Supporting Information Part 2

Stereoselective Synthesis of 2,4,5-Trisubstituted Tetrahydropyrans using an Intramolecular Allylation Strategy

*Peter J. Jervis, Benson M. Kariuki and Liam R. Cox**

School of Chemistry, The University of Birmingham, Edgbaston, Birmingham, B15 2TT.

E-mail: l.r.cox@bham.ac.uk

14a

S55

Peter Jervis Sample 01/12/05 in CDCl₃ at -27C. set temp
drx500. 13C. 1H-decoupled

S56

14a

142.3661

135.8434

128.2699

127.3539

125.8471

117.6075

73.2727

66.6588

65.6833

43.9779

40.3339

Peter Jervis Sample 01/12/05 in CDCl₃ at +27C, set temp
 drx500, 13C-DEPT135, 1H-decoupled

EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20051130
Time 22.00
INSTRUM drx500
PROBHD 5 mm TBI H/C
PULPROG cosygp
TD 2048
SOLVENT CDC13
NS 8
DS 16
SMH 3415.301 Hz
FIDRES 1.667627 Hz
AQ 0.2998772 sec
RG 32
DM 145.400 usec
DE 5.50 usec
TE 300.0 K
d0 0.00000300 sec
d1 2.00000000 sec
d13 0.00000300 sec
d16 0.00010000 sec
IN0 0.00029280 sec

***** CHANNEL f1 *****
NUC1 1H
P0 10.90 usec
P1 10.90 usec
PL1 1.00 dB
SFO1 500.1322908 MHz

***** GRADIENT CHANNEL *****
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 10.00 %
GPZ2 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 1
TD 512
SFO1 500.1323 MHz
FIDRES 6.570509 Hz
SW 6.829 ppm

F2 - Processing parameters
SI 2048
SF 500.1300238 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300238 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 20.00 cm
CX1 20.00 cm
F2PL0 7.464 ppm
F2L0 3742.67 Hz
F2PHI 1.712 ppm
F2HI 856.21 Hz
F1PL0 7.467 ppm
F1L0 3744.54 Hz
F1PHI 1.712 ppm
F1HI 856.21 Hz
F2PPMCM 0.28859 ppm/cm
F2HZCM 144.3313 Hz/cm
F1PPMCM 0.28875 ppm/cm
F1HZCM 144.41652 Hz/cm

S58

14a

Peter Jarvis Sample 01/12/05 in CDC13 at +27C, set temp
 drx500, Gradient HSQC

14a

EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20051130
 Time 18.34
 INSTRUM drx500
 PROBRD 5 mm TBI H/C
 PULPROG invgptp
 TD 2048
 SOLVENT CDC13
 NS 8
 DS 16
 SWH 3415.301 Hz
 FIDRES 1.667627 Hz
 AQ 0.2998772 sec
 RG 32768
 DM 148.400 usec
 DE 5.50 usec
 TE 300.0 K
 CNST2 145.0000000
 d0 0.0000300 sec
 d1 2.0000000 sec
 d4 0.00172414 sec
 d11 0.0300000 sec
 d13 0.0000300 sec
 d16 0.0001000 sec
 d20 0.0010000 sec
 d21 0.00081714 sec
 dN0 0.00081590 sec

CHANNEL f1
 NUC1 1H
 P1 10.00 usec
 p2 21.80 usec
 PL1 1.00 dB
 SFO1 500.1322906 MHz

CHANNEL f2
 CPOPRG2 gprp
 NUC2 13C
 P3 12.00 usec
 p4 24.00 usec
 PCPD2 75.00 usec
 PL2 -1.00 dB
 PL12 15.00 dB
 SFO2 125.7690257 MHz

GRADIENT CHANNEL
 GPMAX1 SINE.100
 GPMAX2 SINE.100
 GPMAX3 SINE.100
 GPX1 0.00 %
 GPX2 0.00 %
 GPX3 0.00 %
 GPY1 0.00 %
 GPY2 0.00 %
 GPY3 0.00 %
 GPZ1 80.00 %
 GPZ2 30.00 %
 GPZ3 20.10 %
 P16 1000.00 usec

