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De Novo Asymmetric Synthesis of Biologically Active Carbohydrate Natural Products and Related SAR studies

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Supporting Spectra

for

**De Novo Asymmetric Synthesis of Biologically Active Carbohydrate
Natural Products and Related SAR studies**

Bulan Wu

**Dissertation submitted to the
Eberly College of Arts and Sciences
at West Virginia University
in partial fulfillment of the requirements
for the degree of**

**Doctor of Philosophy
In
Organic Chemistry**

**John H. Penn, Ph.D., Chair
George A. O'Doherty, Ph.D.
Kung Wang, Ph.D.
Alan Stolzenberg, Ph.D.
Yon Rojanasakul, Ph.D.**

C. Eugene Bennett Department of Chemistry

**Morgantown, West Virginia
2011**

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¹H NMR (600MHz, CDCl₃)

^{13}C NMR (150 MHz, CDCl_3)

¹H NMR (600MHz, CDCl₃)

^{13}C NMR (150 MHz, CDCl_3)

¹³C NMR (150 MHz, CDCl₃)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

¹H NMR (600MHz, CDCl₃)

^{13}C NMR (150 MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

¹H NMR (600MHz, CDCl₃)

¹³C NMR (150 MHz, CDCl₃)

^{13}C NMR (150 MHz, CD_3OD)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: szpul

Solvent: CDCl₃
Temp. 28.0 C / 301.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec

Pulse 30.0 degrees

Acq. time 4.000 sec

Width 9594.6 Hz

4 repetitions

OBSERVE H1, 599.6670065 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 131072

Total time 0 min, 20 sec

¹H NMR
(600MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: szpu1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inova600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

912 repetitions

OBSERVE C13, 150.7863836 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 53 hr, 1 min, 30 sec

¹³C NMR
(150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 28.0 C / 301.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq time 4.000 sec
Width 9594.6 Hz
4 repetitions
OBSERVE H1, 599.6670063 MHz
DATA PROCESSING
Line broadening 0.2 Hz
F1 size 131072
Total time 0 min, 20 sec

¹H NMR
(600MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp: 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inova600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

80 repetitions

OBSERVE C13, 150.7863849 MHz

DECOUPLE H1, 589.670024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3
Temp: 28.0 C / 301.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz

4 repetitions

OBSERVE H1, 599.6670060 MHz
DATA PROCESSING

Line broadening 0.2 Hz
FT size 131072

Total time 0 min, 20 sec

¹H NMR (600 MHz, CDCl₃)

1.242

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inova600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

176 repetitions

OBSERVE C13, 150.7863843 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

¹³C NMR of (150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmr.sys/data
Sample directory:
File: PROTON

Pulse Sequence: szpu1

Solvent: CDCl₃
Temp. 28.0 C / 301.1 K
INOVA-600 "inovae600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz

4 repetitions

OBSERVE H1, 599.6670060 MHz
DATA PROCESSING

Line broadening 0.2 Hz

FT size 131072

Total time 0 min, 20 sec

¹H NMR
(600 MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
User: 1-14-87
INOVA-600 "inova600"

Relax. delay 0.500 sec
Pulse 29.9 degrees
Acq. time 1.400 sec
Width 40000.0 Hz
5280 repetitions
OBSERVE C13, 150.7863843 MHz
DECOUPLE H1, 599.6700024 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 53 hr, 1 min, 30 sec

¹³C NMR
(150 MHz, CDCl₃)

2

4.

File: PROTON

4.

4.

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4.

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INNOVA-600 "inovav600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

144 repetitions

OBSERVE C13, 150.7863855 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 53 hr, 1 min, 30 sec

¹³C NMR
(150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
INNOVA-600 "innova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz
4 repetitions
OBSERVE H1, 599.6670063 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 131072
Total time 0 min, 20 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: szpu1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inova600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

80 Repetitions

OBSERVE C13, 150.7863855 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FI size 131072

Total time 32 min, 34 sec

¹³C NMR
(150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory:
 File: PROTON

Pulse Sequence: szpu1

Solvent: CDCl₃
 Temp. 28.0 C / 301.1 K
 INOVA-600 "inovae600"

Relax. delay 1.000 sec
 Pulse 30.0 degrees
 Acq. time 4.000 sec

Width 9594.6 Hz

4 repetitions

OBSERVE H1, 599.6670062 MHz
 DATA PROCESSING

Line broadening 0.2 Hz

FT size 131072

Total time 0 min, 20 sec

¹H NMR
 (600 MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
User: 1-14-87
INOVA-600 "inova600"

