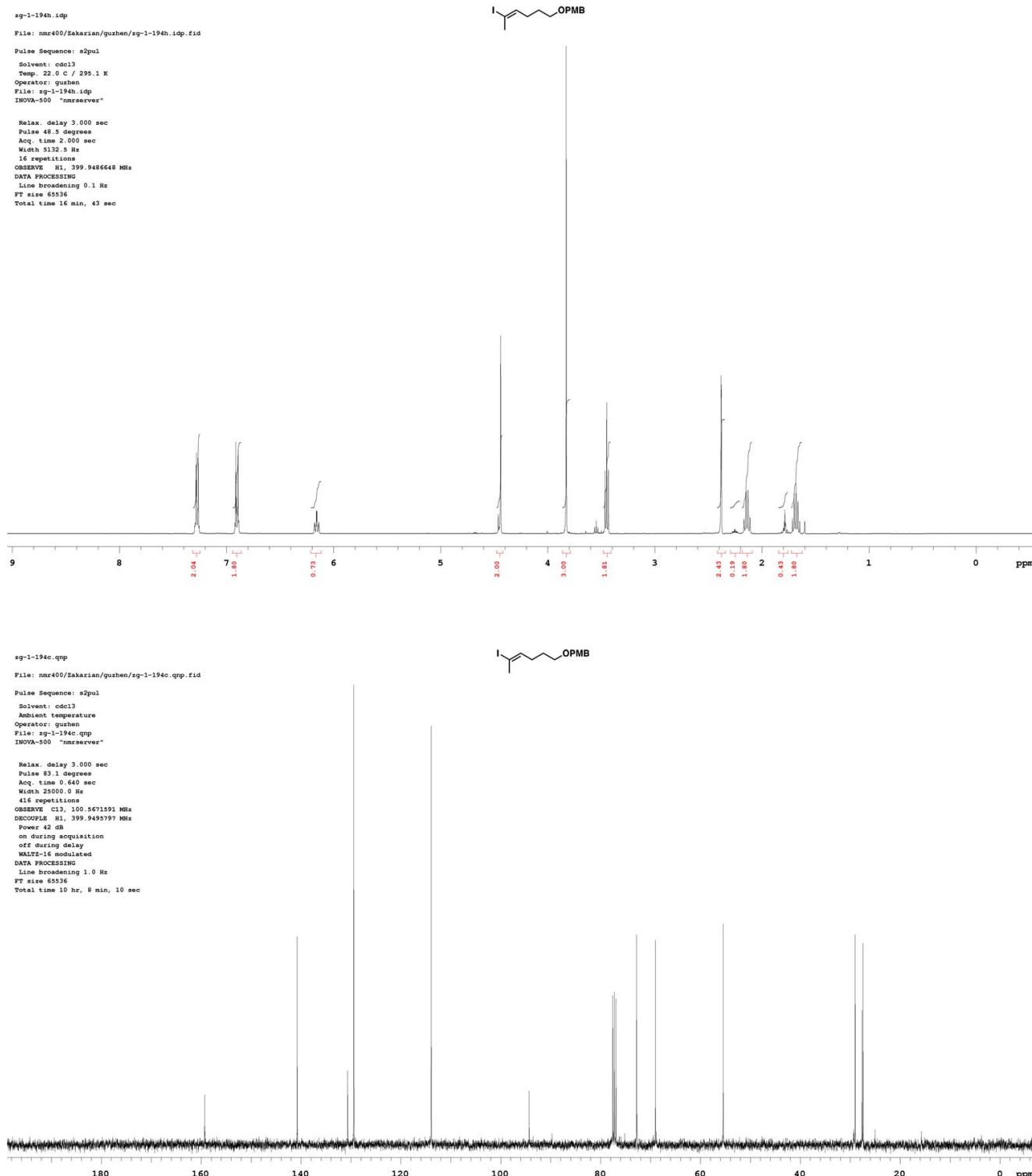


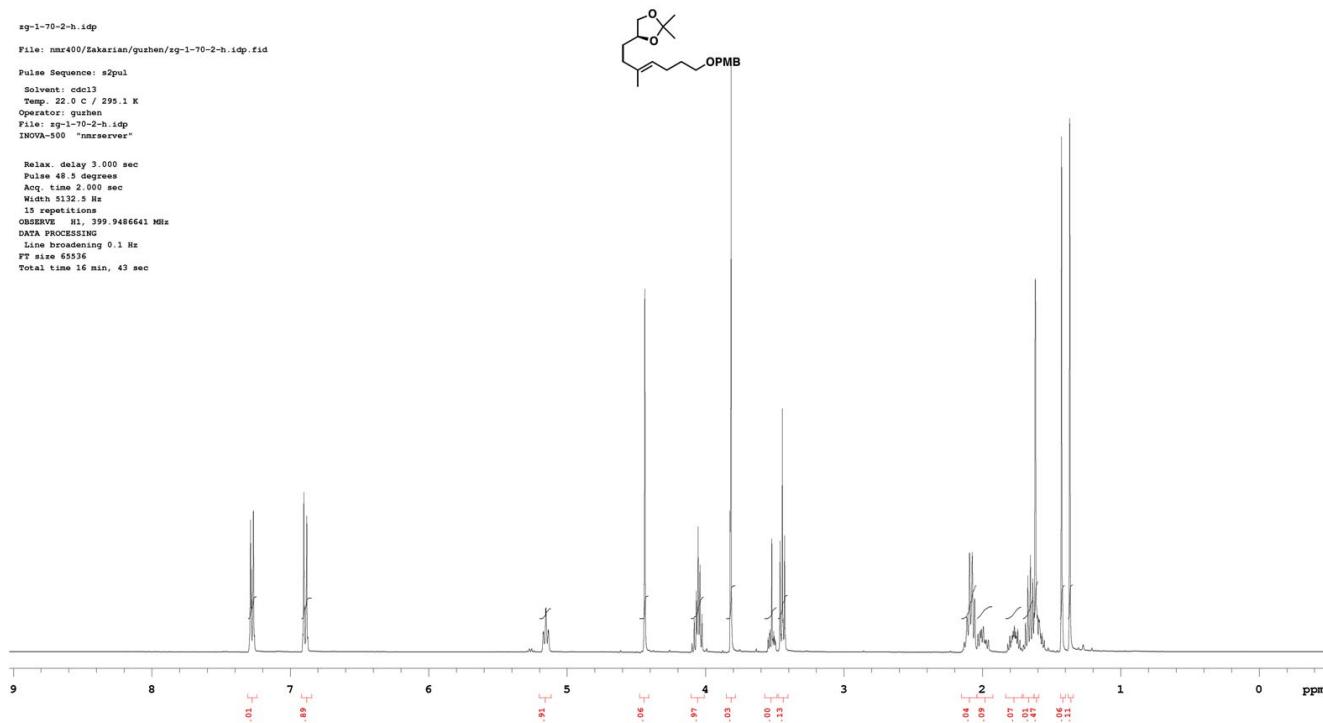












zg-1-70-2c.400

File: nmr400/Zakarian/guzhen/zg-1-70-2c.400.fid

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: guzhen

File: zg-1-70-2c.400

INOVVA-500 "rabi.chem.ucsb.edu"

