APPENDIX 1

Spectra Relevant to Chapter 2:

*Enantioselective Synthesis of Pyrroloindolines by a Formal (3 + 2) Cycloaddition*
Sample Name: JN-1-197-col
Data Collected on: hg3.caltech.edu-mercury300
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-197-col
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: Jul 19 2010

Temp. 25.0 C / 298.1 K
Sample #22, Operator: jni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4810.0 Hz
16 repetitions

OBSERVE H1, 300.0862583 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 45 sec

Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

Sample Name: JN-1-197
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-197
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010

Temp. 25.0 C / 298.1 K
Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3200 repetitions
OBSERVE C13, 125.6602406 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 1 hr, 49 min
JN-1-183-col2

Sample Name:
JN-1-183-col2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-183-col2
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 6 2010

Temp. 25.0 C / 298.1 K
Operator: janeni

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420500 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

Sample Name: JN-1-183-col2
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-183-col2
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Jul 6 2010

Temp. 25.0 C / 298.1 K
Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3200 repetitions
OBSERVE C13, 125.6602397 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 49 min
Appendix 1—Spectra Relevant to Chapter 2

Sample Name: JN-1-181-col
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-181-col
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 27 2010
Temp. 25.0 C / 298.1 K
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 24 sec
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

JN-1-201-col

Sample Name: JN-1-201-col
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-201-col
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 24 2010

Temp. 26.0 C / 299.1 K
Sample #12, Operator: jni

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

OBSERVE H1, 399.7942813 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 57 sec
Sample Name: JN-1-201-col
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-201-col
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 24 2010

Temp. 26.0 C / 299.1 K
Sample #12, Operator: jni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
4000 repetitions
OBSERVE C13, 100.5283515 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 hr, 32 min
Appendix 1—Spectra Relevant to Chapter 2
Sample Name: JN-1-203
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-203
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010

Temp. 25.0 C / 298.1 K
Sample #3, Operator: jni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
5000 repetitions
OBSERVE C13, 100.5283529 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line brodkening 0.5 Hz
FT size 65536
Total time 3 hr, 10 min
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

LMRIV-N-me-TBStryptophol2

Sample Name:
LMRIV-N-me-TBStryptophol2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-N-me-TBStryptophol2
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 10 2010

Temp. 25.0 C / 298.1 K
Operator: lrepka

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3500 repetitions

OBSERVE C13, 125.6602422 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 1 hr, 59 min
Sample Name: LMRIV-291-majordr-hsqc
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-291-majordr-hsqc
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 1 2010

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7942797 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec

rotameric ratio 1.0:1
Appendix 1—Spectra Relevant to Chapter 2
**Appendix 1—Spectra Relevant to Chapter 2**

![Spectrum Image]

**Sample Name:** LMRIV-291-majordr-conc

**Data Collected on:** siena.caltech.edu-vnmrs400

**Archive directory:** /home/lrepka/vnmrsys/data

**Sample directory:** LMRIV-291-majordr-conc

**FidFile:** CARBON01

**Pulse Sequence:** CARBON (s2pul)

**Solvent:** cdcl3

Data collected on: Jul 31 2010

**Temp.** 25.0 °C / 298.1 K

**Sample #11, Operator:** lrepka

**Relax. delay** 1.000 sec

**Pulse** 45.0 degrees

**Acq. time** 1.468 sec

**Width** 22321.4 Hz

**3000 repetitions**

**OBSEERVE** C13, 100.5283980 MHz

**DECOUPLE** H1, 399.7962875 MHz

**Power** 39 dB

continuously on

**WALTZ-16 modulated**

**DATA PROCESSING**

Line broadening 1.0 Hz

**FT size** 65536

**Total time** 2 hr, 3 min

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**Diagram:** Molecular structure with rotameric ratio 1.0:1.
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

JN-1-131-majordiast

Sample Name: JN-1-131-majordiast
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-131-majordiast
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 5 2010

Temp. 25.0 C / 298.1 K
Sample #1, Operator: jni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7943120 MHz
DATA PROCESSING
FT size 32768
Total time 7 min 21 sec

data plot

Me
N
N
H
OMe
O
TFA
Me
exo-94
rotameric ratio
2.4:1
Appendix 1—Spectra Relevant to Chapter 2

