Asymmetric Synthesis of 2*H*-Azirine 3-Carboxylates

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¹H and ¹³C NMR spectra for all new compounds

7.536
7.520
7.497
7.492
7.485
7.477
7.411
7.406
7.398
7.316
7.300
7.264

5.285
5.271
5.096
5.081

3.838

2.421

52

F1: 500.130 F2: 500.130 SW1: 15038 PW: 8.0 usec PD: 1.0 sec OF1: 2500.6 NA: 16 LB: 0.0
 EX: 1puls
 USER: -- DATE: Thu Feb 22 10:00:41
 PTSId: 16384
 WinNuts - \$exNaudicloropur030303

Msulph-dicloroc13
c13.gk

Current Data Parameters
NAME phthalic
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030304
Time 18.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 0
SMH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 2298.8
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 12.00 usec
PL1 2.00 dB
SFO1 100.5961334 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 25.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters

SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 234.884 ppm
F1 23625.79 Hz
F2P -14.99.84 Hz
F2 -1499.84 Hz
PRCM 12.48974 ppm/cm
HZCM 1256.28137 Hz/cm

dicloroiminesulfinyl
c13.gk

130.099
128.877
128.817
126.302
125.484

77.766
77.449
77.131

65.701

55.321

36.095
31.734
31.152
26.012
21.805

Current Data Parameters
NAME halo
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020103
Time 9.55

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg60
TD 65536
SOLVENT CDCl3
NS 600
DS 0

SMH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec

RG 143.7
DM 19.900 usec
DE 6.00 usec
TE 300.0 K

D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 2.00 dB
SFO1 100.5961334 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 25.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 177.065 ppm
F1 17810.14 Hz
F2P 13.873 ppm
F2 1395.40 Hz
PPMCM 8.15963 ppm/cm
HZCM 820.73682 Hz/cm

7.620
7.603
7.304
7.287
7.264
7.262

4.403
4.382
4.128
4.124
4.106
4.102
3.929

2.412
2.396

1.149
1.136
0.985
0.972

59

EX: 1puls	F1: 500.130	F2: 500.130	SW1: 6006	PW: 8.0 usec	PD: 0.0 sec	OF1: 2500.6	NA: 16	LB: 0.0	PTSId: 16384
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USER: --- DATE: Sun Feb 4 03:24:47
 WinNuts - \$exN\$uldicloropud021303

Nsuldicloroiopropurec13
c13.gk

Current Data Parameters
NAME iprodicloro
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030213
Time 13.52

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg60
TD 65536
SOLVENT CDCl3
NS 600
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 3649.1
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 2.00 dB
SFO1 100.5961334 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 25.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

ID NMR plot parameters

CX 20.00 cm
F1P 180.438 ppm
F1 18149.37 Hz
F2P 4.750 ppm
F2 477.76 Hz
PPMCK 8.78441 ppm/cm
HZCM 883.58038 Hz/cm

chlorosulazdn020702pure
c13.gk

Current Data Parameters
NAME dthalo
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020208
Time 14.06

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 10321.3
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 2.00 dB
SFO1 100.5961334 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 25.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

ID NMR plot parameters
CX 20.00 cm
F1P 176.315 ppm
F1 17734.61 Hz
F2P 12.872 ppm
F2 1294.70 Hz
PPMCM 8.17214 ppm/cm
HZCM 821.99561 Hz/cm

7.680
7.666
7.663
7.357
7.344
7.264
7.261

3.804
3.801

2.889
2.871

2.437

1.021
1.008
0.865
0.852

105

F1: 500.130	F2: 500.130	SW1: 15038	PD: 0.0 sec	OF1: 2500.6	LB: 0.0	PTSId: 16384	USER: -- DATE: Thu Feb 1 16:02:28
EX: 1puls		PW: 8.0 usec		NA: 16			WinNuts - Sexycyclopnt021003

8 7 6 5 4 3 2 1 PPM

cyclopue021903C13
c13.gk

ppm 200 175 150 125 100 75 50 25 0

Current Data Parameters
NAME iprodicloro
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030219
Time 18.59