F1 - Acquisition parameters
 N00 4
 TO 512
 SFO1 125.769 MHz
 FIDRES 30.709513 Hz
 SN 125.017 ppm

F2 - Processing parameters
 SI 2048
 SF 500.1300236 MHz
 MDW 65INE
 SSB 2
 LB 0.00 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 1024
 NC2 TPPI
 SF 125.7578064 MHz
 MDW 65INE
 SSB 2
 LB 0.00 Hz
 GB 0

2D NMR plot parameters
 CX2 17.00 cm
 CX1 17.00 cm
 F2PLD 7.524 ppm
 F2L0 3762.88 Hz
 F2PHI 1.738 ppm
 F2HI 889.55 Hz
 F1PLD 136.853 ppm
 F1L0 17222.96 Hz
 F1PHI 38.685 ppm
 F1HI 4892.36 Hz
 F2PCN0 0.34030 ppm/cm
 F2CCW 170.19608 Hz/cm
 F1PCN0 9.78159 ppm/cm
 F1CCW 727.08283 Hz/cm

S59

Peter Jervis Sample 01/12/05 in CDCl3 at +27C, set temp
drx500, Gradient HMBC

14a

NAME
EXPNO
PROCNO

F2 - Acquisition Parameters

Date_ 20051130
Time 19.14
INSTRUM drx500
PROBHD 5 mm TBI H/C
PULPROG inv4gplred
TO 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 3415.301 Hz
FIDRES 1.667627 Hz
AQ 0.2988772 sec
RG 32768
DM 146.400 usec
DE 5.00 usec
TE 300.0 K
DST2 150.000000
d0 0.00000300 sec
d1 2.00000000 sec
d2 0.00312500 sec
d6 0.10000000 sec
d13 0.00000300 sec
d16 0.00010000 sec
IN0 0.0003150 sec

CHANNEL f1
NUC1 1H
P1 10.90 usec
p2 21.80 usec
PL1 1.00 dB
SFO1 500.1322909 MHz

CHANNEL f2
NUC2 13C
P3 12.00 usec
PL2 -1.00 dB
SFO2 125.760257 MHz

GRADIENT CHANNEL
GPMAX1 SINE.100
GPMAX2 SINE.100
GPMAX3 SINE.100
SPX1 0.00 %
SPX2 0.00 %
SPX3 0.00 %
SPY1 0.00 %
SPY2 0.00 %
SPY3 0.00 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
NO 2
TO 512
SFO1 125.760 MHz
FIDRES 30.709513 Hz
SW 125.017 ppm

F2 - Processing parameters
SI 2048
SF 500.1300238 MHz
WDW GSSINE
SSB 2
LB 0.00 Hz
GB 0
PC 0.20

F1 - Processing parameters
SI 1024
MC2 OF
SF 125.7578054 MHz
WDW GSSINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 17.00 cm
CX1 17.00 cm
F2PLO 6.233 ppm
F2LO 3117.51 Hz
F2PHI 3.666 ppm
F2H1 1783.41 Hz
F1PLO 80.910 ppm
F1LO 10175.12 Hz
F1PHI 35.490 ppm
F1H1 4463.15 Hz
F2PWH 0.19691 ppm/cm
F2QWH 76.47598 Hz/cm
F1PWH 2.67179 ppm/cm

S60

Peter Jervis Sample 01/12/05 in CDCl₃ at +27°C, set temp
drx500, Gradient HMB

14a

14a

S62

14a

14a

14b

S65

Peter Jervis Sample 06/02/06 in CDCl₃ at +27C, set temp
drx500, 13C, 1H-decoupled

S66

14b

Peter Jervis Sample 06/02/06 in CDC13 at +27C, set temp
drx500, Gradient HSQC

14b

NAME f908p1d
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060206
Time 17.29
INSTRUM crys300
PROBHD 5 mm TSI H/C
PULPROG invgptp
TD 2048
SOLVENT CDC13
NS 8
DS 16
SWH 4045.307 Hz
FIDRES 1.975248 Hz
AQ 0.2631828 sec
RG 32788
DM 123.600 usec
DE 5.56 usec
TE 300.0 K
CMT2 145.0000000
d0 0.0000000 sec
d1 2.0000000 sec
d4 0.00172414 sec
d11 0.03000000 sec
d13 0.00000000 sec
d16 0.0010000 sec
d20 0.00110000 sec
d21 0.00061714 sec
dN0 0.0001340 sec