JN-1-131-majordiast

Sample Name: JN-1-131-majordiast
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-131-majordiast
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 5 2010

Temp. 25.0 C / 298.1 K
Sample #1, Operator: jni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
9700 repetitions
OBSERVE C13, 100.5283531 MHz
DECouple H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 6 hr, 9 min
JN-1-131-minordiast3

Sample Name: JN-1-131-minordiast3
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-131-minordiast3
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 5 2010

Operator: janeni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE N1 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec

Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2
JN-1-151-major

Sample Name: JN-1-151-major
Data Collected on: hg3.caltech.edu-mercury300
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-151-major
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 24 2010

Temp. 25.0 C / 298.1 K
Sample #20, Operator: jni
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4810.0 Hz
16 repetitions
OBSERVE H1, 300.0862588 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 1 sec

Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

Sample Name: JN-1-151

Data Collected on: siena.caltech.edu-vnmrs400

Archive directory: /home/jni/vnmrsys/data

Sample directory: JN-1-151

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 26 2010

Sample #1, Operator: jni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
8500 repetitions

OBSERVE C13, 100.5283573 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 5 hr, 23 min

ex-95 rotameric ratio 1:1

rotameric ratio
1:1

200 180 160 140 120 100 80 60 40 20
ppm

ex-95 rotameric ratio 1:1
Sample Name: JN-1-151-minordiast3
Data Collected on: Aug 4 2010
Operator: janeni

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions

OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec

Operator: janeni
Appendix 1—Spectra Relevant to Chapter 2

JN-1-151-minordiast3

Sample Name:
JN-1-151-minordiast3
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-151-minordiast3
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 4 2010
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602444 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 24 min

endo-95
rotamer ratio
3.0:1

Me
N
N
H
OBn
Me
endo-95
rotamer ratio
3.0:1
Appendix 1—Spectra Relevant to Chapter 2
Sample Name: LMRIV-scale-up-12h-majordr
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-scale-up-12h-majordr
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 29 2010

Temp. 25.0 C / 298.1 K
Sample #1, Operator: lrepka

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
11500 repetitions
OBSERVE C13, 100.5283829 MHz
DECouple H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 17 min

Appendix 1—Spectra Relevant to Chapter 2
Sample Name: LMRIV-287-minor-8-5-10
Data Collected on: hg3.caltech.edu-mercury300
Archive directory:/home/lrepka/vnmrsys/data
Sample directory: LMRIV-287-minor-8-5-10
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 5 2010

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

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4810.0 Hz
8 repetitions
OBSERVE H1, 300.0862322 MHz
DATA PROCESSING
FT size 32768
Total time 3 min 35 sec

endo-96
rotameric ratio
10:1
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

Sample Name: JN-1-191-majordiast
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-191-majordiast
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010

Temp. 25.0 C / 298.1 K
Sample #5, Operator: jni

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7942813 MHz
DATA PROCESSING
FT size 32768
Total time 7 min 21 sec

Rotamic ratio 1.7:1
Appendix 1—Spectra Relevant to Chapter 2

JN-1-191-majordiast

Sample Name: JN-1-191-majordiast
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-191-majordiast
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010

Temp. 25.0 C / 298.1 K
Sample #5, Operator: jni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
11500 repetitions
OBSERVE C13, 100.5283544 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 17 min

Me
Me

N

N

OBn

TFA

exo-98a
rotameric ratio
1.7:1
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

JN-1-191-minordiast

Sample Name: JN-1-191-minordiast
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-191-minordiast
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 18 2010

Temp. 25.0 C / 298.1 K
Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
12000 repetitions
OBSERVE C13, 125.6602389 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 6 hr, 50 min

endo-98a 
rotameric ratio 68:1

MeO
Me
N
Me
H
TFA
OBn
Me
endo-98a
rotameric ratio
68:1
Sample Name: JN-1-195-major diast4
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-195-major diast4
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3

