A Concise Total Synthesis of (−)-Maoecrystal Z

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Supporting Information 2 (Spectral Data):
Sample Name: jc-1-245-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-245-A
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 31 2011

Temp. 25.0 C / 298.1 K
Sample #27, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 52 sec
Sample Name: jc-1-245-A

Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-245-A
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 31 2011

Temp. 25.0 C / 298.1 K
Sample #27, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
256 repetitions

OBSERVE C13, 125.6569604 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 8 min 45 sec

ppm

Plotname: --Not assigned--

OTBS

SI-1

200 180 160 140 120 100 80 60 40 20 0 ppm
Sample Name: jc-1-246-B

Data Collected on: indy.caltech.edu-inova500

Archive directory: /home/jcha/vnmrsys/data

Sample directory: jc-1-246-B

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 1 2011

Temp. 25.0 C / 298.1 K

Sample #46, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

OBSERVE H1, 499.7290203 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 3 min 45 sec

Plotname: --Not assigned--

major diastereomer
Sample Name: jc-1-246-B
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-246-B
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 1 2011

Temp. 25.0 C / 298.1 K
Sample #46, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions

OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min
Sample Name: jc-1-246-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-246-A
FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 4 2011

Temp. 25.0 C / 298.1 K
Sample #46, Operator: jcha
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7290200 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min 45 sec

minor diastereomer
Sample Name: jc-1-246-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-246-A
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 4 2011

Temp. 25.0 C / 298.1 K
Sample #46, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min

--Not assigned--

O

13

minor diastereomer
Sample Name: jtsy-v-287f15char1
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jtsy-v-287f15char1
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 26 2011

Temp. 25.0 C / 298.1 K
Sample #14, Operator: yeomanjo

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec

O

OTBS

12

ppm

S33
Sample Name: jtsy-v-287f15char1
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jtsy-v-287f15char1
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Apr 26 2011

Temp. 25.0 C / 298.1 K
Sample #14, Operator: yeomanjo

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Sample Name: jtsy-vi-183rf12-15
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jtsy-vi-183rf12-15
FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 21 2011

Sample #14, Operator: yeomanjo

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE  H1, 499.7225128 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec
Sample Name: jtsy-vi-183rf12-15
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jtsy-vi-183rf12-15
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Jul 21 2011

Sample #14, Operator: yeomanjo
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1700 repetitions
OBSERVE C13, 125.6553262 MHz
DECOUPLE H1, 499.7250019 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 58 min
Sample Name: dep-spirolactone-rf
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: dep-spirolactone-rf
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 20 2011

Sample #33, Operator: yeomanjo
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7225033 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec

Plotname: --Not assigned--

[Chemical structure image]

SI-2

ppm
Sample Name: dep-spirolactone-rf
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: dep-spirolactone-rf
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 20 2011

Sample #33, Operator: yeomanjo

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1160 repetitions
OBSERVE C13, 125.6553291 MHz
DECOUPLE H1, 499.7250019 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 58 min
Sample Name: jc-1-210-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-210-A
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 8 2011

Temp. 25.0 C / 298.1 K
Sample #41, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7290121 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min 45 sec

Plotname: --Not assigned--
Sample Name: jc-1-210-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-210-A
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 8 2011

Temp. 25.0 C / 298.1 K
Sample #41, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6569729 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min

![NMR Spectrum Image](image-url)

Plotname: --Not assigned--
Sample Name: jc-1-231-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-231-A
FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 2 2011

Temp. 25.0 C / 298.1 K
Sample #33, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7290190 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min 45 sec

Plotname: --Not assigned--
Sample Name: jc-1-231-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-231-A

FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 2, 2011

Temp. 25.0 C / 298.1 K
Sample #33, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions

OBSERVE C13, 125.6569671 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min
Sample Name: jc-1-241-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-241-A
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 21 2011

Temp. 25.0 C / 298.1 K
Sample #33, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions

OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 52 sec
Sample Name: jc-1-241-A-carbon
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-241-A-carbon
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 21 2011

Temp. 25.0 C / 298.1 K
Sample #33, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Sample Name: jc-1-251-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-251-A
FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Apr 13 2011

