Solution syntheses of protected Type-II Lewis blood group oligosaccharides: a study for automated synthesis

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Supplementary Information

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General Methods. All chemicals were reagent grade and used as supplied unless otherwise noted. Dichloromethane (CH_2Cl_2), tetrahydrofuran (THF), and toluene were purified by a JT Baker Cycle-Tainer Solvent Delivery System. Analytical thin-layer chromatography was performed on E. Merck silica gel 60 F₂₅₄ plates (0.25 mm). Compounds were visualized by dipping the plates in a cerium sulfate-ammonium molybdate solution followed by heating. Liquid flash column chromatography was performed using forced flow of the indicated solvent on Silicycle silica (230 - 400 mesh). ^1H NMR spectra were obtained using a 400 MHz or 500 MHz spectrometer and are reported in parts per million (δ) relative to CDCl_3 (7.27 ppm). Coupling constants (J) are reported in Hertz (Hz). ^{13}C NMR spectra were obtained at either 100 MHz or 125 MHz and are reported in δ relative to CDCl_3 (77.23 ppm) as an internal reference. ^{31}P spectra were obtained at 120 MHz and are reported in δ relative to H_3PO_4 (0.0 ppm) as an external reference. Optical rotations were measured at room temperature.

Precursor to Compound 23

Current Data Parameters
NAME May29-2001-1k1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010529
Time 15:29

INSTRUM spect
PROBHD 5mm BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 80.6
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.90 usec
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 8.196 ppm
F1 3279.57 Hz
F2P -0.158 ppm
F2 -63.36 Hz
PPMCM 0.41773 ppm/cm
HZCM 167.14641 Hz/cm

Precursor to Compound 23

Current Data Parameters
NAME May29-2001-1k1
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010529
Time 15.37
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 48
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 2298.8
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 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

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

CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127493 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 199.346 ppm
F1 20056.79 Hz
F2P -10.637 ppm
F2 -1090.39 Hz
PPMCH 10.50920 ppm/cm
HZCM 1057.35913 Hz/cm

Precursor to Compound 23

Current Data Parameters
NAME May22-2001-Ik1
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010522
Time 14.15
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SMH 8278.146 Hz
FTRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.90 usec
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 8.153 ppm
F1 3262.23 Hz
F2P -0.048 ppm
F2 -19.12 Hz
PPNCH 0.41004 ppm/cm
HZCM 164.06738 Hz/cm

Precursor to Compound 23

Current Data Parameters
NAME May22-2001-1k1
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010522
Time 14.25
INSTRUM spect
PROBHD 5mm BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 31
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 16384
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 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

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

CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127516 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
FIP 219.758 ppm
F1 22110.47 Hz
F2P -9.533 ppm
F2 -959.18 Hz
PPMCM 11.46458 ppm/cm
HZCM 1153.48267 Hz/cm

Compound 23

Current Data Parameters
NAME Jun20-2001-1k1
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010620
Time 18.06
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 40.3
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

CHANNEL f1

NUC1 ¹H
P1 7.90 usec
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.086 ppm
F1 3235.59 Hz
F2P -0.048 ppm
F2 -19.37 Hz
PPMCM 0.40674 ppm/cm
HZCM 162.74783 Hz/cm

Compound 23

Current Data Parameters
NAME Jun20-2001-Ik1
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010620
Time 18.14
INSTRUM spect
PROBHD 5mm BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 50
DS 4
SWH 25125.629 Hz
FIDRES 0.363387 Hz
AQ 1.3042164 sec
RG 4096
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 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127547 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1 221.055 ppm
F2 22240.91 Hz
F2p -10.625 ppm
F2 -1069.06 Hz
PPMCM 11.58400 ppm/cm
HZCM 1165.49805 Hz/cm

Precursor to Compound 7

Current Data Parameters
NAME Jun27-2001-Ik1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010627
Time 14.28
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

NUC1 1H
P1 7.90 usec
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.043 ppm
F1 3218.24 Hz
F2P 0.062 ppm
F2 24.87 Hz
PPMCM 0.39904 ppm/cm
HZCM 159.66879 Hz/cm

