Asymmetric Synthesis of Cyclic α -Amino Phosphonates using Masked Oxo

Sulfinimines (*N*-Sulfinyl Imines)

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Supporting information

General Procedures: Column chromatography was performed on silica gel, Merck grade 60 (230-400 mesh). TLC plates were visualized with UV, in an iodine chamber, or with phosphomolybdic acid, unless otherwise noted. ^1H NMR and ^{13}C NMR spectra were recorded at 500 and 125 MHz, respectively. Solvent were purified, dried, using the Glass Contour solvent dispensing system. Unless stated otherwise, all the reagents were purchased from commercial sources and used without additional purification.

Spectral data, ^1H and ^{13}C NMR, for all new compounds

9a - ¹H NMR

51

lco/48_13Dec2003

F1: 299.986

F2: 75.439

SW1: 3600

PW: 4.4 usec

PD: 1.0 sec

QF1: 1499.9

NA: 32

LB: 0.0

PTSId: 16384

USER: -- DATE: Dec 13 2003

WinNuts - SPROTON.fid

9a-¹H NMR

S2

9b - ¹H NMR

S3

96-¹³C NMR

S4

cl3.gk

Current Data Parameters
NAME tomlee
EXPNO 1840
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020502
Time 14.04
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 6502
DE 19.900 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
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 EN
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CK 20.00 cm
FIP 207.913 ppm
F1 20912.93 Hz
F2 -9.618 ppm
FPCW 10.87653 ppm/cm
HZCN 1094.01697 Hz/Hz

9C-1HNR

54

Current Data Parameters	
NAME	tonylee
EXPNO	1940
PROCNO	1

F2 - Acquisition Parameters

Date_	Time	INSTRUM	PROBHD	PULPROG	TD	SOLVENT	NS	DS	SMW	FIDRES	AQ	RG	DE	TE	DI
20200517	13.50	5 mm QNP 1H/13	zg30	65536		CDCl3	8	0	8278.146 Hz	0.126314 Hz	3.9584243 sec	71.8	60.400 usec	300.0 K	1.00000000 sec
													6.00 usec		

```
===== CHANNEL f1 =====
NUC1      1H
P1        7.50 usec
PL1       0.00 dB
SF01      400.0220001 MHz
```

F2 - Processing parameters	
SI	32768
SF	400.0200313 MHz
WDW	EM
SSS	0
LB	0.30 Hz
GB	0
PC	1.00
1D NMR plot parameters	
CX	20.00 cm
F1P	9.586 ppm
F1	3834.46 Hz
F2P	-0.422 ppm
F2	-168.70 Hz
PPMCH	0.50037 ppm/cm
HZCN	200.15914 Hz/cm

1D NMR plot parameters

1D NMR plot parameters	
CX	20.00 cm
F1P	9.586 ppm
F1	3834.46 Hz
F2P	-0.422 ppm
F2	-168.70 Hz
PPMCM	0.50037 ppm/cm
HZCM	200.15814 Hz/cm

cl3.gk

gc-¹³C NMR

S6

Current Data Parameters
 NAME tonylee
 EXPNO 1941
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020517
 Time 14.25
 INSTRUM spect
 PROBRD 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 5160.6
 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
 P2 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
 FIP 209.531 ppm
 F1 21075.73 Hz
 F2P -4.223 ppm
 F2 -424.81 Hz
 PPMCH 10.68774 ppm/cm
 HZCM 1075.02686 Hz/cm

h1.gk

100 - 400 MHz
S7

Current Data Parameters
NAME tomylee
EXPNO 1800
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020430
Time 13.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.0220001 MHz

F2 - Processing parameters
SI 32768
SF 400.0200374 MHz
WDW EN
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.200 ppm
F1 4080.24 Hz
F2P -0.283 ppm
F2 -113.13 Hz
PPMCM 0.52415 ppm/cm
HZCM 209.66850 Hz/cm

```

Current Data Parameters
NAME          tonylee
EXPNO         1801
PROCNO        1

F2 - Acquisition Parameters
Date_         20020430
Time          14.01
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            600
DS            0
SMA           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG            6502
WDW            19,900 usec
DE            6.00 usec
TE            300.0 K
D1            2.0000000 sec
d11           0.0300000 sec
d12           0.0002000 sec

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

===== CHANNEL f2 =====
waltz16
NUC2          1H
P2            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            100.5850377 MHz
WDW           EM
SSB           0
GB            4.00 Hz
PC            1.40

=====
OLD NMR plot parameters
CX            20.00 cm
CY            12.50 cm
F1P           215.000 ppm
F1F1          21625.79 Hz
F2P           -5.000 ppm
F2F2          -502.92 Hz
PPMCH         11.00000 ppm/cm
HZCH          1106.43542 Hz/cm