CHANNEL f1

NUC1 1H
P1 10.00 usec
p2 21.20 usec
PL1 1.00 dB
SFO1 500.1318637 MHz

CHANNEL f2

CPDPRG2 gpcp
NUC2 13C
P3 12.00 usec
p4 24.00 usec
PCPD2 76.00 usec
PL2 -1.00 dB
PL12 15.00 dB
SFO2 125.7671861 MHz

GRADIENT CHANNEL

GRAN1 SIZE:100
GRAN2 SIZE:100
GRAN3 SIZE:100
SPX1 0.00 %
SPX2 0.00 %
SPX3 0.00 %
SPY1 0.00 %
SPY2 0.00 %
SPY3 0.00 %
SPZ1 80.00 %
SPZ2 30.00 %
SPZ3 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters

MD0 4
TD 512
SFO1 125.7672 MHz
FIDRES 36.438900 Hz
SW 148.343 ppm

F2 - Processing parameters

SI 2048
SF 500.1300238 MHz
WDW 0.0100
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters

SI 1024
NC2 TPPI
SF 125.7577945 MHz
WDW 0.0100
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters

CK2 17.30 cm
CK1 17.00 cm
F2PL0 5.891 ppm
F2L0 2946.11 Hz
F2PHI 0.764 ppm
F2H1 382.24 Hz
F1PL0 138.426 ppm
F1L0 17408.17 Hz
F1PHI 11.598 ppm
F1H1 1466.15 Hz
F2PCW 0.30195 ppm/cm
F2DCW 150.81096 Hz/cm
F1PCW 7.45992 ppm/cm

S69

Peter Jarvis Sample 06/02/06 in CDCl₃ at +27°C, set temp
dnx500, Gradient HSGC

S70

14b

14b

14b

14b

14c

14c

— 136.0767

— 117.4874

— 69.2170

— 66.6582

— 65.2456

— 45.2284

— 44.3184

— 38.9038

— 24.3508

— 23.3227

— 22.2755

S75

14d

S76

14d

S78

14e

14e

— 138.5403
 — 135.9967
 — 128.2861
 — 127.5817
 — 127.4370
 — 117.4189

— 72.9181
 — 68.4098
 — 66.8563
 — 66.5174
 — 65.2684

— 44.2879
 — 38.6145
 — 36.1052

14f

14f

136.0234
 135.6159
 133.6588
 129.5541
 127.5817

117.4874

71.7111

67.0390
 66.4298
 65.1161

44.2499

34.9096

26.8753

19.3132

S81

500 MHz

14g

S82

14g

— 154.4560

— 142.2948

— 135.6412

— 117.8811

— 110.0498

— 106.7627

66.7817
 66.2385
 65.5604

— 43.8629

— 36.0674

S83

14g

— 142.2987

— 135.6372

— 117.8890

— 110.0538

— 106.7627

— 66.7778
— 66.2385
— 65.5565

— 43.8590

— 36.0634

S84

9. CDCl₃

Peter Jervis Sample 04/07/06 in CDCl3 at +27C, set temp
drx500, Gradient COSY90

14g

Current Data Parameters
NAME j103p1id
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060704
Time 21.47
INSTRUM drx500
PROBHD 5 mm TBI H/C
PULPROG cosy90
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 4166.567 Hz
FIDRES 2.034505 Hz
AQ 0.2456100 sec
RG 406.4
CW 120.000 usec
DE 5.50 usec
TE 300.0 K
d0 0.00000300 sec
d1 2.00000000 sec
d13 0.00000300 sec
d16 0.00010000 sec
IN0 0.00024000 sec

===== CHANNEL f1 =====
NUC1 1H
P0 11.00 usec
P1 11.00 usec
PL1 1.00 dB
SFO1 500.1319360 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 10.00 %
GPZ2 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 1
TD 512
SFO1 500.1319 MHz
FIDRES 0.138021 Hz
SW 8.331 ppm