Precursor to Compound 7

Current Data Parameters
NAME Jun27-2001-1k1
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010627
Time 14.35
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 162
DS 4
SWH 25125.629 Hz
FIDRES 0.385387 Hz
AQ 1.3042164 sec
RG 4096
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 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127516 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.289 ppm
F1 22163.87 Hz
F2P -0.245 ppm
F2 -24.65 Hz
PPMCM 11.02669 ppm/cm
HZCM 1109.42503 Hz/cm

Compound 7

Current Data Parameters
 NAME Jun28-2001-Ik1
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010628
 Time 12.05
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 362
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

----- CHANNEL f1 -----
 NUC1 ¹H
 P1 7.90 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300049 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.076 ppm
 F1 3631.71 Hz
 F2P -0.026 ppm
 F2 -10.32 Hz
 PPMCM 0.45511 ppm/cm
 HZCM 182.10161 Hz/cm

Compound 7

Current Data Parameters
NAME Jun29-2001-1k1
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010629
Time 16.17
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 1024
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 574.7
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 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127507 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.644 ppm
F1 22199.64 Hz
F2P 0.594 ppm
F2 59.79 Hz
PPMCH 11.00251 ppm/cm
HZCM 1106.99231 Hz/cm

S14

Compound 9

Current Data Parameters
NAME J0102-2001-1k1
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010703
Time 5.37

INSTRUM spect
PROBHD 5mm BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10000
DS 4

SH1 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127501 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.077 ppm
F1 22142.54 Hz
F2P 0.027 ppm
F2 2.69 Hz
PRMCH 11.00251 ppm/cm
HZCH 1106.99231 Hz/cm

Compound 11

Current Data Parameters
NAME Jul10-2001-Ik1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010710
Time 10.22
INSTRUM spect
PROBHD 5mm BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SMH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.90 usec
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.343 ppm
F1 3338.19 Hz
F2P 0.155 ppm
F2 62.20 Hz
PPMCM 0.40937 ppm/cm
HZCM 163.79964 Hz/cm

Compound 11

Current Data Parameters
NAME Jul12-2001-1k1
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010713
Time 8.45
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 13514
DS 4
SWH 25125.629 Hz
FIDRES 0.383367 Hz
AQ 1.3042164 sec
RG 2048
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 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127501 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.200 ppm
F1 20142.63 Hz
F2P 0.027 ppm
F2 2.69 Hz
PPMCM 10.00864 ppm/cm
HZCM 1006.99689 Hz/cm

S18

Compound 14

Current Data Parameters
NAME ps15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031025
Time 13.06
INSTRUM drx2500
PROBHD 5 mm BBO BB-
PULPROG zg45-cpd
TD 131072
SOLVENT CDC13
NS 60000
DS 0
SWH 31152.648 Hz
FIDRES 0.237676 Hz
AQ 2.1037557 sec
RG 13004
DW 16.050 usec
DE 30.00 usec
TE 298.0 K
D1 0.30000001 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SF01 125.7715724 MHz

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

CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -5.00 dB
PL12 13.50 dB
PL13 15.50 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 131072
SF 125.7577908 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 209.745 ppm
F1 26377.05 Hz
F2P 31879 ppm
F2 487.85 Hz
PPMCM 10.29328 ppm/cm
HZCM 1294.46021 Hz/cm

Chemical structure of compound 10, a branched oligosaccharide. The structure features a central NHTCAPIVO unit (a 1,2:3,6-di-O-isopropylidene- α -D-glucopyranose derivative) linked via glycosidic bonds to various sugar derivatives. The branches include units with BzO (benzoyl), OBn (benzyl), and OPiv (isopropyl) protecting groups, and a terminal unit with a vinyl group.