Temp. 25.0 C / 298.1 K  
Sample #40, Operator: jcha  
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 2.500 sec  
Width 8000.0 Hz  
32 repetitions  
OBSERVE H1, 499.7290203 MHz  
DATA PROCESSING  
Line broadening 0.2 Hz  
FT size 65536  
Total time 1 min 52 sec
Sample Name: jc-1-251-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-251-A
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 13 2011

Temp. 25.0 C / 298.1 K
Sample #40, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min
Sample Name: jc-1-253-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-253-A
FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 20 2011

Sample #1, Operator: jcha
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7225125 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec

Plotname: --Not assigned--

less polar diastereomer
Sample Name: jc-1-253-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-253-A
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 20 2011

Sample #1, Operator: jcha

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6553262 MHz
DECOUPLE H1, 499.7250019 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 50 min
Sample Name: jc-1-253-B
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-253-B
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 13 2011

Temp. 25.0 C / 298.1 K
Sample #15, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 52 sec

more polar diastereomer

ppm
Sample Name: jc-1-253-B
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-253-B
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 13 2011

Temp. 25.0 °C / 298.1 K
Sample #15, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min

more polar diastereomer
Sample Name: jc-1-254-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-254-A
FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 17 2011

Temp. 25.0 °C / 298.1 K
Sample #15, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7290200 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min 45 sec
Sample Name: jc-1-254-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-254-A
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Apr 17 2011

Temp. 25.0 C / 298.1 K
Sample #15, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min
Sample Name: jc-1-256-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-256-A
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 19 2011

Temp. 25.0 C / 298.1 K
Sample #46, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE  H1, 499.7290208 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min 45 sec
Sample Name: jc-1-256-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-256-A
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 19 2011

Temp. 25.0 C / 298.1 K
Sample #46, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions

OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min
Sample Name: jc-1-257-A

Data Collected on:
indy.caltech.edu-inova500

Archive directory:
/home/jcha/vnmrsys/data

Sample directory:
jc-1-257-A

Pulse File: PROTON04

Pulse Sequence: PROTON (s2pul)
Solvent: cdd3

Data collected on: Jul 10 2011

Sample #2, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions

OBSERVE H1, 499.7290205 MHz

DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 56 sec
Sample Name: jc-1-257-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-257-A
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 10 2011
Sample #2, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1250 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 42 min
Sample Name: jc-1-258-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-258-A
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 3 2011

Temp. 25.0 C / 298.1 K
Sample #7, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
128 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 29 sec
Sample Name: jc-1-258-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-258-A
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: May 3 2011

Temp. 25.0 C / 298.1 K
Sample #7, Operator: jcha
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
COSY of SI-5
HSQC of SI-5
HMBC of SI-5
NOESY of SI-5
Sample Name: jtsy-vi-105hi
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jtsy-vi-105hi
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 6 2011

Temp. 25.0 C / 298.1 K
Sample #12, Operator: yeomanjo

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 0 sec

Plotname: --Not assigned--

TBSO

less polar
diastereomer
Sample Name: jtsy-vi-105hi
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jtsy-vi-105hi
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 6 2011

Temp. 25.0 C / 298.1 K
Sample #12, Operator: yeomanjo

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions

OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Sample Name: jtsy-vi-129min46-47
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jtsy-vi-129min46-47
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: Jun 29 2011

Sample #20, Operator: yeomanjo
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7290394 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 24 sec

Plotname: --Not assigned--

more polar diastereomer
Sample Name: jsy-vi-129min46-47
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jsy-vi-129min46-47
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Jun 29 2011

Sample #18, Operator: yeomanjo
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3000 repetitions
OBSERVE C13, 125.6569623 MHz
DECouple H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 42 min

more polar diastereomer
Sample Name: jc-1-288-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-288-A
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 16 2011

Temp. 25.0 C / 298.1 K
Sample #46, Operator: jcha

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7290200 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 49 sec

ppm

Plotname: --Not assigned--
Sample Name: jc-1-288-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-288-A
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Jun 16 2011

Temp. 25.0 C / 298.1 K
Sample #46, Operator: jcha
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Sample Name: jc-1-272-A-char