F2 - Processing parameters
SI 2048
SF 500.1300238 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300238 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 20.00 cm
CX1 20.00 cm
F2PLO 7.525 ppm
F2LO 3763.64 Hz
F2PHI 1.139 ppm
F2HI 569.47 Hz
F1PLO 7.566 ppm
F1LO 3783.98 Hz
F1PHI 1.122 ppm
F1HI 561.33 Hz
F2PPMCM 0.31833 ppm/cm
F2HZCM 159.70866 Hz/cm
F1PPMCM 0.31833 ppm/cm
F1HZCM 159.70866 Hz/cm

S85

14g

2D NMR plot parameters	
CX2	17.00 cm
CX1	17.00 cm
F2PLD	7.493 ppm
F2L	3747.36 Hz
F2PHI	1.944 ppm
F2HI	972.30 Hz
F1PLD	145,840 ppm
F1L	18340.46 Hz
F1PHI	34.693 ppm
F1HI	4357.86 Hz
F2PHCON	0.32839 ppm/cm
F2HCON	163.23914 Hz/cm
F1PHCON	6.54040 ppm/cm
F1HCON	822.50586 Hz/cm

14g

Peter Jervis Sample 04/07/06 in CDCl3 at +27C, set temp
drx500, Gradient HMBC

14g

NAME j103pjid
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060704
Time 19.04
INSTRUM drx500
PROBHD 5 mm TBI H/C
PULPROG invgdp17md
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 4166.067 Hz
FIDRES 2.034665 Hz
AQ 0.2459100 sec
RG 32768
OR 120.000 usec
DE 5.50 usec
TE 300.0 K
CMST2 160.000000 sec
d0 0.0000000 sec
d1 2.0000000 sec
d2 0.00312500 sec
d6 0.1000000 sec
d13 0.0000000 sec
d16 0.00010000 sec
d10 0.00002560 sec

CHANNEL f1
NUC1 1H
P1 11.00 usec
p2 22.00 usec
PL1 1.00 dB
SFO1 500.1319360 MHz

CHANNEL f2
NUC2 13C
P3 12.00 usec
PL2 -1.00 dB
SFO2 125.7594785 MHz

GRADIENT CHANNEL
GPMAX1 SINE.100
GPMAX2 SINE.100
GPMAX3 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
BPZ1 50.00 %
BPZ2 30.00 %
BPZ3 40.10 %
P15 1000.00 usec

F1 - Acquisition parameters
MD 2
TD 512
SFO1 125.7595 MHz
FIDRES 34.148642 Hz
SW 139.004 ppm

F2 - Processing parameters
SI 2048
SF 500.1300238 MHz
WDW GSINE
SSB 2
LB 0.00 Hz
GB 0
PC 0.20

F1 - Processing parameters
SI 1024
MC2 GF
SF 125.7577845 MHz
WDW GSINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 17.00 cm
CX1 17.00 cm
F2PL0 5.581 ppm
F2L0 2781.15 Hz
F2PHI 3.628 ppm
F2HI 1614.56 Hz
F1PL0 157.922 ppm
F1L0 19699.94 Hz
F1PHI 100.963 ppm
F1HI 12689.36 Hz
F2PPMCH 0.11486 ppm/cm
F2HZCH 57.44485 Hz/cm
F1PPMCH 3.35405 ppm/cm
F1HZCH 421.79791 Hz/cm

S88

Peter Jervis Sample 04/07/06 in CDC13 at +27C, set temp
drx500, Gradient HMBC

14g

Current Data Parameters
NAME j103p1d
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20080704
Time 19.04
INSTRUM drx500
PROBHD 5 mm TBI H/C
PULPROG inv4gplirnd
TD 2048
SOLVENT CDC13
NS 8
DS 16
SWH 4166.867 Hz
FIDRES 2.034505 Hz
AQ 0.2498100 sec
RG 32788
DM 120.000 usec
DE 5.50 usec
TE 300.0 K
CHST2 150.0000000
d0 0.00000300 sec
d1 2.00000000 sec
d2 0.00312500 sec
d6 0.10000000 sec
d13 0.00000300 sec
d16 0.00010000 sec
IN0 0.00002860 sec

----- CHANNEL f1 -----
NUC1 1H
P1 11.00 usec
p2 22.00 usec
PL1 1.00 dB
SFO1 500.1319360 MHz