Compound 16

Current Data Parameters
NAME ps14
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031024
Time 13.35
INSTRUM drx2500
PROBHD 5 mm BBO BB-
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 4943
DS 0
SWH 31152.648 Hz
FIDRES 0.237676 Hz
AQ 2.1037557 sec
RG 13004
DW 16.050 usec
DE 30.00 usec
TE 298.0 K
D1 0.30000001 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C

PL1 9.00 usec
PL1 0.00 dB
SFO1 125.7715724 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16

NUC2 1H
PCPD2 80.00 usec
PL2 -5.00 dB
PL12 13.50 dB
PL13 15.50 dB
SFO2 500.1320005 MHz

F2 - Processing parameters

SI 131072
SF 125.7577664 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 220.443 ppm
F1 27722.39 Hz
F2P -1.185 ppm
F2 -149.00 Hz
PPMCM 11.08138 ppm/cm
HZCM 1393.56958 Hz/cm

Compound 19

Current Data Parameters
 NAME Sep05-2001-1K1
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010905
 Time 20.32
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 228.1
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 ¹H
 P1 7.90 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300049 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters.
 CX 20.00 cm
 F1P 7.941 ppm
 F1 3177.34 Hz
 F2P 0.304 ppm
 F2 121.60 Hz
 PPMCM 0.38184 ppm/cm
 HZCM 152.78693 Hz/cm

Compound 19

Current Data Parameters
NAME ps16
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031028
Time 9.14
INSTRUM drx2500
PROBHD 5 mm BBO BB-
PULPROG zg45_cpd
TD 131072
SOLVENT CDCl3
NS 32213
DS 0
SWH 31152.648 Hz
FIDRES 0.237676 Hz
AQ 2.1037557 sec
RG 13004
DW 16.050 usec
DE 30.00 usec
TE 298.0 K
D1 0.30000001 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SF01 125.7715724 MHz

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

CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -5.00 dB
PL12 13.50 dB
PL13 15.50 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 131072
SF 125.7577606 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
FIP 220.181 ppm
F1 27689.50 Hz
F2P 1.316 ppm
F2 165.53 Hz
PPMCM 10.94325 ppm/cm
HZCM 1376.19824 Hz/cm

Compound 36

Current Data Parameters
 NAME Sep27-2001-IK1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010927
 Time 11.31
 INSTRUM spect
 PROBHD 5mm BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 43
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 203.2
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.90 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300049 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10 NMR plot parameters
 CX 20.00 cm
 F1P 8.251 ppm
 F1 3301.32 Hz
 F2P 0.063 ppm
 F2 25.33 Hz
 PPMCM 0.40937 ppm/cm
 HZCM 163.79964 Hz/cm

Compound 36

Current Data Parameters
NAME Sep27-2001-1k1
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010927
Time 11.42
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 326
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 1024
OW 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 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127478 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.660 ppm
F1 22201.17 Hz
F2P 0.050 ppm
F2 4.99 Hz
PPMCH 11.03050 ppm/cm
HZCH 1109.80908 Hz/cm

Compound 36

LOC ETHZ NMR Gemini 300 MHz Nr.3 10/24/03 13:14:47 USER:plove GROUP:seeber SAMPLE:OBZgalphos

P-31 STANDARD PARAMETERS PHOSPHATE REGION

File: PHOSPHORUS

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: plove

GEMINI-300BB. "gem3oc"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.002 sec

Width 31948.9 Hz

32 repetitions

OBSERVE P31, 121.4760004 MHz

DECOUPLE H1, 300.0848347 MHz

Power 42 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 min, 13 sec

2.037

Compound 32

Current Data Parameters
 NAME Oct20-2001-Ik1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011020
 Time 11.02
 INSTRUM spect
 PROBHD 5mm BB0 BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 256
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.90 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300049 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.158 ppm
 F1 3264.20 Hz
 F2P 0.110 ppm
 F2 43.89 Hz
 PPMCM 0.40241 ppm/cm
 HZCM 161.01549 Hz/cm

Compound 32

Current Data Parameters
 NAME Oct22-2001-Ik1
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011023
 Time 9.10
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 14550
 DS 4
 SHF 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 4597.6
 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 15.25 usec
 PL1 3.00 dB
 SF01 100.6237959 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127470 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 FIP 221.227 ppm
 F1 22256.28 Hz
 F2P 0.337 ppm
 F2 33.93 Hz
 PPMCM 11.04450 ppm/cm
 HZCM 1111.21765 Hz/cm

Compound 33

Current Data Parameters
 NAME Oct22-2001-Ik1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011022
 Time 9.39
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 406.4
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.90 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1299925 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.328 ppm
 F1 3332.26 Hz
 F2P 0.210 ppm
 F2 84.11 Hz
 PPMCM 0.40589 ppm/cm
 HZCM 162.40758 Hz/cm