Data collected on: Jul 16 2010
Temp. 25.0 C / 298.1 K
Operator: janeni

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec

Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

Data collected on: Jul 16 2010
Temp. 25.0 C / 298.1 K
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees Acq. time 1.042 sec Width 31446.5 Hz 13000 repetitions
OBSERVE  C13, 125.6602389 MHz
DECOUPLE  H1, ... Power 39 dB continuously on WALTZ-16 modulated
DATA PROCESSING Line broadening 1.0 Hz
Total time 7 hr, 24 min

Appendix 1—Spectra Relevant to Chapter 2

20 40 60 80 100 120 140 160 180 ppm
Appendix 1—Spectra Relevant to Chapter 2
JN-1-195-minordiast

Sample Name: JN-1-195-minordiast
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-195-minordiast
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 15 2010

Temp. 25.0 C / 298.1 K
Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602387 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 7 hr, 24 min
Sample Name: JN-1-171-majdiast
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-171-majdiast
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 14 2010
Temp. 25.0 C / 298.1 K
Sample #4, Operator: jni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7942813 MHz
DATA PROCESSING
FT size 32768
Total time 7 min 21 sec

Appendix 1—Spectra Relevant to Chapter 2

![Spectrum Image]

rotameric ratio
2.0:1

ppm
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

Sample Name: JN-1-171-mindiast2
Data Collected on: hg3.caltech.edu-mercury300
Archive directory: /home/jni/vnmrsys/data
Sample directory: JN-1-171-mindiast2
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 13 2010

Temp. 25.0 C / 298.1 K
Sample #17, Operator: jni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4810.0 Hz
16 repetitions
OBSERVE H1, 300.0862580 MHz
DATA PROCESSING
FT size 32768
Total time 7 min 9 sec

Me
N
N
H
OBn
O
TFA
endo-98c
rotameric ratio 6.7:1

ppm
Appendix 1—Spectra Relevant to Chapter 2

JN-1-171-minordiast2

Sample Name: JN-1-171-minordiast2
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-171-minordiast2
Fid File: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 14, 2010

Temp. 25.0 C / 298.1 K
Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31445.5 Hz
13000 repetitions
OBSERVE C13, 125.6602382 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.7 Hz
FT size 65536
Total time 7 hr, 24 min
Appendix 1—Spectra Relevant to Chapter 2
Sample Name: LMRIV-177-majordr-7-31-10
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-177-majordr-7-31-10
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 31 2010

Operator: lrepka
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.022 sec
Width 32051.3 Hz
13250 repetitions
OBSERVE C13, 125.6602418 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 28 min
Appendix 1—Spectra Relevant to Chapter 2

LMRIV-177-minordr-hsqc

Sample Name: LMRIV-177-minordr-hsqc
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-177-minordr-hsqc
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 1 2010

Operator: lrepka
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420853 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 36 sec
Appendix 1—Spectra Relevant to Chapter 2

[Image of a spectrum with chemical structures and labels]
JN-1-215-major diast2

Sample Name: JN-1-215-major diast2
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-215-major diast2
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 3 2010

Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602480 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 7 hr, 24 min

Appendix 1—Spectra Relevant to Chapter 2
Sample Name: JN-1-215-minordiast2
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-215-minordiast2
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 2 2010
Operator: janeni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec

Appendix 1—Spectra Relevant to Chapter 2

endo-98e
rotameric ratio
9.0:1

ppm
JN-1-215-minordiast2

Sample Name: JN-1-215-minordiast2
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-1-215-minordiast2
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 2 2010

Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
12500 repetitions

OBSERVE C13, 125.6602413 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 7 hr, 7 min

endo 98e
rotameric ratio 90:1
Appendix 1—Spectra Relevant to Chapter 2

Sample Name: LMRIV-285-major-rd25
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-285-major-rd25
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2010

Operator: lrepka
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions

OBSERVE H1, 499.7420502 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min 40 sec

rotameric ratio 1.7:1
LMRIV-285-majordr-2-again

Sample Name: LMRIV-285-majordr-2-again
Data Collected on: siena.caltech.edu-vnmrs400
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-285-majordr-2-again
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2010