```

hl.gk

10d - ¹H NMR

S9

Current Data Parameters
 NAME tonylee
 EXPNO 1930
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020517
 Time 13.04
 INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 80.6
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 0.00 dB
 SFO1 400.0220001 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0200323 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.542 ppm
 F1 3817.02 Hz
 F2P 0.029 ppm
 F2 11.54 Hz
 PPMCM 0.47566 ppm/cm
 HZCM 190.27379 Hz/cm

cl3.gk

10d-8vnmk

S10

Current Data Parameters
 NAME tonylee
 EXPNO 1931
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020517
 Time 13.39
 INSTRUM spect
 PROBED 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 4096
 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
 FIP 234.884 ppm
 F1 23625.79 Hz
 F2P -14.911 ppm
 F2 -1499.84 Hz
 FPRCH 12.48974 ppm/cm
 HZCH 1256.28137 Hz/cm

12a - 1H NMR

S11

F2 - Acquisition Parameters
 Date_ 20010730
 Time 15.02
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 114
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 DI 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 0.00 dB
 SFO1 400.0224703 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0200138 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FIP 10.326 ppm
 F1 4130.62 Hz
 F2P -0.526 ppm
 F2 -210.35 Hz
 FWHM 0.54260 ppm/cm
 HZ/CN 217.04893 Hz/cm

12a - 13C NMR

F2 - Acquisition Parameters
 Date_ 20031077
 Time 15.04
 INSTRUM spect
 PROBD 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 1024
 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
 P2P2 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
 MDW 0
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.40
 1D NMR plot parameters
 CX 20.00 cm
 F1P 201.375 ppm
 F1 20255.28 Hz
 P2P -1.457 ppm
 F2 -146.52 Hz
 PPMCH 10.14157 ppm/cm
 HZCH 1020.09033 Hz/cm

c13.gk

S12

12a - 1 VAR

S13

ppm

F2 - Acquisition Parameters
 Date_ 20010720
 Time 17.26
 INSTRUM spect
 PROBED 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 75187.969 Hz
 FIDRES 1.147277 Hz
 AQ 0.4338644 sec
 RG 20642.5
 DW 6.650 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 31P
 P1 5.00 usec
 PL1 8.00 dB
 SFO1 161.9227676 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 161.9310466 MHz
 NCM EX
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 27.168 ppm
 F1 4399.38 Hz
 F2P 21.559 ppm
 F2 3490.99 Hz
 PPMCH 0.28049 ppm/cm
 HzCH 45.41938 Hz/cm

12b - 1H NMR

S14

10	8	6	4	2	0	PPM
spec: CDCl ₃						
F1: 400.022	F2: 1.000	SW: 1.8278	OF: 1962.5	DATE: Mon Jun 17 19:06:48 2002		
EX:		PD: 1.0 sec	NA: 8	PTS: 32768		
			LB: 0.0			
						WinNuts - Stony

12b - 12b

c13.gk

S15

F2 - Acquisition Parameters

Date_ 20011102
Time 13.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 0
SMR 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 1296.2
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 100.596134 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

CI 20.00 cm
CT 12.50 cm
F1P 215.000 ppm
F1 21625.79 Hz
F2P -5.000 ppm
F2 -502.92 Hz
PACH 11.00000 ppm/cm
HZCH 1106.4379 Hz/cm

12b - 3PNAIR

S16

p31_h1dec.gk

F2 - Acquisition Parameters

Date_ 20020524
Time 15.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 75187.969 Hz
FIDRES 1.147277 Hz
AQ 0.4358644 sec
RG 9195.2
DM 6.650 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 5.00 usec
PL1 8.00 dB
SFO1 161.9227676 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 161.9310486 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FIP 28.010 ppm
F1 4535.62 Hz
F2P 22.584 ppm
F2 3656.99 Hz
PPMCM 0.27130 ppm/cm
HZCM 43.93150 Hz/cm

h1.gk

S17

12c

F2 - Acquisition Parameters

Date_ 20020618
Time 12.59
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 400.0220001 MHz