Relax. delay 0.800 sec

Pulse 69.2 degrees

Acq. time 0.640 sec

Width 25000.0 Hz

968 repetitions

OBSERVE C13, 100.5671544 MHz

DECOPPLE H1, 399.9495797 MHz

Power 42 dB

on during acquisition

off during delay

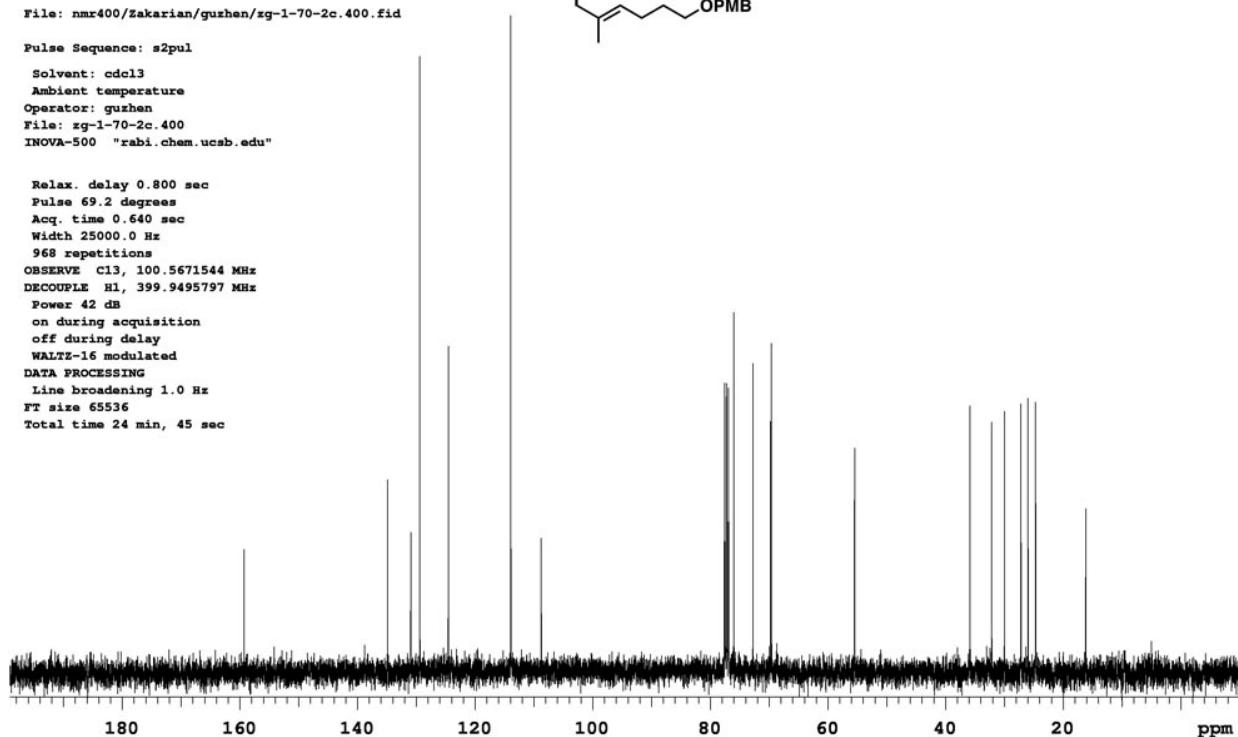
WALTZ-16 modulated

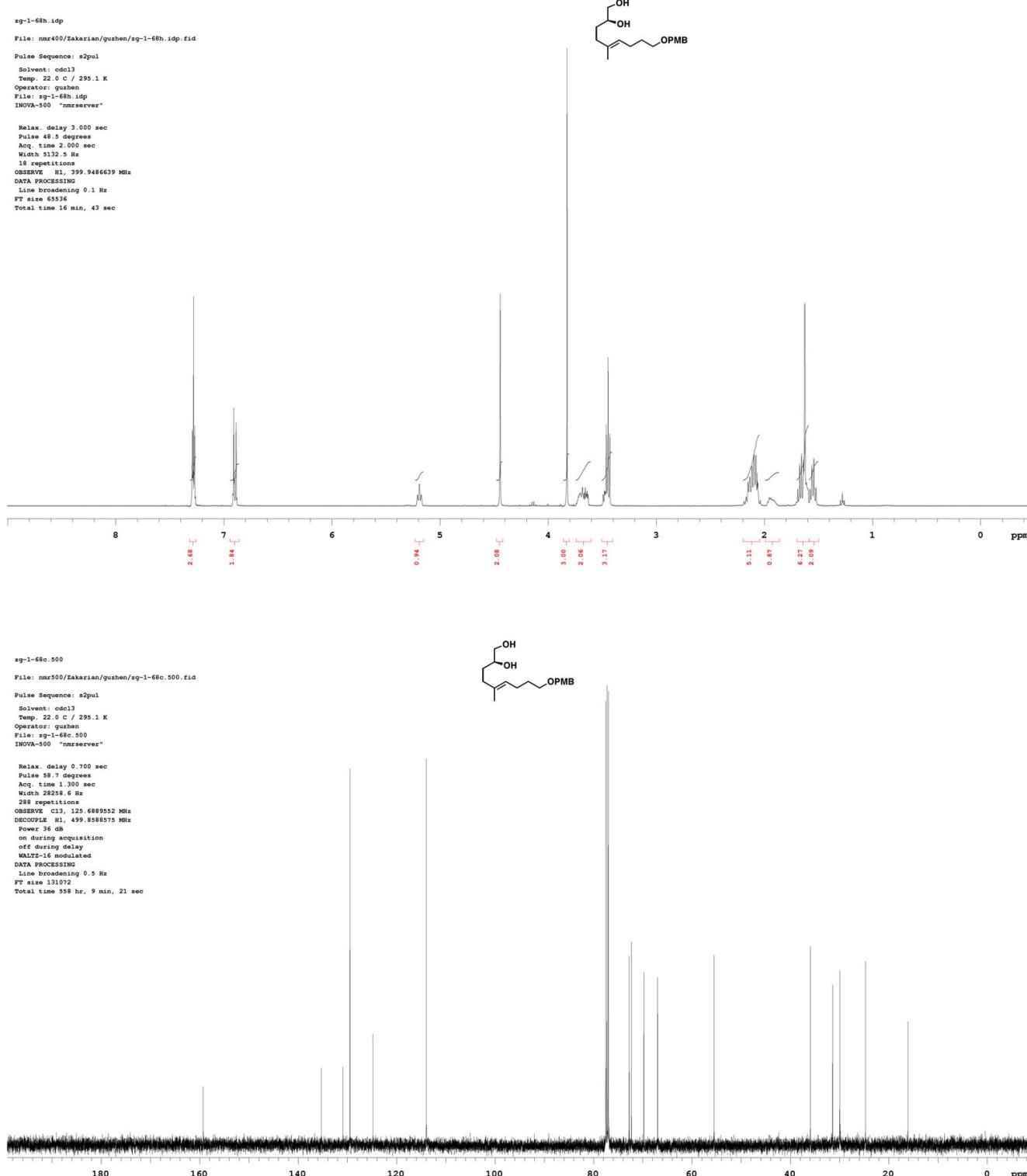
DATA PROCESSING

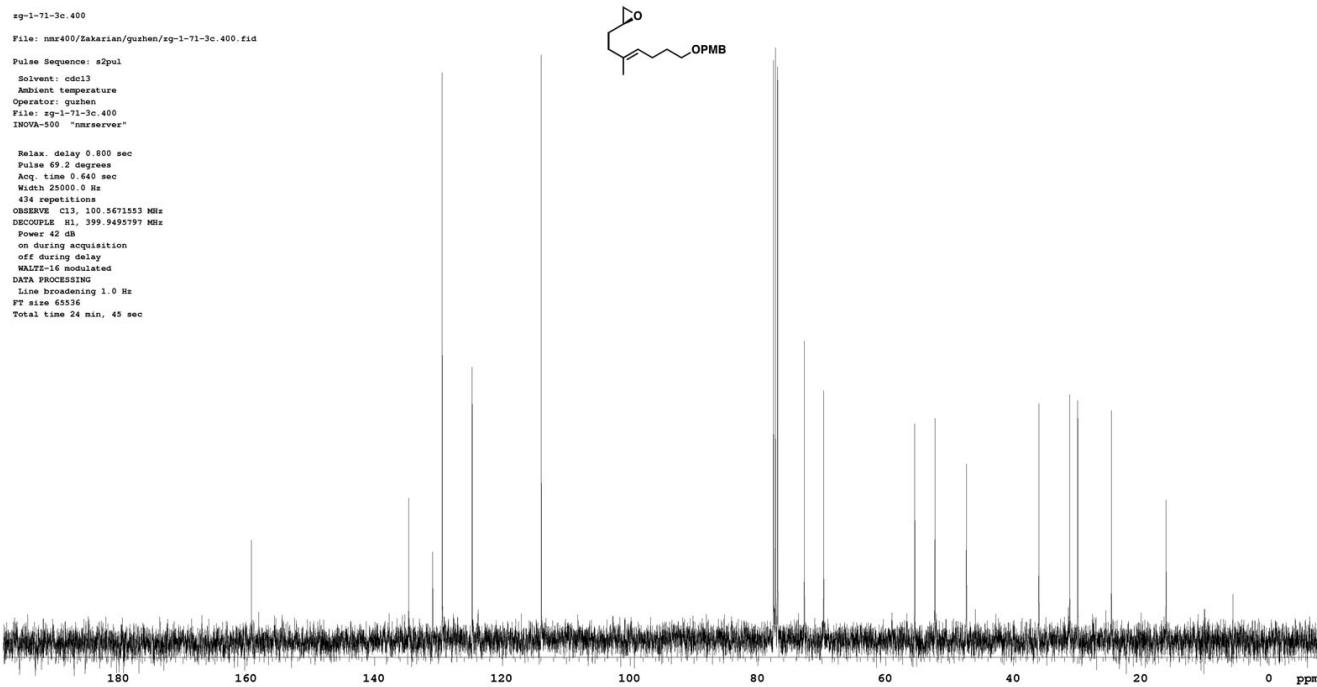
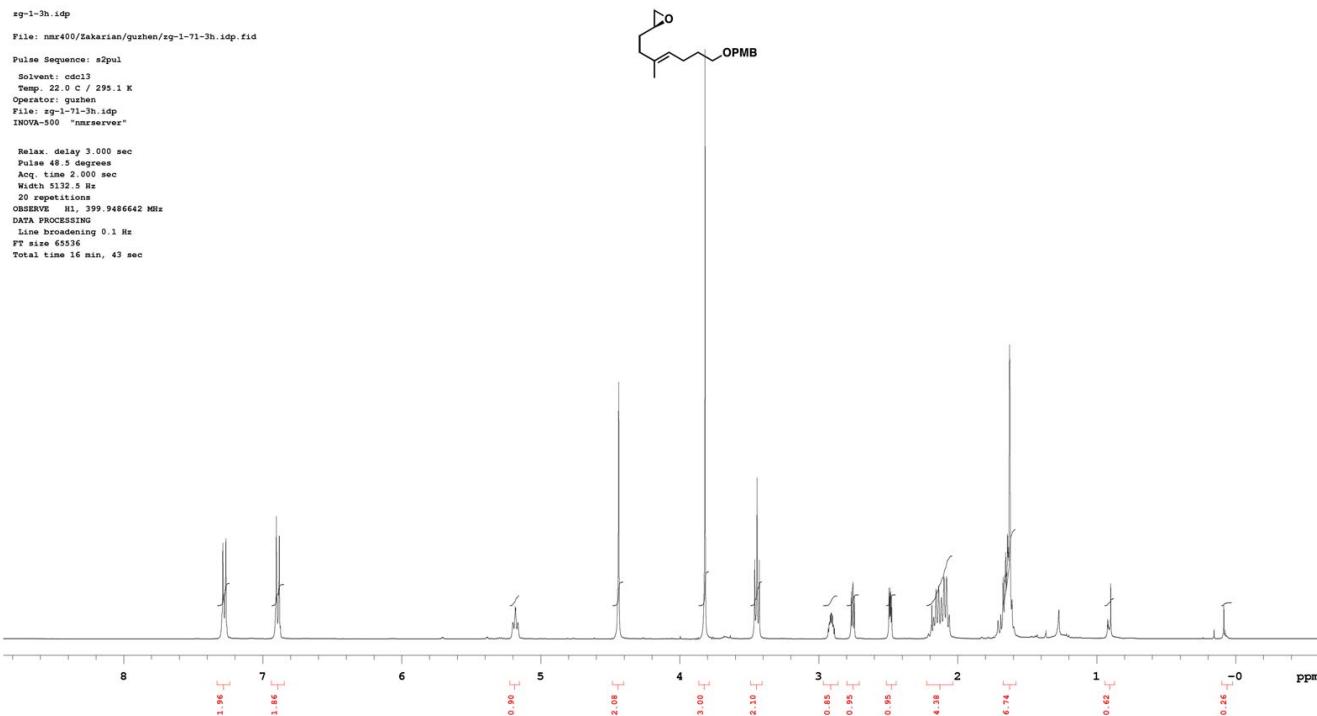
Line broadening 1.0 Hz

