Enantioselective Total Synthesis of (−)-Acetylaranotin, A Dihydrooxepine Epidithiodiketopiperazine

Julian A. Codelli, Angela L. A. Puchlopek, and Sarah E. Reisman*

The Warren and Katharine Schlinger Laboratory for Chemistry and Chemical Engineering, Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, California 91125
reisman@caltech.edu

Supporting Information 2 (NMR Spectra)
Sample Name: JAC6-67
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC6-67
FidFile: PROTON02

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

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

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7290200 MHz

DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: JAC6-67

Data Collected on:
indy.caltech.edu-inova500

Archive directory: /home/jcodelli/vnmrsys/data

Sample directory: JAC6-67

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdc13

Data collected on: Jan 18 2011

Temp. 25.0 C / 298.1 K

Sample #8, Operator: jcodelli

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

512 repetitions

OBSERVE C13, 125.6569681 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: ALP-III-28characterize
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: ALP-III-28characterize
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 12 2011

Sample #46, Operator: apuchlop

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.725135 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: ALP-II-157flash
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: ALP-II-157flash
FidFile: CARBON01

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

Sample #46, Operator: apuchlop

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 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 51 min
Sample Name: ALP-II-229
Sample directory: /home/apuchlop/vnmrsys/data
Archive directory: indy.caltech.edu-inova500
Data Collected on: ALP-II-229
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 7 2011
Temp. 25.0 C / 298.1 K
Sample #33, Operator: apuchlop
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7290200 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: ALP-II-229
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: ALP-II-229
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 7 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6569690 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
JAC6-79 CD3CN indy

Sample Name: JAC6-79
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC6-79
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Feb 19 2011

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

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

OBSERVE H1, 499.7316752 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 36 sec

Plotname: --Not assigned--
Sample Name: JAC6-79
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC6-79
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn
Data collected on: Feb 19 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6575114 MHz
DECOUPLE H1, 499.7341599 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
JAC6-85 CD3CN indy

Sample Name: JAC6-85
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC6-85
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Feb 20 2011

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

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7316752 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 36 sec

Plotname: --Not assigned--
Sample Name: JAC6-85

Data Collected on: inova500

Archive directory: /home/jcodelli/vnmrsys/data

Sample directory: JAC6-85

FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn

Data collected on: Feb 20 2011

Temp. 25.0 C / 298.1 K

Sample #7, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
5000 repetitions

OBSERVE C13, 125.6575066 MHz

DECOUPLE H1, 499.7341599 MHz
Power 39 dB
continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz
FT size 65536
Total time 2 hr, 50 min
ALP-II-175characterize

Sample Name: ALP-II-175characterize
Data Collected on:indy.caltech.edu-inova500
Archive directory:/home/apuchlop/vnmrsys/data
Sample directory:ALP-II-175characterize
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Jun 14 2011

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

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7316752 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec

Plotname: --Not assigned--

MeO₂C
Ph
N
CO₂Et
Teoc
S-4
Sample Name: ALP-II-175characterize
Data Collected on: inova500
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: ALP-II-175characterize
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn
Data collected on: Jun 15 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
7000 repetitions
OBSERVE C13, 125.6575066 MHz
DECOUPLE H1, 499.7341599 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 hr, 59 min
Sample Name: JAC6-113_115
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC6-113_115
FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)
Solvent: dmso
Data collected on: Apr 27 2011

Temp. 60.0 C / 333.1 K
Sample #9, Operator: jcodelli

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7966888 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 0 sec
Sample Name: JAC6-113_115
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC6-113_115
FidFile: CARBON02
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Apr 27 2011

Temp. 60.0 C / 333.1 K
Sample #9, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
256 repetitions
OBSERVE C13, 100.5289808 MHz
DECOUPLE H1, 399.7987005 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 9 min 45 sec
Sample Name: JAC7-93
Data Collected on:indy.caltech.edu-inova500
Archive directory:/home/jcodelli/vnmrsys/data
Sample directory:JAC7-93
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 29 2011

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

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7290200 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 0 sec
Sample Name: JAC7-93
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-93
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 29 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 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 51 min
ALP-III-17characterize

Sample Name: ALP-III-17characterize
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: ALP-III-17characterize
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 27 2011

Sample #47, Operator: apuchlop

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.725128 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: ALP-III-17characterize
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: ALP-III-17characterize
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 27 2011

Sample #47, Operator: apuchlop

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 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 51 min
JAC3-215A CDC13 indy

Sample Name: JAC3-215A
Data Collected on: inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC3-215A
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 21 2010