F2 - Processing parameters

SI 32768
SF 400.0200372 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
CY 12.50 cm
F1P 10.000 ppm
F1 4000.20 Hz
F2P -0.500 ppm
F2 -200.01 Hz
PPMCH 0.52500 ppm/cm
HZCH 210.01053 Hz/cm

c13.gk

Current Data Parameters
NAME tonylee
EXPNO 2181
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020618
Time 13.43
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 1290.2
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
SF01 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
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
CA 20.00 cm
CY 12.50 cm
F1P 215.000 ppm
F1 21625.79 Hz
F2P -5.000 ppm
F2 -502.92 Hz
PPMCH 11.00000 ppm/cm
HZCH 1106.43579 Hz/cm

p31_h1dec.gk

12c - ³¹P vpr

S19

F2 - Acquisition Parameters
 Date_ 20020618
 Time 13.09
 INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 75187.969 Hz
 FIDRES 1.14777 Hz
 AQ 0.4358644 sec
 RG 4597.6
 LW 6.650 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.0002000 sec

===== CHANNEL f1 =====
 NUC1 31P
 P1 12.00 usec
 PL1 8.00 dB
 SFO1 161.9227676 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 161.9310486 MHz
 MDW EX
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 FIP 182.162 ppm
 FI 29497.62 Hz
 FZ -282.160 ppm
 F2 -45690.43 Hz
 PPMCM 23.21607 ppm/cm
 HZCM 3759.40234 Hz/cm

Integral

ppm

96.683
-1.185
4.502

100

-150

-200

520

Date_ 20020423

Time 17.01

INSTRUM spect 17.01

INSTRON 5 mm ONP 1H/13 spect

PULPROG 7030

655536
2930
FULTON

65536
cnc12

SOLVENT CDCl₃

88

0

8278.146 Hz SWH

FIDRES 0.126314 Hz

3.9584243 sec

181 RG

60.400 usec

6.00 usec

0.00 usec
300.0 K

```

15 300.0 K
01 1.0000000 sec

```

395 00000000.1

===== CHANNEL. F1 =====

===== CHANNEL 11 =====
17

1301
05 2
IH

PI	7.50 usec
PI	0.00 usec

PL1 0.00 dB

400.0220001 MHz

10

P2 - Processing parameters

32768

400.0200137 MHz

MDW EM

0 0

0.30 Hz

[illegible]

1000

NOT

in the not distant future

LD NMR plot parameters

20.00 cm

10.506 ppm

4202.47 Hz

72p -1.113 ppm

-445.36 Hz

0.58095 ppm/c

232.39168 Hz/cm⁻¹

13a - ¹³C NMR

S 21

c13k.gk

Time 20.07
 INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1600
 DS 0
 SFR 25125.629 Hz
 FIDRES 0.363387 Hz
 AQ 1.3042164 sec
 RG 2896.3
 DW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 DI 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 =====
 CPOPC2 waltz16
 NUC2 1H
 P2 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 100.5851729 MHz
 WDW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 FIP 201.812 ppm
 F1 20299.33 Hz
 F2P -0.676 ppm
 F2 -68.04 Hz
 FWHZ 10.12444 ppm/cm
 HZCN 1018.36841 Hz/cm

13a - pvr

p31_h1dec.gk

S 2 2

24.7476

ppm

F2 - Acquisition Parameters

Date_ 20020423
Time 17.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 75187.969 Hz
FIDRES 1.147277 Hz
AQ 0.4358644 sec
RG 20642.5
DM 6.650 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 5.00 usec
PL1 8.00 dB
SFO1 161.9227676 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 161.9310486 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CI 20.00 cm
F1P 55.751 ppm
F1 9027.85 Hz
F2P -8.577 ppm
F2 -1388.82 Hz
FREQH 321639 ppm/cm
HZCN 520.83311 Hz/cm