Data Collected on: indy.caltech.edu-inova500

Archive directory: /home/jcha/vnmrsys/data

Sample directory: jc-1-272-A-char

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3

Data collected on: Jun 2 2011

Temp. 25.0 C / 298.1 K
Sample #2, Operator: jcha

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions

OBSERVE H1, 499.7290203 MHz

DATA PROCESSING

Line broadening 0.2 Hz
FT size 65536

Total time 4 min 49 sec
Sample Name: jc-1-272-A-char
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-272-A-char
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun  2 2011

Temp. 25.0 C / 298.1 K
Sample #2, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6569642 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min
Sample Name: jtsy-vi-163pHPLC
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jtsy-vi-163pHPLC
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 1 2011

Sample #14, Operator: yeomanjo
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7290166 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 24 sec
Sample Name: jtsy-vi-163pHPLC
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/yeomanjo/vnmrsys/data
Sample directory: jtsy-vi-163pHPLC
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 1 2011

Sample #14, Operator: yeomanjo

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
6000 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 hr, 25 min
COSY of 9
HSQC of 9
HMBC of 9
NOESY of 9
Sample Name: jc-1-274-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-274-A
FidFile: current

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 20 2011

Sample #2, Operator: jcha

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7225125 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 49 sec
Sample Name: jc-1-274-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-274-A
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 18 2011

Sample #46, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1725 repetitions
OBSERVE C13, 125.6553262 MHz
DECOUPLE H1, 499.7250019 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 58 min

OAc

H
O

OAc

Ac

O

30
Sample Name: jc-1-274-B
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-274-B
FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 26 2011

Sample #1, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7225125 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min 45 sec

Plotname: --Not assigned--
Sample Name: jc-1-274-B
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-274-B
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 26 2011

Sample #1, Operator: jcha
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2250 repetitions
OBSERVE C13, 125.6553271 MHz
DECOUPLE H1, 499.7250019 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 58 min
Sample Name: jc-1-276-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-276-A
FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 30 2011

Sample #1, Operator: jcha
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7290200 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 24 sec

![Chemical Structure Image]
Sample Name: jc-1-276-A
Data Collected on:
   indy.caltech.edu-inova500
Archive directory:
   /home/jcha/vnmrsys/data
Sample directory:
   jc-1-276-A
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 30 2011

Sample #1, Operator: jcha

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
9400 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 58 min
Sample Name: jc-1-277-pyridine
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-277-pyridine
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: pyridine
Data collected on: Aug 2 2011

Sample #46, Operator: jcha
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
128 repetitions
OBSERVE  H1, 499.7217632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 23 min

maocystal Z(6)
Sample Name: jc-1-277-pyridine
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-277-pyridine
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: pyridine
Data collected on: Aug 2 2011

Sample #1, Operator: jcha

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
15000 repetitions
OBSERVE C13, 125.6552985 MHz
DECOUPLE H1, 499.7248470 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 6 hr, 27 min

maocystal Z (6)
Sample Name: jc-1-277-cdcl3
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcha/vnmrsys/data
Sample directory: jc-1-277-cdcl3
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 2 2011

Sample #46, Operator: jcha
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
128 repetitions
OBSERVE H1, 499.7225128 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 23 min

maecrystal Z(6)
natural sample, from isolation paper:
*Org. Lett.* **2006**, *8*, 4727-4730

![Chemical Structure](image)

### Synthetic Sample

**1H NMR**
- *δ*: 0.63, 0.97, 1.02, 1.09, 1.10, 1.15, 1.16, 1.42, 1.73, 2.17, 2.60, 2.66, 2.80, 3.06, 3.39, 4.59, 4.65, 4.73, 5.39

**spectroscopic data**
- *λ*: 3.200 sec
- *O*: 60.0 °C
- *q. time*: 2.500 sec
- *dch*: 6000.0 Hz
- *N*: 8 repetitions
- *ENU*: 499.727632 MHz
- A **PROCESSING**
- *me broaden*: 0.2 Hz
- *size*: 45536
- *al time*: 23 min

**Maocrystal Z (6)**