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg60

TD 65536
SOLVENT CDCl3

NS 600
DS 0

SMH 25125.629 Hz
FIDRES 0.383387 Hz

AQ 1.3042164 sec
RG 2580.3

DM 19.900 usec
DE 6.00 usec

TE 300.0 K
D1 2.00000000 sec

d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 2.00 dB

SFO1 100.5961334 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB

PLI2 20.00 dB
PLI3 25.00 dB

SFO2 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz

WDW EM
SSB 0
LB 4.00 Hz

GB 0
PC 1.40

ID NMR plot parameters
CX 20.00 cm
CY 12.50 cm

FLP 215.000 ppm
F1 21625.79 Hz

F2p -5.000 ppm
F2 -502.92 Hz

PRPCH 11.00000 ppm/cm
HZCM 1106.43579 Hz/cm

Tsazdmcr021802
c13.gk

Current Data Parameters
NAME dthalo
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020218
Time 16.25

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg60

TD 65536
SOLVENT CDCl3
NS 600

DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz

AQ 1.3042164 sec
RG 6502

DW 19.900 usec
DE 6.00 usec

TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 2.00 dB
SFO1 100.5961334 MHz

===== CHANNEL f2 =====
CEPPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 25.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

ID NMR plot parameters
CX 20.00 cm
F1P 175.313 ppm
F1 17633.91 Hz
F2P 12.872 ppm
F2 1294.70 Hz
PRKCM 8.12208 ppm/cm
HZCM 816.96045 Hz/cm

03/12/2002

mengBrpure031202
c13k.gk

03/12/2002

from Halo-Azidine

14a

Current Data Parameters
NAME dihalo
EXPNO 213
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020312
Time 23.59

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg60
TD 65536
SOLVENT CDCl3
NS 4800

DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 7298.2

DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 2.00 dB
SF01 100.5961334 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 25.00 dB
SF02 400.0216001 MHz

F2 - Processing Parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 185.556 ppm
F1 18664.13 Hz
F2P 634.83 ppm
F2 634.83 Hz
PWCN 8.96221 ppm/cm
HZCN 901.46484 Hz/cm

PhMgBr030103C13
c13.gk

Current Data Parameters
NAME pentadoloro
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030301
Time 19.21

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg60
TD 65536
SOLVENT CDCl3
NS 1000
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 2580.3
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 2.00 dB
SFO1 100.5961334 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 25.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 215.000 ppm
F1 21625.79 Hz
F2P -5.000 ppm
F2 -502.92 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.43579 Hz/cm

7.462
7.446
7.349
7.335
7.319
7.308
7.293
7.278
7.262
7.199
7.184

3.695

2.667
2.652
2.636

2.326

1.739
1.723
1.708
1.692
1.678
1.663

5.64

2.77

2.08

3.00

2.12

1.10

9.01

175

0.00

7

6

5

4

3

2

PPM

F1: 500.130	F2: 500.130	SW1: 15038	PD: 1.0 sec	OF1: 2500.6	LB: 0.0	PTSId: 16384
EX: 1puls		PW: 8.0 usec		NA: 16		WinNuts - SexPhMgBrpud030103

USER: -- DATE: Tue Feb 20 07:03:34

EX: 1puls

F1: 500.130	F2: 500.130	SW1: 15038	PW: 8.0 usec	PD: 1.0 sec	NA: 16	LB: 0.0	PTSId: 16384
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WinNuts - SexVinyMgBipue030103

USER: -- DATE: Tue Feb 20 07:09:24

VinyMgBr030103C13
c13.gk

Current Data Parameters
NAME pentadichloro
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030302
Time 3.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 299960
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 2580.3
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 2.00 dB
SFO1 100.596134 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 25.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 197.578 ppm
F1 19873.43 Hz
F2P 3.237 ppm
F2 325.64 Hz
PFCM 9.71704 ppm/cm
HZCM 977.38953 Hz/cm

7.472
7.469
7.455
7.347
7.334
7.330
7.317
7.302
7.288
7.263

3.732

2.074
2.054

1.257
1.132
1.118
1.105
1.091

EX: 1puls F1: 500.130 F2: 500.130 SW1: 8000 PW: 8.0 usec PD: 1.0 sec OF1: 2500.6 NA: 16 LB: 0.0 PTSId: 16384 WmNuis - SexPmMgBraddtopun030703