Integral

ppm

100.00

0

10

20

30

40

50

cl3k.gk

136 - 136.1

S24

F2 - Acquisition Parameters

Date_ 20010511
 Time 16.21
 INSTRUM spect
 PROBEHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1600
 DS 0
 SSB 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 456.1
 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 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 199.595 ppm
 F1 20076.26 Hz
 F2P 0.213 ppm
 F2 21.39 Hz
 PPMCH 9.96911 ppm/cm
 HZCH 1002.74323 Hz/cm

h1.gk

13C NMR

S25

F2 - Acquisition Parameters
 Date_ 20020430
 Time 16.53
 INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 362
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 0.00 dB
 SFO1 400.0220001 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.0200140 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 LD NMR plot parameters
 CX 20.00 cm
 F1P 10.363 ppm
 F1 4145.43 Hz
 F2P -0.851 ppm
 F2 -340.31 Hz
 FPMCN 0.56069 ppm/cm
 HZCN 224.28679 Hz/cm

c13k.gk

S26

13C- NMR

F2 - Acquisition Parameters

Date_ 20020430
Time 18.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1600
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 3649.1
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
FIP 202.620 ppm
F1 20380.50 Hz
F2P -2.056 ppm
F2 -206.79 Hz
FPCN 10.23377 ppm/cm
HZCN 1029.36475 Hz/cm

p31_hldec.gk

S27

13c-³¹P NMR

F2 - Acquisition Parameters

Date_ 20020430
Time 16.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 75187.969 Hz
FIDRES 1.147277 Hz
AQ 0.4358644 sec
RG 20642.5
DW 6.650 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

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

NUC1 31P
P1 5.00 usec
PL1 8.00 dB
SFO1 161.9227676 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

SF 32768
SF 161.9310486 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 62.950 ppm
F1 10193.49 Hz
F2P -11.079 ppm
F2 -1794.09 Hz
F1FCK 3.70145 ppm/cm
F2FCK 599.37939 Hz/cm

24.8993

ppm

13d

h1.gk

S 28

F2 - Acquisition Parameters

Date_ 20020617
 Time 15.53
 INSTRUM spect
 PROHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 90.5
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 400.0220001 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0200309 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D RMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 FIP 10.000 ppm
 FI 4000.20 Hz
 F2P -0.500 ppm
 F2 -200.01 Hz
 PPMCH 0.52500 ppm/cm
 RECN 210.01051 Hz/cm

c13.gk

S29

13d - cont

F2 - Acquisition Parameters
 Date_ 20020617
 Time 16.29
 INSTRUM spect
 PROBRD 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 1625.5
 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
 P2 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 100.5850630 MHz
 NW 500
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CI 20.00 cm
 CT 12.50 cm
 FL1 215.000 ppm
 F1 21625.79 Hz
 F2 -502.92 Hz
 PPMH 11.00000 ppm/cm
 HZCH 1106.43575 Hz/cm

p31_hldec.gk

S 30

13d - PDMR

F2 - Acquisition Parameters
 Date_ 20020617
 Time 15.55
 INSTRUM spect
 PROBED 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 75187.969 Hz
 FIDRES 1.147277 Hz
 AQ 0.435864 sec
 RG 7298.2
 DW 6.650 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 31P
 P1 12.00 usec
 PL1 8.00 dB
 SFO1 161.9227676 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 161.9310486 MHz
 NQW EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 FLF 182.162 ppm
 F1 29497.62 Hz
 F2 -282.160 ppm
 F2 -45690.43 Hz
 PPMCH 23.21607 ppm/cm
 HZCH 3759.40234 Hz/cm

p31_h1dec.gk

14 - PMP
S33

24.8865

55.598

F2 - Acquisition Parameters

Date_ 20010509
Time 18.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SHE 75187.969 Hz
FIDRES 1.147277 Hz
AQ 0.4358644 sec
RG 406.4
IN 6.650 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 5.00 usec
PL1 8.00 dB
SFO1 161.9227676 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 161.9310486 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FIP 29.719 ppm
F1 4815.72 Hz
F2P 21.028 ppm
F2 3405.07 Hz
PACH 0.43557 ppm/cm
HCH 70.53202 Hz/cm

ppm

Integral

ppm

16a - HINMR
S34

16a - ¹³C NMR

S35

ppm

16a - ³¹P NMR
S36

16b - H₂O

S37

ppm

16b

533

ppm

166-³¹P NMR

p31_h1dec.gk

S39

ppm