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.258 sec
Width 26041.7 Hz
10500 repetitions
OBSERVE C13, 100.5283569 MHz
DECOUPLE H1, 299.7962875 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 6 hr, 35 min

Appendix 1—Spectra Relevant to Chapter 2
Sample Name: LMRIV-285-minor-rd25
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-285-minor-rd25
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2010

Operator: lrepka

Relax. delay 25.000 sec
Pulse 45.0 degrees
Aqc. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420512 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min 40 sec
**LMRIV-285-minordr-2-carbon**

**Sample Name:** LMRIV-285-minordr-2-carbon  
**Data Collected on:** indy.caltech.edu-inova500  
**Archive directory:** /home/lrepka/vnmrsys/data  
**Sample directory:** LMRIV-285-minordr-2-carbon  
**FidFile:** CARBON01

**Pulse Sequence:** CARBON (s2pul)  
**Solvent:** cdc13  
**Data collected on:** Jul 30 2010

**Operator:** lrepka

**Relax. delay 1.000 sec**  
**Pulse 45.0 degrees**  
**Acq. time 1.022 sec**  
**Width 32051.3 Hz**  
**13000 repetitions**  
**OBSERVE C13, 125.6602398 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 7 hr, 20 min**

**endo-98% rotameric ratio 16.7:1**
Appendix 1—Spectra Relevant to Chapter 2

LMRIV-275-pentanewash

Sample Name: LMRIV-275-pentanewash
Data Collected on: hg3.caltech.edu-mercury300
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-275-pentanewash
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 14 2010

Temp. 25.0 C / 298.1 K
Sample #33, Operator: lrepka
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4810.0 Hz
8 repetitions
OBSERVE H1, 300.0862580 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 31 sec

rotameric ratio
>20:1

171
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 15 2010
Temp. 25.0 C / 298.1 K
Sample #8, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
11500 repetitions
OBSERVE C13, 100.5283535 MHz
DECOUPLE H1, 399.7992875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 17 min
LMRIV-267-major diastereomer

Sample Name: LMRIV-267-major
data
Data Collected on:
indy.caltech.edu-inova500
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-267-major
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010

Temp. 25.0 C / 298.1 K
Operator: lrepka

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions

OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec
Appendix 1—Spectra Relevant to Chapter 2
Sample Name: LMRIV-267-minor3
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-267-minor3_20100717_01
FidFile: data_s2pul_001

Pulse Sequence: PROTON (s2pul)
Solvent: CDCl3
Data collected on: Jul 17 2010

Temp. 25.0 C / 298.1 K
Operator: lrepka

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 7996.0 Hz
8 repetitions
OBSERVE H1, 499.7420487 MHz
DATA PROCESSING
FT size 32768
Total time 3 min 36 sec
Sample Name: LMRIV-267-minor3
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRIV-267-minor3_20100717_01
FidFile: data_s2pul_002

Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Jul 17 2010

Temp. 25.0 C / 298.1 K
Operator: lrepka

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
12000 repetitions
OBSERVE C13, 125.6602386 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 6 hr, 50 min
Appendix 1—Spectra Relevant to Chapter 2

177
JN-1-211-majordiast

Sample Name:
JN-1-211-majordiast

Data Collected on:
siena.caltech.edu-vnmrs400

Archive directory:
/home/jni/vnmrsys/data

Sample directory:
JN-1-211-majordiast

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jul 28 2010

Temp. 25.0 C / 298.1 K

Sample #6, Operator: jni

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.285 sec

Width 25510.2 Hz

11000 repetitions

OBSERVE C13, 100.5283568 MHz

DECOUPLE B1, 399.7962875 MHz

Power 39 dB continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 6 hr, 58 min
Appendix 1—Spectra Relevant to Chapter 2
JN-1-211-minordiast

Sample Name:  
JN-1-211-minordiast  
Data Collected on:  
indy.caltech.edu-inova500  
Archive directory:  
/home/janeni/vnmrsys/data  
Sample directory:  
JN-1-211-minordiast  
FidFile: CARBON01  

Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Jul 28 2010  

Operator: janeni  

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.042 sec  
Width 31446.5 Hz  
13000 repetitions  
OBSERVE C13, 125.6602401 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 7 hr, 24 min

endo-72  
rotameric ratio  
15.7:1  

Appendix 1—Spectra Relevant to Chapter 2

ppm

200 180 160 140 120 100 80 60 40 20 0

20 40 60 80 100 120 140 160 180
Sample Name: LMRV-047-majordr
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRV-047-majordr
File Name: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: dcdcl3
Data collected on: Sep 16 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: lrepka

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions

OBSERVE H1, 499.7420517 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 65536
Total time 3 min 40 sec
LMRV-047-majordr

Sample Name: LMRV-047-majordr
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/lrepka/vnmrsys/data
Sample directory: LMRV-047-majordr
Fidfile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Sep 16 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: lrepka

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
11500 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 65536
Total time 6 hr, 33 min

Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

JN-3-101-carbon

Sample Name:
JN-3-101-carbon
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-3-101-carbon
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 25 2012
Sample #17, Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7225127 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 56 sec
Appendix 1—Spectra Relevant to Chapter 2

JN-3-101-carbon

Sample Name: JN-3-101-carbon
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/janeni/vnmrsys/data
Sample directory: JN-3-101-carbon
Fidfile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Jan 25 2012

Sample #17, Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
10000 repetitions
OBSERVE C13, 125.6553277 MHz
DECOUPLE H1, 499.7250019 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 5 hr, 41 min
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

### WHK-3-101-Rep-F2_INDY_CDCL3

**Sample Name:** WHK-3-101-Rep-F2_INDY_CDCL3
**Sequence:** CARBON
**Temperature:** 25
**Spectrometer:** Varian 400
**Operator:** janwar

#### Acquisition
- **Date:** Jan 9 2012
- **Solvent:** ed23
- **File:** /data/Harvestor/wh/whkyar/data/WHK-3-101-1
- **File:** /data/Harvestor/wh/whkyar/data/WHK-3-101-1
- **Time:** 1.84 s

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#### Transmitter
- **F1:** C13
- **F2:** 125.669
- **F3:** 1313.4
- **F4:** 56
- **F5:** 4089

#### Decoupler
- **DA:** H1
- **DCD:** 0
- **DDC:** yyy
- **DDC2:** w
- **DDC3:** 23
- **DDC4:** 11900

#### Presaturation
- **n**

#### Special
- **Temp:** not used
- **Gain:** 10
- **Gain:** 10
- **HSL:** 0.000
- **IELD:** 0.100
- **DATA:** 10.000

#### Flags
- **IL:** n
- **ILP:** n
- **ILP:** n

#### Processing
- **LB:** 0.10
- **LB:** 0
- **LB:** 0
- **LB:** not used

#### Display
- **HP:** -0.1
- **WP:** 2756.4
- **WP:** 0
- **WP:** 0
- **WP:** 157.7

#### Plot
- **WS:** 294
- **WS:** 2
- **WS:** 0
- **WS:** 13000
- **WS:** 20
- **WS:** 0

---

[Image of NMR spectrum with chemical structures and peaks labeled]
Appendix 1—Spectra Relevant to Chapter 2

Sample Name: JN-3-063b-colmajor
Date collected: 2011-11-01
Solvent: cd3c6d6
Spectrometer: nmr600
Temperature: 25
Sample owner: jol
Operator: autoiser

JN-3-063b-colmajor

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Diagram of chemical structure:

- Benzene ring
- Methoxy group (OMe)
- TFA group
- Carbon atoms labeled
- Protons and other nuclei marked

Integration and peak assignment:

- Integration values from 224 to 14441
- ppm values from 220 to 20 ppm

Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

### WHX:3:60–F2-Rep-F2:INDY_CDCL3

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[Graph of spectrum with ppm values from 0.6 to 7.3 showing peaks at various frequencies.]

---

193
Appendix 1—Spectra Relevant to Chapter 2
Appendix 1—Spectra Relevant to Chapter 2

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**Diagram:**

- Substance: N,N,N-Ph\(_2\)-Me-TFA-OMe
- Molar Mass: 110g

**Chemical Structure:**

![Chemical Structure Diagram](image-url)

**Notes:**

- PRESATURATION: n, n