USER: -- DATE: Wed Feb 28 03:39:19

205

PhMgBraddto030703uc13
c13.gk

Current Data Parameters
NAME itrodicloro
EXPNO 50
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030309
Time 15.39

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg60

TD 65536
SOLVENT CDCl3

NS 600
DS 0

SMH 25125.629 Hz
FIDRES 0.383387 Hz

AQ 1.3042164 sec
RG 2580.3

DM 19.900 usec
DE 6.00 usec

TE 300.0 K
D1 2.00000000 sec

d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 2.00 dB
SFO1 100.5961334 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 25.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters
SI 37768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FLP 215.000 ppm
F1 21625.79 Hz
F2 -5.000 ppm
P2P -502.92 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.43579 Hz/cm

F1: 500.130	F2: 500.130	SW1: 6006	PD: 1.0 sec	OF1: 2500.6	LB: 0.0	PTSID: 16384
EX: 1puls		PW: 8.0 usec		NA: 16		WinNuts - \$exHClazirine-uppur042504

USER: -- DATE: Tue Apr 25 08:02:32

7.282
7.270
7.267
7.265
7.256
7.252
7.183
7.180
7.169
7.152

17

3.715
3.674
3.633

3.215
3.181

2.673
2.637
2.608
2.594
2.578
2.459
2.457
1.995
1.983

1.630
1.615
1.599
1.585
1.508
1.491
1.488
1.481
1.475
1.464
1.454
1.254

245

8	7	6	5	4	3	2	1PPM
deng/AzilCdimeDielsC13042604_25Apr2004							
F1: 500.130	F2: 500.130	SW1: 6006	PD: 1.0 sec	OF1: 2500.6	NA: 16	LB: 0.0	PTSId: 16384
EX: 1puls		PW: 8.0 usec					WinNuts - \$exL CdimeDielsC13042604

170.082

139.971

125.850
125.729
123.117
117.805
117.252

17

74.954
74.531
74.108

50.404
49.528

41.433
40.157

33.276
28.651
26.578
25.670
24.607

16.198
13.852

255

150

100

50

PPM

deng/AzilCdimeDielsCI3042604_25Apr2004

F1: 75.440

F2: 299.987

SW1: 18859

PW: 3.9 usec

PD: 5.0 sec

OF1: 8297.5

NA: 320

LB: 0.0

PTSI: 65536

WinNuts - \$CARBON.fid

USER: -- DATE: Apr 25 2004

265

F1: 500.130	F2: 500.130	SW1: 6006	PD: 1.0 sec	OF1: 2500.6	NA: 16	LB: 0.0	PTSId: 16384
EX: 1puls		PW: 8.0 usec					WinNuts - \$experi-LC-diels042704

USER: -- DATE: Thu Apr 27 10:21:39

275

deng/peri-LC-diels042704C13_27Apr2004
 F1: 75.439 F2: 299.986 SW1: 18859 PW: 3.9 usec PD: 5.0 sec OF1: 8297.5 NA: 1024 LB: 0.0
 EX: szpul PTSId: 65536
 USER: -- DATE: Apr 27 2004
 WinNuts - \$CARBON.fid

F1: 500.130 F2: 500.130 SW1: 6006 PW: 8.0 usec PD: 1.0 sec OF1: 2500.6 NA: 16 LB: 0.0
 EX: 1 puls
 USER: -- DATE: Tue May 2 01:20:12
 PTSId: 16384
 WinNuts - SexLC-cypent-dicls050204

172.813

142.520

132.338
128.500
128.416
128.289
125.682

77.525
77.101
76.678

66.450

58.971

53.821
52.377
48.457
47.448

35.826

31.147
28.438
26.837

295

180 160 140 120 100 80 60 40 20 PPM

deng/LC-cyclpen-dielso50204C13_02May2004
F1: 75.439 F2: 299.986 SW1: 18859 PW: 3.9 usec PD: 5.0 sec OF1: 8297.5 NA: 1024 LB: 0.0
EX: s2pul
USER: -- DATE: May 2 2004
WinNuts - \$CARBON.fid