----- CHANNEL f2 -----
NUC2 13C
P3 12.00 usec
PL2 -1.00 dB
SFO2 125.7694765 MHz

----- GRADIENT CHANNEL -----
GPMAX1 SINE.100
GPMAX2 SINE.100
GPMAX3 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
NDS 2
TD 512
SFO1 125.7695 MHz
FIDRES 34.145542 Hz
SW 139.004 ppm

F2 - Processing parameters
SI 2048
SF 500.1300235 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 0.20

F1 - Processing parameters
SI 1024
MC2 SF
SF 125.7577945 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 17.00 cm
CX1 17.00 cm
F2PLO 2.819 ppm
F2LO 1409.72 Hz
F2PHI 1.863 ppm
F2HI 831.51 Hz
F1PLO 156.972 ppm
F1LO 19740.43 Hz
F1PHI 115.701 ppm
F1HI 14980.31 Hz
F2PPMCH 0.00623 ppm/cm
F2QZCH 26.12404 Hz/cm
F1PPMCH 2.42769 ppm/cm

S89

14g

14g

14g

14g

14g

14h

Peter Jervis Sample 27/02/06 in CDCl₃ at +27C, set temp
drx500, 13C, 1H-decoupled

S96

Peter Jervis Sample 27/02/06 in CDCl₃ at +27C, set temp
drx500, 13C-DEPT135, 1H-decoupled

S97

14h

135.8037
130.4269
129.9670
128.4840
127.5244
126.4221

117.7780

71.6232

66.4288
65.2155

43.9383

38.5773

14h

NAME fe27p1d
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060227
Time 22.43
INSTRUM drx500
PROBHD 5 mm TBI H/C
PULPROG cosygp
TD 2048
SOLVENT CDC13
NS 8
DS 16
SWH 4251.701 Hz
FIDRES 2.075026 Hz
AQ 0.2408948 sec
RG 574.7
DW 117.600 usec
DE 5.50 usec
TE 300.0 K
d0 0.00000300 sec
d1 2.00000000 sec
d13 0.00000300 sec
d15 0.00010000 sec
IN0 0.00023520 sec

===== CHANNEL f1 =====
NUC1 1H
P0 10.60 usec
P1 10.60 usec
PL1 1.00 dB
SF01 500.1320017 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 10.00 %
GPZ2 10.00 %
P15 1000.00 usec

F1 - Acquisition parameters
ND0 1
TD 512
SF01 500.132 MHz
FIDRES 8.304103 Hz
SW 8.501 ppm

F2 - Processing parameters
SI 2048
SF 500.1300238 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300238 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 20.00 cm
CX1 20.00 cm
F2PL0 7.491 ppm
F2LO 3746.71 Hz
F2PHI 1.572 ppm
F2HI 786.30 Hz
F1PL0 7.550 ppm
F1LO 3775.78 Hz
F1PHI 1.564 ppm
F1HI 782.15 Hz
F2PPNCH 0.29596 ppm/cm
F2HZCH 148.02065 Hz/cm
F1PPNCH 0.29929 ppm/cm
F1HZCH 149.68146 Hz/cm

S98

S99

14h

Handwritten 1H and 13C NMR spectra. The 1H NMR spectrum (top) shows peaks at approximately 7.2 (d, 2H), 6.8 (d, 2H), 6.5 (t, 1H), 5.2 (d, 2H), 4.8 (d, 2H), 4.2 (t, 2H), 3.8 (s, 3H), and 1.8 (s, 3H). The 13C NMR spectrum (bottom) shows peaks at approximately 155, 150, 145, 135, 130, 125, 120, 115, 110, 105, 100, 95, 90, 85, 80, 75, 70, 65, 60, 55, 50, 45, 40, 35, 30, 25, 20, 15, 10, 5, and 0 ppm. The sample is identified as "Sample for analysis".