Relax. delay 0.500 sec
Pulse 29.9 degrees
Acq. time 1.400 sec
Width 40000.0 Hz
48 repetitions
OBSERVE C13, 150.7863867 MHz
DECUPLE H1, 599.6700024 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 32 min, 34 sec

¹³C NMR
(150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmr.sys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
File: tftcyclo-pd-top
INDVA-600 "nmr"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz
4 repetitions
OBSERVE H1, 599.6670084 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FI Size 131072
Total time 0 min, 20 sec

¹H NMR of (17)
(600 MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: szpul
Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
User: 1-14-87
File: tr1cyc10-pd-less-C
INOVA-600 "nmr."

Relax. delay 0.500 sec
Pulse 29.9 degrees
Acq. time 1.400 sec
Width 40000.0 Hz
1856 repetitions
OBSERVE C13, 150.7863855 MHz
DECOUPLE H1, 599.6700024 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 530 hr, 15 min, 3 sec

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROT0N

Pulse Sequence: s2pu1

Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
INOVA-600 "inovab600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz

Single scan

OBSERVE HI, 599.6670085 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 131072

Total time 0 min, 5 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
User: 1-14-87
INOVA-600 "inovae600"

Relax. delay 0.500 sec
Pulse 29.9 degrees
Acq. time 1.400 sec
Width 40000.0 Hz
672 repetitions
OBSERVE C13, 150.7863873 MHz
DECOUPLE H1, 599.6700024 MHz
Power 40 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 32 min, 34 sec

¹³C NMR
(150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:

Pulse Sequence: szpul

Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
File: 020409-1H
INOVA-600 "nmr"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz
Single scan
OBSERVE H1, 599.6670079 MHz
DATA PROCESSING
FT size 131072
Total time 0 min, 5 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
 Solvent: CDCl₃
 Temp: 28.0 C / 301.1 K
 User: 1-14-87
 INOVA-600 "nmr"
 Relax. delay 0.500 sec
 Pulse 29.9 degrees
 Acq. time 1.400 sec
 Width 36003.6 Hz
 64 repetitions
 OBSERVE C13, 150.7863852 MHz
 DECOUPLE H1, 599.6700024 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 32 min, 34 sec

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp. 28.0 C / 301.1 K

File: 020609-1H

INOVA-600 "nmr"

Relax. delay 1.000 sec

Pulse 30.0 degrees

Acq. time 4.000 sec

Width 9594.6 Hz

Single scan

OBSERVE HI 599.6670071 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 131072

Total time 0 min, 5 sec

¹H NMR
(600 MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: szpul

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INVOA-600 "inovae600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 36003.6 Hz

336 repetitions

OBSERVE C13, 150.7863852 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

¹³C NMR
(150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 28.0 C / 301.1 K
INOVA-600 "inovab600"

Relax. delay: 1.000 sec
Pulse: 30.0 degrees
Acq. time: 4.000 sec
Width: 9394.6 Hz
4 repetitions
OBSERVE: H1, 599.6670076 MHz
DATA PROCESSING
Line broadening: 0.2 Hz
F1 size: 131072
Total time: 0 min, 20 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: s2put

Solvent: CDC13
Temp: 29.0 C

Temp. 28.0 C / 301.1 K
User: 1-14-87

User: 1-14-87

INOVA-600 "inova600"

Relax. delay 0.500 sec
Pulse 29 degrees

Pulse 29.9 degrees
Acq. time 1.400 sec

Width 36003.6 Hz
288 repetitions

OBSERVE C13, 150

DECUPLE HI, 599
Power 40 dB

continuously on
WALTZ-16 modulat

DATA PROCESSING

Line broadening
FT size 131072

Total time 32 min

^1H NMR of Cleistrioside-6
(600 MHz, CD_3OD)

¹³C NMR of Cleistroidide-6
(150 MHz, CD₃OD)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: szpu1

Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz
4 repetitions
OBSERVE H1, 599.6670076 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 131072
Total time 0 min, 20 sec

¹H NMR
(600 MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INNOVA-600 "inovae00"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

144 repetitions

OBSERVE C13, 150.7863855 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

¹³C NMR
(150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrSYS/data
 Sample directory:
 File: PROTON

Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp: 28.0 C / 301.1 K
 INOVA-600 "inovas600"