FT size 65536

Total time 24 min, 45 sec

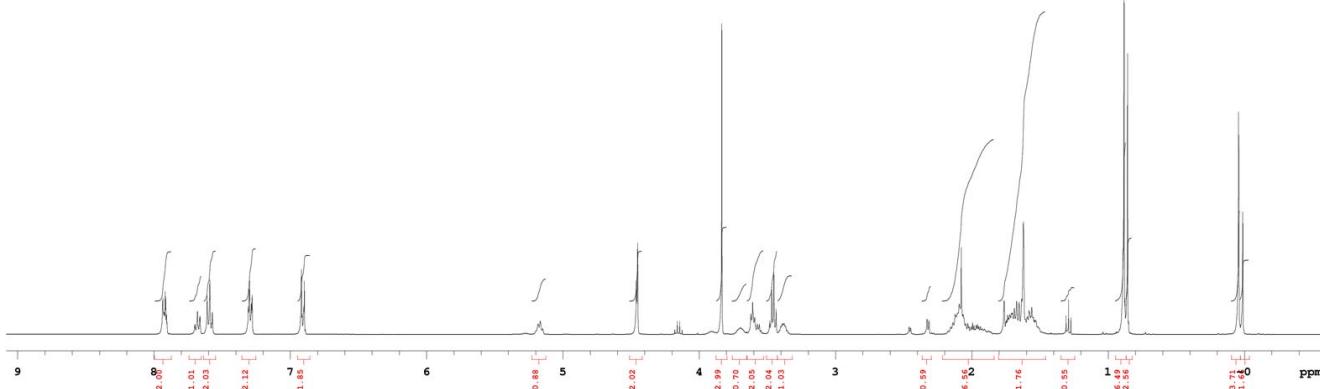
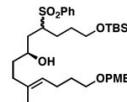






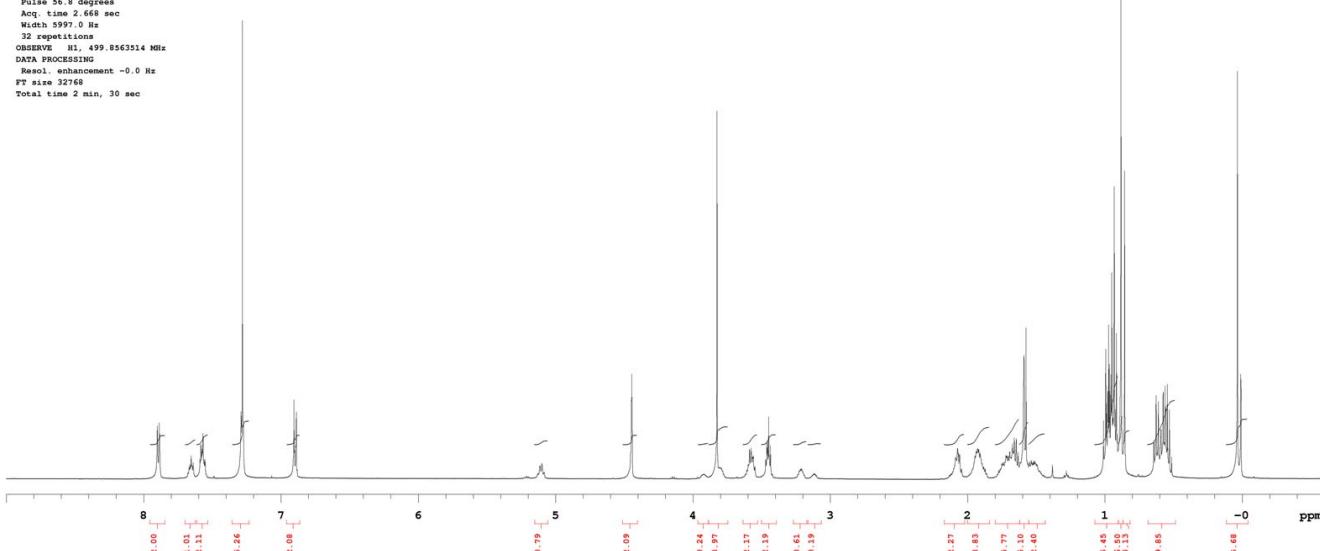
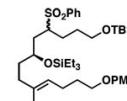
zg-1-243h.idp
 File: nmr400/Zakarian/guzhen/zg-1-243h.idp.fid
 Pulse Sequence: s2pul
 Solvent: cdc13
 Temp: 22.0 C / 295.1 K
 Operator: guzhen
 File: zg-1-243h.idp
 INOVA-500 "nmrserver"