2D NMR plot parameters	
C12	17.00 cm
C11	17.00 cm
F2P1D	7.564 ppm
F2L0	3752.94 Hz
F2P1H	1.610 ppm
F2H1	804.95 Hz
F1P1D	137.533 ppm
F1L0	17295.91 Hz
F1P1H	36.282 ppm
F1H1	4562.71 Hz
F2P1MCH	0.34673 ppm/cm
F2H1MCH	173.40921 Hz/cm
F1P1MCH	8.95599 ppm/cm

14h

14h

Peter Jervis Sample 27/02/06 in CDCl3 at +27C, set temp
drx500, Gradient HMBC

14h

NAME fe27pjid
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060227
Time 20.01
INSTRUM drx500
PROBHD 5 mm TBI H/C
PULPROG invgcpbprnd
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 4251.701 Hz
FIDRES 2.076028 Hz
AQ 0.2408948 sec
RG 32768
CW 117.500 usec
DE 5.50 usec
TE 300.0 K
CHST2 160.0000000
d0 0.00000300 sec
d1 2.00000000 sec
d2 0.00312500 sec
d5 0.10000000 sec
d13 0.00000300 sec
d16 0.00010000 sec
TNO 0.00002320 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.60 usec
p2 21.20 usec
PL1 1.00 dB
SFO1 500.1320017 MHz

----- CHANNEL f2 -----
NUC2 13C
P3 12.00 usec
PL2 -1.00 dB
SFO2 125.7679977 MHz

----- GRADIENT CHANNEL -----
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 2
TD 512
SFO1 125.766 MHz
FIDRES 42.093212 Hz
SW 171.364 ppm

F2 - Processing parameters
SI 2048
SF 500.1300238 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 0.20

F1 - Processing parameters
SI 1024
HC2 0F
SF 125.7577945 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CK2 17.00 cm
CK1 17.00 cm
F2PLD 7.628 ppm
F2LO 3815.22 Hz
F2PHI 1.145 ppm
F2HI 572.47 Hz
F1PLD 141.717 ppm
F1LO 17622.07 Hz
F1PHI 27.077 ppm
F1HI 3405.15 Hz
F2PPMCH 0.38140 ppm/cm
F2HZCH 190.75014 Hz/cm
F1PPMCH 6.74395 ppm/cm
F1HZCH 845.05444 Hz/cm

S102

14h

Peter Jervis Sample 27/02/06 in CDCl₃ at +27C, set temp
dpx500, Gradient HMBC

14h

14h

Peter Jervis Sample 27/02/06 in CDCl₃ at +27C, set temp,
drx500, 1D-GOESY

S105

14h

14h

14h

Peter Jervis Sample 27/02/06 in CDCl₃ at +27C, set temp,
drx500, 1D-GOESY

S109

14h

15a

15a

S111

15b

15b

S113

17a

S114

S115

Peter Jervis Sample 05/04/06 in CDCl₃ at +27C, set temp
drx500, 13C, 1H-decoupled

S116

Peter Jervis Sample 05/04/06 in CDCl3 at +27C, set temp
drx500, 13C-DEPT135, 1H-decoupled

Peter Jervis Sample 05/04/06 in CDCl3 at +27C, set temp
drx500, Gradient COSY90

17a

NAME ap00pjid
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060404
Time 21.49
INSTRUM drx500
PROBHD 5 mm TBI H/C
PULPROG cosygp
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 4251.701 Hz
FIDRES 2.076026 Hz
AQ 0.2408948 sec
RG 128
DN 117.600 usec
DE 5.50 usec
TE 300.0 K
d0 0.00000300 sec
d1 2.00000000 sec
d13 0.00000300 sec
d16 0.00010000 sec
IN0 0.00023520 sec

===== CHANNEL f1 =====
NUC1 1H
P0 10.70 usec
P1 10.70 usec
PL1 1.00 dB
SFO1 500.1319130 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 10.00 %
GPZ2 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 1
TD 512
SFO1 500.1319 MHz
FIDRES 8.304103 Hz
SW 8.501 ppm

F2 - Processing parameters
SI 2048
SF 500.1300238 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 GF
SF 500.1300238 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 20.00 cm
CX1 20.00 cm
F2PLO 7.451 ppm
F2LO 3726.52 Hz
F2PHI 1.822 ppm
F2HI 911.43 Hz
F1PLO 7.522 ppm
F1LO 3761.62 Hz
F1PHI 1.818 ppm
F1HI 909.36 Hz
F2PPMCH 0.29144 ppm/cm
F2HZCH 140.75455 Hz/cm
F1PPMCH 0.28517 ppm/cm
F1HZCH 142.62297 Hz/cm