Temp. 25.0 C / 298.1 K
Sample #8, Operator: jcodelli

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 53 sec
Sample Name: JAC3-215A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC3-215A
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 21 2010
Temp. 25.0 C / 298.1 K
Sample #8, Operator: jcodelli
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1600 repetitions
OBSERVE  C13, 125.6602433 MHz
DECOUPLE  H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 54 min
JAC7-97 CD3CN indy

Sample Name:
JAC7-97

Data Collected on:
indy.caltech.edu-inova500

Archive directory:
/home/jcodelli/vnmrsys/data

Sample directory:
JAC7-97

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn

Data collected on: May 1 2011

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

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions

OBSERVE H1, 499.7316752 MHz

DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 0 sec

OBSERVATION

Plotname: --Not assigned--
JAC7-97 CD3CN indy

Sample Name: JAC7-97
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-97
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn
Data collected on: May 1 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions
OBSERVE C13, 125.6575076 MHz
DECOUPLE H1, 499.7341599 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 47 min

\[
\text{Plotname: --Not assigned--}
\]

\[
\begin{align*}
\text{HO} & \quad \text{OH} \\
\text{HO} & \quad \text{H} \quad \text{19} \\
\text{Teoc} & \quad \text{CO}_2\text{Et}
\end{align*}
\]
Sample Name: JAC7-99
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-99
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: May 6 2011

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

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7316752 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 0 sec
Sample Name: JAC7-99
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-99
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn
Data collected on: May 6 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions
OBSERVE C13, 125.6574961 MHz
DECOUPLE H1, 499.7341599 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 47 min
ALP-II-263 characterize?

Sample Name: ALP-II-263 characterize
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: ALP-II-263 characterize
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Jun 13 2011

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

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7316752 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
ALP-II-263 characterize

Sample Name: ALP-II-263 characterize
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: ALP-II-263 characterize
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn
Data collected on: Jun 13 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6574951 MHz
DECOUPLE H1, 499.7341599 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min

Plotname: --Not assigned--

![NCH20]

TBSO

CO2Et

Teoc
ALP-II-265characterize

Sample Name: ALP-II-265characterize
Data Collected on:
  indy.caltech.edu-inova500
Archive directory:
  /home/apuchlop/vnmrsys/data
Sample directory:
  ALP-II-265characterize
FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Jun 14 2011

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

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7316752 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: ALP-II-265characterize
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: ALP-II-265characterize
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn
Data collected on: Jun 14 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6575019 MHz
DECOUPLE H1, 499.7341599 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min
JAC6-203 CDCl3 indy

Sample Name: JAC6-203
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC6-203
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 18 2011

Temp. 25.0 C / 298.1 K
Sample #8, Operator: jcodelli

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7290200 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 36 sec
JAC6-201 CDCl3 indy

Sample Name: JAC6-201
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC6-201
FidFile: CARBON01

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

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6569662 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

Plotname: --Not assigned--
JAC7-237 60 degrees C CD3CN siena

Sample Name: JAC7-237
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-237
FidFile: PROTON04

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Jul 1 2011

Temp. 60.0 C / 333.1 K
Sample #6, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7969204 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec
Sample Name: ALP-III-25
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: alp-III-25
Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn
Data collected on: Jul 29 2011
Temp. 60.0 C / 333.1 K
Operator: jcodelli
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
2500 repetitions
OBSERVE C13, 100.5288812 MHz
DECOUPLE H1, 399.7989164 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 35 min

Plotname: --Not assigned--
Sample Name: JAC7-245hplc
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-245hplc
FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 10 2011

Temp. 50.0 C / 323.1 K
Sample #8, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions

OBSERVE H1, 399.7947957 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec
JAC7-245hplc CDCl3 siena

Sample Name: JAC7-245hplc
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-245hplc
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 10 2011

Temp. 50.0 C / 323.1 K
Sample #4, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
2000 repetitions
OBSERVE C13, 100.5284739 MHz
DECOUPLE H1, 399.7968015 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min
Sample Name: alp-iii-29b
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/apuchlop/vnmrsys/data
Sample directory: alp-iii-29b
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Aug 9 2011

Temp. 60.0 C / 333.1 K
Sample #6, Operator: apuchlop

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7969169 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec
alp-iii-29b CD3CN 60degreesC siena

Sample Name: alp-iii-29b
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory:

Sample directory:

FidFile: ALP-III-29B13C60degreesCsiena

Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn
Data collected on: Aug 9 2011

Temp. 60.0 C / 333.1 K
Operator: apuchlop

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
512 repetitions

OBSERVE C13, 100.5288789 MHz
DECLUPLE H1, 399.7989164 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min
JAC7-251 50degreesC CDCl3 siena

Sample Name: JAC7-251
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-251
FidFile: PROTON03

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

Temp. 50.0 C / 323.1 K
Sample #8, Operator: jcodelli

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7947957 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 0 sec
JAC7-251 50 degreesC CDC13 siena