Relax delay 3.000 sec
 Pulse 48.5 degrees
 Acq. time 2.000 sec
 Width 51.1 Hz
 16 repetitions
 OBSERVE H1, 399.9486576 MHz
 DATA PROCESSING
 Line broadening 0.1 Hz
 FT size 65536
 Total time 16 min, 43 sec



zg-1-222h.500
 File: nmr500/Zakarian/guzhen/zg-1-222h.500.fid
 Pulse Sequence: s2pul
 Solvent: cdc13
 Temp: 22.0 C / 295.1 K
 Operator: guzhen
 File: zg-1-222h.500
 INOVA-500 "nmrserver"

Relax delay 2.000 sec
 Pulse 56.8 degrees
 Acq. time 2.668 sec
 Width 5997.0 Hz
 32 repetitions
 OBSERVE H1, 399.8563514 MHz
 DATA PROCESSING
 Resol. enhancement -0.0 Hz
 FT size 32768
 Total time 2 min, 30 sec

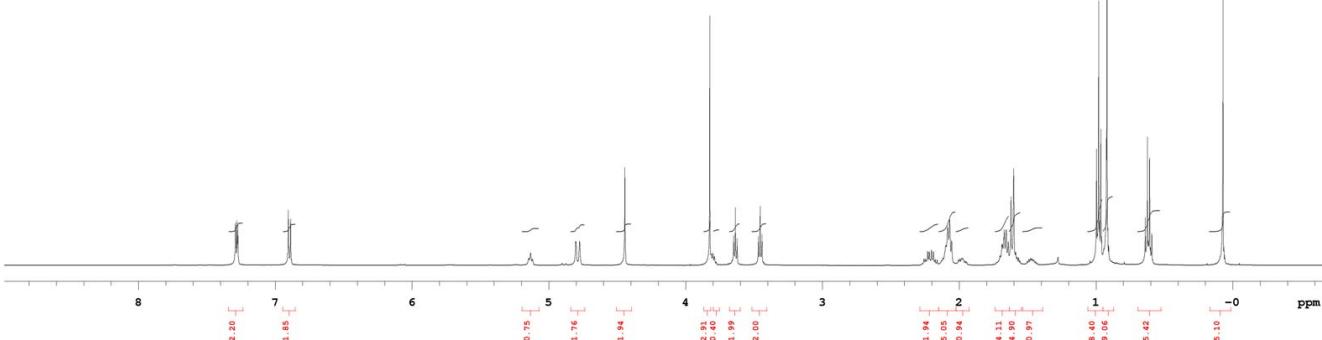
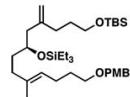