Compound 33

Current Data Parameters
 NAME Dec17-2001-1k1
 EXPNO 50
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011217
 Time 12.53
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2550
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 8192
 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 15.25 usec
 PL1 3.00 dB
 SF01 100.6237959 MHz

===== CHANNEL f2 =====
 CPOPRG2 waltz16
 NUC2 1H
 PCPD2 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 S1 32768
 SF 100.6127555 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.303 ppm
 F1 22165.34 Hz
 F2P 1.373 ppm
 F2 138.16 Hz
 PRACH 10.94651 ppm/cm
 HZCM 1101.35869 Hz/cm

Compound 34

Current Data Parameters
NAME Dec17-2001-Ik1
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011217
Time 19.00
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.90 usec
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 8.156 ppm
F1 3263.45 Hz
F2P 0.131 ppm
F2 52.42 Hz
PPHCH 0.40125 ppm/cm
HZCM 160.55148 Hz/cm

Compound 34

Current Data Parameters
NAME Dec17-2001-Ik1
EXPNO 90
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011218
Time 9.49
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15740
DS 4
SWH 25125.629 Hz
FIDRES 0.363387 Hz
AQ 1.3042164 sec
RG 2048
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 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127486 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID NMR plot parameters
CX 20.00 cm
F1P 220.652 ppm
F1 22200.40 Hz
F2P 0.602 ppm
F2 60.56 Hz
PPMCM 11.00251 ppm/cm
HZCM 1106.95231 Hz/cm

Compound 35

Current Data Parameters
 NAME Nov05-2001-1k1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011106
 Time 12.32
 INSTRUM spect
 PROBD 5mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 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 7.90 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300056 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 7.970 ppm
 F1 3189.20 Hz
 F2P 0.108 ppm
 F2 43.14 Hz
 PPMCM 0.39313 ppm/cm
 HZCM 157.30333 Hz/cm

Compound 35

Compound 37

Current Data Parameters
 NAME Nov10-2001-IK1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011110
 Time 9.51
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 101.6
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.90 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300056 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.017 ppm
 F1 3207.76 Hz
 F2P 0.061 ppm
 F2 24.56 Hz
 PPMCM 0.39777 ppm/cm
 HZCM 159.15942 Hz/cm

Compound 37

Current Data Parameters
NAME Nov09-2001-1k1
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters

Date_ 20011110
Time 8.19
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT DMS
NS 15000
DS 4
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 2.0000000 sec
d11 0.0300000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127509 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.069 ppm
F1 22141.76 Hz
F2P 0.859 ppm
F2 86.42 Hz
PPMCH 10.96051 ppm/cm
HZCM 1102.76709 Hz/cm

Compound 39

Current Data Parameters
 NAME ps8
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031008
 Time 10.52
 INSTRUM drx2500
 PROBED 5 mm BBI 1H-
 PULPROG zg30
 TD 80126
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.100003 Hz
 AQ 4.9999123 sec
 RG 143.7
 DW 62.400 usec
 DE 30.00 usec
 TE 298.0 K
 D1 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.10 usec
 PL1 -1.00 dB
 SFO1 500.1332008 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300085 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 7.922 ppm
 F1 3961.87 Hz
 F2P 0.060 ppm
 F2 29.93 Hz
 PPMCM 0.39309 ppm/cm
 HZCM 196.59709 Hz/cm

Compound 39

Current Data Parameters
NAME ps8
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031009
Time 8.44
INSTRUM drx2500
PROBHD 5 mm BBO BB-
PULPROG zg45.cpd
TD 131072
SOLVENT CDC13
NS 30000
DS 0
SWH 31152.648 Hz
FIDRES 0.237676 Hz
AQ 2.1037557 sec
RG 13004
DM 16.050 usec
DE 30.00 usec
TE 298.0 K
D1 0.30000001 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7715724 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -5.00 dB
PL12 13.50 dB
PL13 15.50 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 131072
SF 125.7577614 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.7789 ppm
F1 27765.99 Hz
F2P 0.083 ppm
F2 10.40 Hz
PPMCM 11.03534 ppm/cm
HZCM 1387.77905 Hz/cm