S117

17a

Peter Jervis Sample 05/04/06 in CDCl3 at +27C, set temp
drx500, Gradient HSGC

17a

NAME ap05p1d
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20090404
Time 16.32
INSTRUM drx500
PROBHD 5 mm TBI H/C
PULPROG invigptp
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 4051.701 Hz
FIDRES 2.078626 Hz
AQ 0.2409948 sec
RG 32768
DM 117.600 usec
DE 5.50 usec
TE 300.0 K
CNS12 145.0000000
d0 0.0000300 sec
d1 2.0000000 sec
d4 0.00172414 sec
d11 0.0300000 sec
d13 0.0000300 sec
d16 0.0010000 sec
d20 0.0011000 sec
d21 0.00051714 sec
d40 0.00051695 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.70 usec
p2 21.40 usec
PL1 1.00 dB
SFO1 500.139130 MHz

----- CHANNEL f2 -----
CPCPRG2 garp
NUC2 13C
P3 12.00 usec
p4 24.00 usec
PCPR2 76.00 usec
PL2 -1.00 dB
PL12 15.00 dB
SFO2 125.7687641 MHz

----- GRADIENT CHANNEL -----
GPMAN1 SINE.100
GPMAN2 SINE.100
GPMAN3 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
SPY1 0.00 %
SPY2 0.00 %
SPY3 0.00 %
GPZ1 80.00 %
GPZ2 30.00 %
GPZ3 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters
M00 4
TD 512
SFO1 125.7686 MHz
FIDRES 30.422508 Hz
SK 123.646 ppm

F2 - Processing parameters
SI 2048
SF 500.1305238 MHz
WDW 05SINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 TPP1
SF 125.7577845 MHz
WDW 05SINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters

CK2 17.00 cm
CK1 17.00 cm
F2PL0 7.534 ppm
F2L0 3768.04 Hz
F2PHI 1.125 ppm
F2H1 264.74 Hz
F2PL1 130.410 ppm
F2L1 16400.09 Hz
F2PHI2 37.515 ppm
F2H1 4717.84 Hz
F2PHI2CH 0.37676 ppm/cm
F2H2CH 188.42987 Hz/cm
F1PH2CH 5.48440 ppm/cm
F1H2CH 687.19080 Hz/cm

S119

17a

Peter Jervis Sample 05/04/06 in CDCl₃ at +27°C, set temp
drx500, Gradient HSQC

S121

17a

17b

S122

17b

S123

S124

7a

7a

S125

S126

7b

S127

7c

7c

7d

S131

7e

7e

S133

7f

7f

74.5821

65.5731
65.2418

46.3936

26.7801

19.2180
19.1609

-1.9148

S135

7g

7g

S137

7h

7h

S139

S140

16a

16a

S141

S142

16b

16b

— 202.4204

S143

— 84.7259

— 74.5691
— 73.0316

— 56.7823

— 47.7451

— 33.4090

— 26.8257

— 22.2477

— 13.4789

— 6.5965

— -2.6401

S144

13a

13a

S145

S146

13b

S147

13c

S149

13d

S150

13d

S151

S152

13e

S153

13e

¹H NMR spectrum of compound **1** in CDCl₃. The spectrum shows peaks from 0 to 8 ppm. Key features include: aromatic signals at 7.3–7.7 ppm (integrals 3.8225, 5.6306); a multiplet at 5.3–5.6 ppm (integrals 0.8182, 0.9098); a multiplet at 3.8–4.2 ppm (integral 4.6716); a multiplet at 2.5–2.7 ppm (integrals 1.0000, 1.0008); a multiplet at 1.5–1.7 ppm (integrals 1.7544, 3.1854); a large peak at 1.1 ppm (integral 8.6680); and a TMS peak at 0.0 ppm (integral 7.5373). Chemical shift values are labeled above the peaks.

13g

13h

S159

12a

12a

S161

12b

S163

12b

S164

12c

12c

12d

12d

S167

12e

12e

12f

12g

12g

12h

12h

10

9

S177

9

S178