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sg-1-145-1h.500
File: nmr500/Zakarian/guzhen/zg-1-145-1h.500.fid
Pulse Sequence: s2pul
Solvent: cdc13
Temp: 22.0 C / 295.1 K
Operator: guzhen
File: zg-1-145-1h.500
INNOVA-500 "nmrserver"

```

Relax delay 2.000 sec
Pulse 56.8 degrees
Acq. time 2.668 sec
Width 1.6 Hz
16 repetitions
OBSERVE H1, 499.8563514 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 2 min, 30 sec

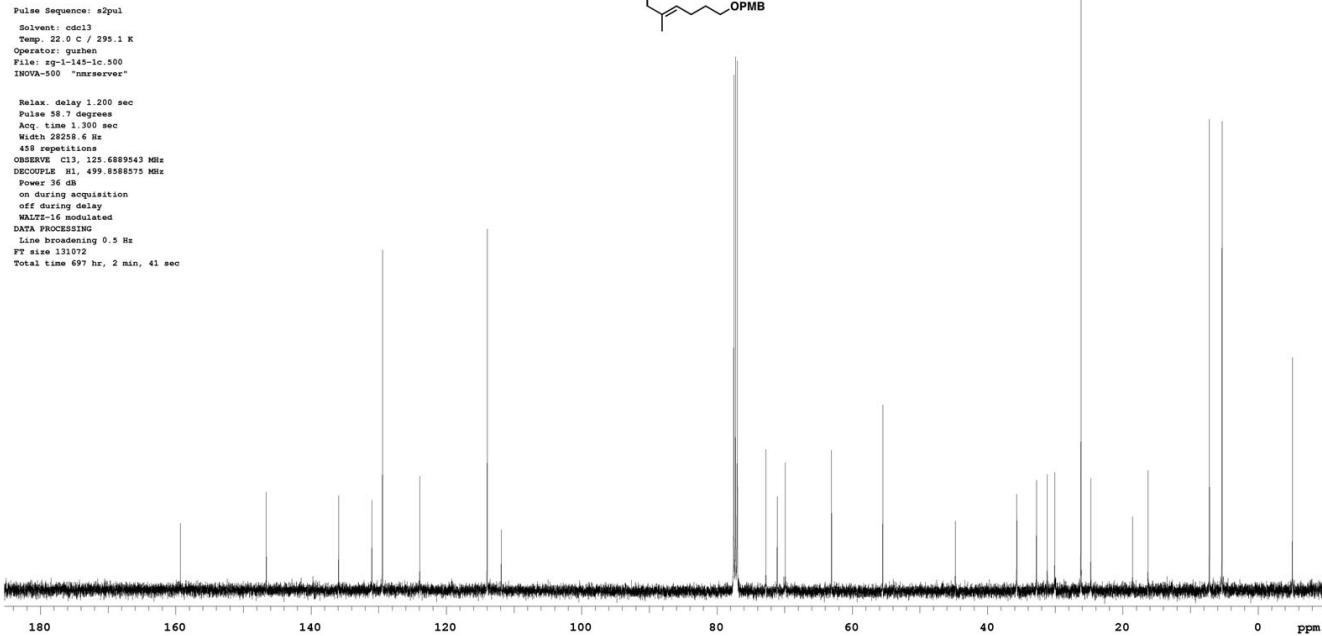
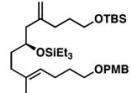


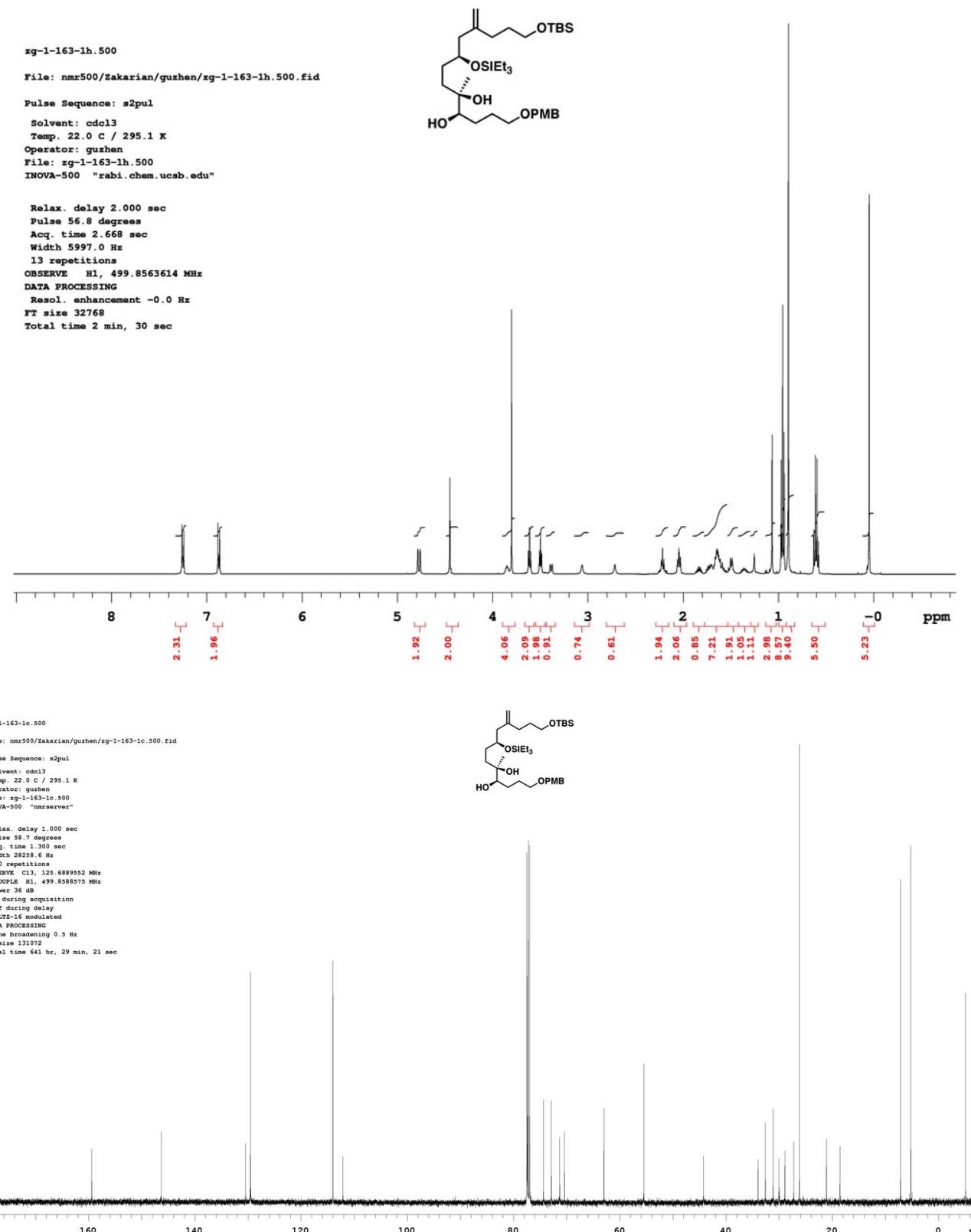
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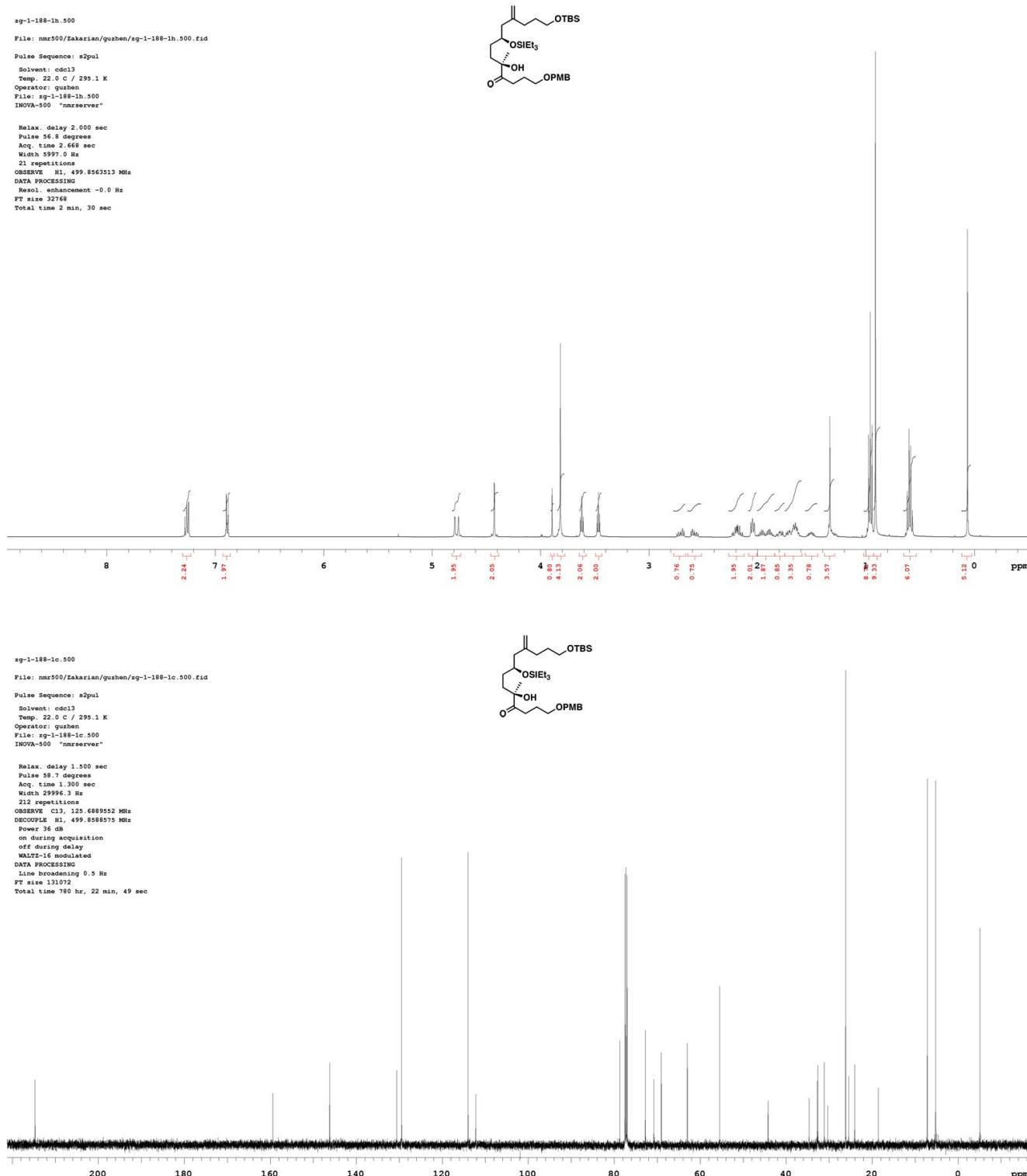
sg-1-145-1c.500
File: nmr500/Zakarian/guzhen/zg-1-145-1c.500.fid
Pulse Sequence: s2pul
Solvent: cdc13