Sample Name: JAC7-237
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jcdelli/vnmrsys/data
Sample directory: JAC7-237
FidFile: jac7-251-50degrees-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 14 2011

Temp. 50.0 C / 323.1 K
Operator: jcdelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
4000 repetitions
OBSERVE C13, 100.5284739 MHz
DECOUPLE H1, 399.7968015 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
PT size 65536
Total time 2 hr, 32 min

[Chemical structure diagram]

[Graph showing NMR peaks]
Sample Name: JAC7-133
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-133
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2011

Temp. 25.0 C / 298.1 K
Sample #8, Operator: jcodelli

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
Sample Name: JAC7-133
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-133
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2011

Temp. 25.0 C / 298.1 K
Sample #8, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 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 47 min

Plotname: --Not assigned--
JAC7-249 CDC13 indy

Sample Name: JAC7-249
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-249
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 10 2011

Sample #14, Operator: jcodelli

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
Sample Name: JAC7-249
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-249
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 10 2011

Sample #15, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions

OBSERVE  C13, 125.6569662 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 47 min
Sample Name: JAC7-143temp=50
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-143temp=50
FidFile: PROTON01

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

Temp. 50.0 C / 323.1 K
Sample #7, Operator: jcodelli

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7947953 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 36 sec
Sample Name:
JAC7-143

Data Collected on:
siena.caltech.edu-vnmrs400

Archive directory:
/home/jcodelli/vnmrsys/data

Sample directory:
JAC7-143

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3

Data collected on: Jul 17 2011

Temp. 50.0 C / 323.1 K
Sample #1, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
5000 repetitions

OBSERVE C13, 100.5284739 MHz
DECOUPLE H1, 399.7968015 MHz
Power 39 dB
continuously on

WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 hr, 10 min
JAC7-211 CD3CN indy

Sample Name: JAC7-211
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-211
FidFile: PROTON05

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Aug 13 2011

Sample #10, Operator: jcodelli

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7251677 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec
JAC7-211 CD3CN indy

Sample Name: JAC7-211
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-211
FidFile: CARBON02
Pulse Sequence: CARBON (s2pul)
Solvent: cd3cn
Data collected on: Aug 14 2011

Sample 10, Operator: jcodelli

Relax. delay 0.050 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
10000 repetitions
OBSERVE  C13, 125.6558744 MHz
DECOUPLE  H1, 499.7276454 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 hr, 3 min
JAC7-209 CDC13 indy

Sample Name: JAC7-209
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-209
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 15 2011

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

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
JAC7-209 CDCl3 indy

Sample Name: JAC7-209
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-209
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 15 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 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 51 min
JAC7-221 CDC13 indy

Sample Name: JAC7-221
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-221
FidFile: PROTON01

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

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

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7290210 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
JAC7-239 CDC13 indy

Sample Name: JAC7-239
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-239
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 29 2011

Sample #7, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1700 repetitions
OBSERVE C13, 125.6569633 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 58 min
JAC7-243 CDC13 indy

Sample Name: JAC7-243
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-243
FidFile: PROTON02

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

Sample #7, Operator: jcodelli

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.725125 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 0 sec
JAC7-243 CDCl₃ indy

Sample Name:
JAC7-243

Data Collected on:
indy.caltech.edu-inova500

Archive directory:
/home/jcodelli/vnmrsys/data

Sample directory:
JAC7-243

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: CDCl₃
Data collected on: Jun 30 2011

Sample #7, Operator: jcodelli

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
10000 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 5 hr, 41 min
JAC7-301 CDC13 siena

Sample Name: JAC7-301
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-301
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 5 2011

Sample #9, Operator: jcodelli

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
64 repetitions
OBSERVE H1, 399.7947965 MHz
DATA PROCESSING
FT size 32768
Total time 8 min 4 sec

acetylaranotin (1)
JAC7-301 DMSO indy

Sample Name: JAC7-301
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/jcodelli/vnmrsys/data
Sample directory: JAC7-301
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: dmso
Data collected on: Aug 6 2011

Sample #7, Operator: jcodelli

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
40 repetitions
OBSERVE H1, 499.7248788 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 8 min 1 sec
JAC7-301 DMSO-d6 daytona

Sample Name:
JAC7-301
Data Collected on:
daytona.caltech.edu—inova500
Archive directory:
Sample directory:
FidFile: jac7-301carbon
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Aug 6 2011

Temp. 25.0 °C / 298.1 K
Operator: jcodelli
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31421.8 Hz
26500 repetitions
OBSERVE C13, 125.6876974 MHz
DECOUPLE H1, 499.8537553 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 15 hr, 6 min