Relax. delay 1.000 sec
 Pulse 30.0 degrees
 Acq. time 4.000 sec
 Width 9594.6 Hz
 16 repetitions
 OBSERVE H1, 599.6670079 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 131072
 Total time 1 min, 20 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INNOVA-600 "inovae600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

336 repetitions

OBSERVE C13, 150.786385 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 53 hr, 1 min, 30 sec

¹³C NMR (150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 28.0 C / 301.1 K
INOVA-600 "inovas60"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz
4 repetitions
OBSERVE H1, 599.6670081 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 131072
Total time 0 min, 20 sec

¹H NMR (600 MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: s2put1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inova600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

928 repetitions

OBSERVE C13, 150.7863855 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory:
File: PROTON

Pulse Sequence: szpu1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

INOVA-600 "inova600"

Relax. delay 1.000 sec

Pulse 30.0 degrees

Acq. time 4.000 sec

Width 9594.6 Hz

4 Repetitions

OBSERVE H1, 599.6670084 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 131072

Total time 0 min, 20 sec

¹H NMR
(600 MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: szpu1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inova600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

1008 repetitions

OBSERVE C13, 150.7863836 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

¹³C NMR
(150 MHz, CDCl₃)

^{13}C NMR
(150 MHz, CD_3OD)

¹H NMR
(600 MHz, CDCl₃)

¹H NMR of Cleistroside-4
(600 MHz, CD₃OD)

^{13}C NMR of Cleistrose-4
(150 MHz, CD_3OD)

^{13}C NMR
(150 MHz, CD_3OD)

¹H NMR of Cleistetoside-7
(600 MHz, CD₃OD)

^{13}C NMR of Cleistroside-7
(150 MHz, CD_3OD)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory:
 File: PROTON

Pulse Sequence: szpu1
 Solvent: CDCl3
 Temp: 28.0 C / 301.1 K
 INOVA-600 "inovas600"

Relax. delay 1.000 sec
 Pulse 30.0 degrees
 Acq. time 4.000 sec
 Width 9594.6 Hz
 4 repetitions
 OBSERVE H1, 599.6670074 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 131072
 Total time 0 min, 20 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: szpul

Solvent: CDCl₃

Temp. 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inovae600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

240 repetitions

OBSERVE C13, 150.7863843 MHz

DECUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

¹³C NMR of (150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 28.0 C / 301.1 K
INOVA-600 "inovaa600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz
4 repetitions
OBSERVE H1, 599.6670081 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 131072
Total time 0 min, 20 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: szpul

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inovas600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

1760 Repetitions

OBSERVE: C13, 150.7863849 MHz

DECUPLE: H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 21 hr, 12 min, 36 sec

^1H NMR of Cleistroside-3
(600 MHz, CD_3OD)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmr-sys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 28.0 C / 301.1 K
INOVA-600 "inovas600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz

Single scan
OBSERVE H1, 599.6670079 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 131072
Total time 0 min, 5 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INDVA-600 "inovav600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 40000.0 Hz

2912 repetitions

OBSERVE C13, 150.7863843 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 5 hr, 18 min, 9 sec

¹³C NMR
(150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: szpu1
Solvent: CDCl3
Temp: 28.0 C / 301.1 K
INOVA-600 "inovab600"

Relax. delay 1.000 sec
Pulse: 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz
4 repetitions
OBSERVE H1, 599.6670081 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 131072
Total time 0 min, 20 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 28.0 C / 301.1 K
User: 1-14-87
INNOVA-600 "inov600"

Relax. delay 0.500 sec
Pulse 29.9 degrees
Acq. time 1.400 sec
Width 40000.0 Hz
1536 repetitions
OBSERVE CH3, 150.7863843 MHz
DECUPLE H1, 599.6700024 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 53 hr, 1 min, 30 sec

¹³C NMR
(150 MHz, CDCl₃)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 28.0 C / 301.1 K
INOVA-600 "inovaa600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz
8 repetitions
OBSERVE H1, 599.6670082 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 131072
Total time 2 min, 40 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: szpul

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inova600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 36003.6 Hz

240 repetitions

DECOUPLE C13, 150.7863846 MHz

Power 40 dB

continuously on

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

¹³C NMR
(150 MHz, CDCl₃)

¹H NMR of Cleistetroside-6
(600 MHz, CD₃OD)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
File: 020909-1H
INOVA-600 "nmr"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz

4 repetitions

OBSERVE H1, 599.6670079 MHz
DATA PROCESSING

Line broadening 0.2 Hz
FT size 131072

Total time 0 min, 20 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: szpu1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

File: 020909-1C

INOVA-600 "nmr"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 36003.6 Hz

240 repetitions

OBSERVE C13, 150.7863857 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 47133 hr, 24 min, 0 sec

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmr-sys/data
 Sample directory:
 File: PROTON

Pulse Sequence: szpu1
 Solvent: CDCl3
 Temp: 28.0 C / 301.1 K
 INOVA-600 "inovab600"

Relax. delay: 1.000 sec
 Pulse: 30.0 degrees
 Acq. time: 4.000 sec
 Width: 3394.6 Hz

4 repetitions

OBSERVE: H1, 599.6670076 MHz
 DATA PROCESSING

Line broadening: 0.2 Hz
 FT size: 131072
 Total time: 0 min, 20 sec

Pulse Sequence: szpu1
Solvent: CDCl3
Temp. 28.0 C / 301.1 K
User: 1-14-87
INOVA-600 "1nova600"

Relax. delay 0.500 sec
Pulse 29.9 degrees
Acq. time 1.400 sec
Width 36003.6 Hz
224 repetitions
OBSERVE CH 139.7863868 MHz
DECUPLE H1, 539.6700024 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 32 min, 34 sec

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:

Pulse Sequence: szpul

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

File: 021309-1H

INOVA-600 "inova600"

Relax. delay 1.000 sec

Pulse 30.0 degrees

Acq. time 4.000 sec

Width 9594.6 Hz

4 repetitions

OBSERVE H1, 599.6670039 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 131072

Total time 0 min, 20 sec

¹H NMR
(600 MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

File: 021309-1C

INOVA-600 "inova600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 36003.6 Hz

240 repetitions

OBSERVE C13, 150.7863857 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

File: 021609-7H

INDVA-600 "inova600"

Relax. delay 1.000 sec

Pulse 30.0 degrees

Acq. time 4.000 sec

Width 9394.6 Hz

4 repetitions

OBSERVE H1, 599.6670081 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 131072

Total time 0 min, 20 sec

¹H NMR
(600 MHz, CDCl₃)

STANDARD CARBON PARAMETERS

Pulse Sequence: szpu1

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

INOVA-600 "inova600"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 36003.6 Hz

224 repetitions

OBSERVE C13, 150.7863857 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

¹³C NMR
(150 MHz, CDCl₃)

¹H NMR of Cleistroside-NEW I
(600 MHz, CD₃OD)

¹³C NMR of Cleistrotoside-NEW1
(150 MHz, CD₃OD)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp: 28.0 C / 301.1 K
File: 021609-13H
INOVA-600 "nmr"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 9594.6 Hz
4 repetitions
OBSERVE H1 593.6670081 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 131072
Total time 0 min, 20 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp: 28.0 C / 301.1 K

User: 1-14-87

File: 021609-13C

INOVA-600 "nmr"

Relax. delay 0.500 sec

Pulse 29.9 degrees

Acq. time 1.400 sec

Width 36003.6 Hz

160 repetitions

OBSERVE C13, 150.7863846 MHz

DECOUPLE H1, 599.6700024 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min, 34 sec

¹³C NMR (150 MHz, CDCl₃)

¹H NMR of Cleistroidide-NEW2
(600 MHz, CD₃OD)

¹³C NMR of Cleistetoside-NEW2
(150 MHz, CD₃OD)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

¹H NMR (600MHz, CDCl₃)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, C_6D_6)

^{13}C NMR (150 MHz, C_6D_6)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

¹³C NMR (150 MHz, CDCl₃)

¹H NMR (600MHz, CDCl₃)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, C_6D_6)

^{13}C NMR (150 MHz, C_6D_6)

¹H NMR (600MHz, CDCl₃)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, $\text{CDCl}_3 + \text{CD}_3\text{OD}$)

^{13}C NMR (150 MHz, $\text{CDCl}_3 + \text{CD}_3\text{OD}$)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^1H NMR (600MHz, CDCl_3)

^{13}C NMR (150 MHz, CDCl_3)

^1H NMR (600MHz, CD_3OD)

^{13}C NMR (150 MHz, CD_3OD)

^{13}C NMR (150 MHz, CDCl_3)

^{13}C NMR (150 MHz, CD_3OD)