Temp: 22.0 C / 295.1 K
Operator: guzhen
File: zg-1-145-1c.500
INNOVA-500 "nmrserver"

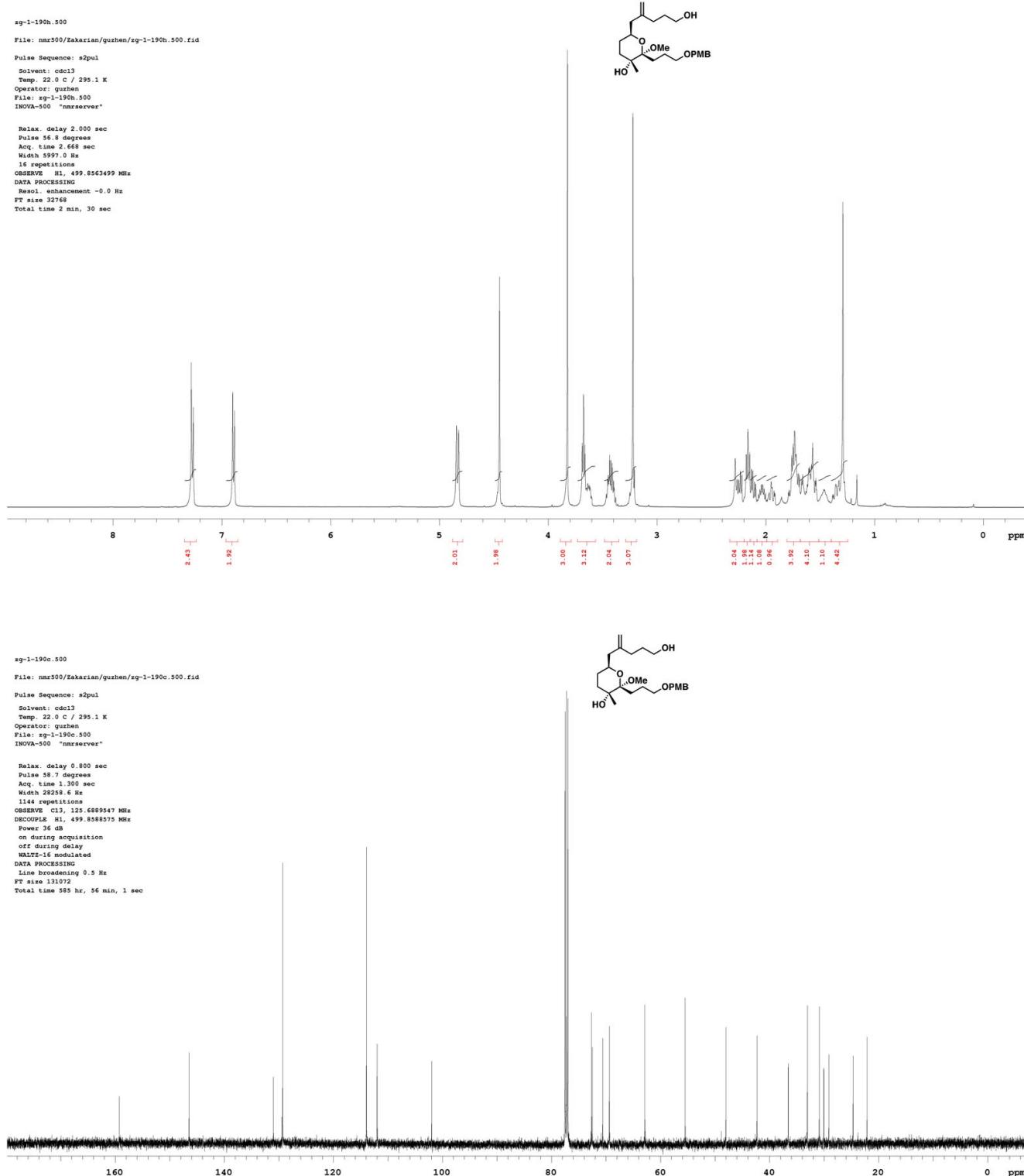
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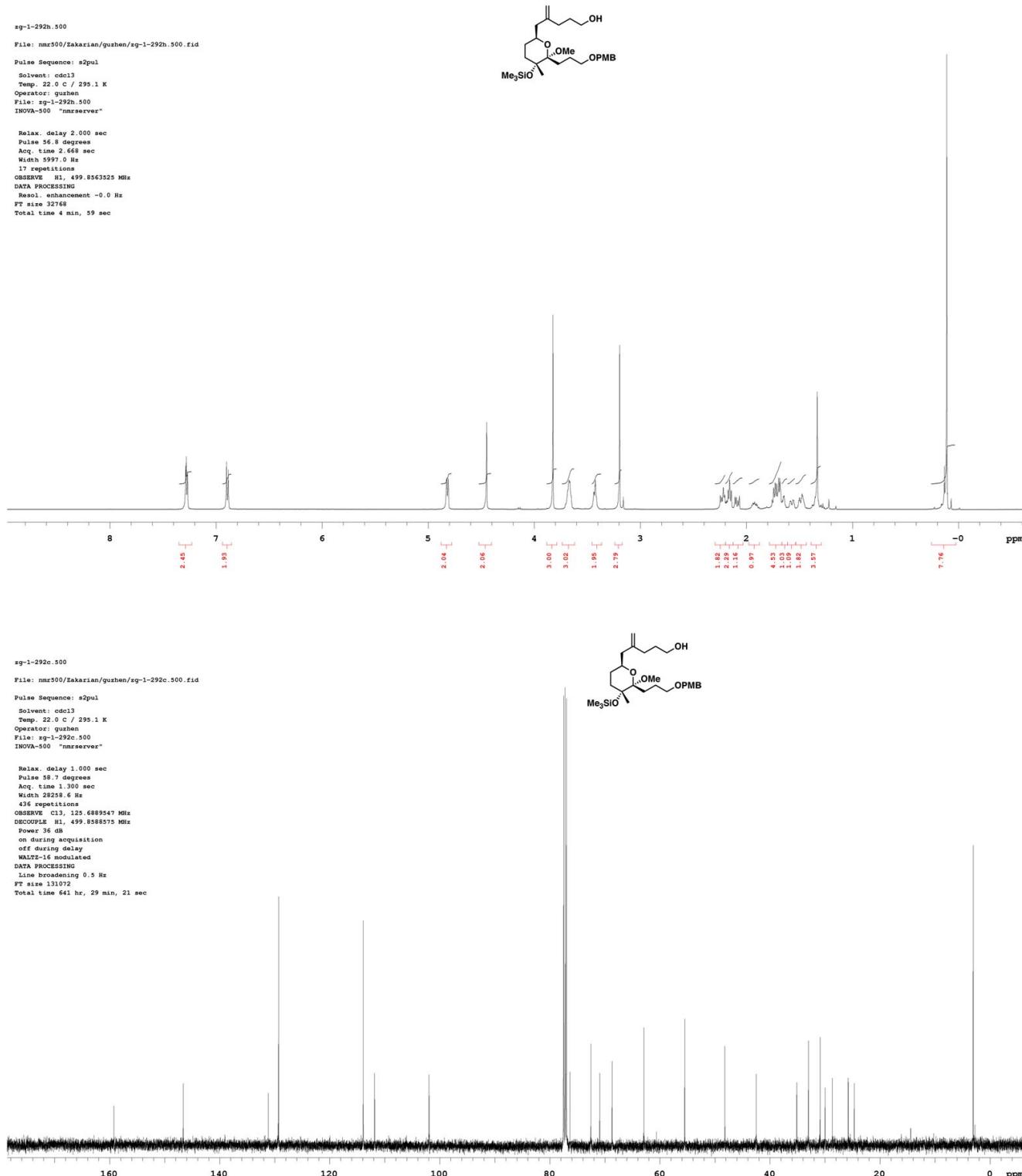
Relax delay 1.200 sec
Pulse 59.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
458 repetitions
OBSERVE C13, 131.6889543 MHz
DECODED, 499.8588573 MHz
Power 36 dB
on during acquisition
off during delay
SW1=16 mHz/detected
Data points=131072
Line broadening 0.5 Hz
FT size 131072
Total time 697 hr, 2 min, 41 sec





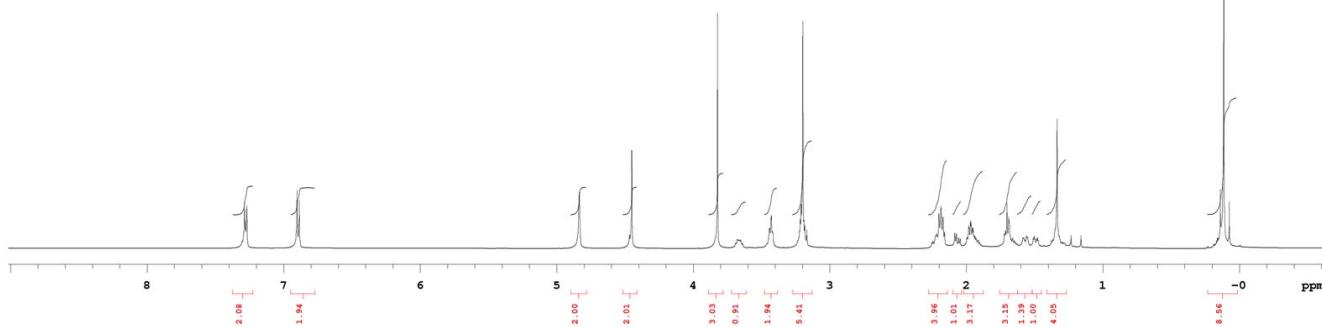
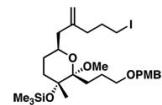






sg-1-228h.500
File: nmr500/Zakarian/guzhen/sg-1-228h.500.fid
Pulse Sequence: s2pul
Solvent: cdc13
Temp: 22.0 C / 295.1 K
Operator: guzhen
File: sg-1-228h.500
INNOVA-500 "nmrserver"

Relax delay 3.000 sec
Pulse 81.1 degrees
Acq. time 2.732 sec
Width 1.00 Hz
16 repetitions
OBSERVE: H1, 499.8563515 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 6 min, 7 sec



sg-1-228c.500
File: nmr500/Zakarian/guzhen/sg-1-228c.500.fid
Pulse Sequence: s2pul
Solvent: cdc13
Temp: 22.0 C / 295.1 K
Operator: guzhen
File: sg-1-228c.500
INNOVA-500 "nmrserver"

Relax delay 1.000 sec
Pulse 59.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
220 repetitions
OBSERVE: C13, 131.6889862 MHz
DATA PROCESSING, 499.8568573 MHz
Power 36 dB
on during acquisition
off during delay
SW1=16 nscaited
DATA POINTS=1200
Line broadening 0.5 Hz
FT size 131072
Total time 641 hr, 29 min, 21 sec

