Supporting information

S1

**Facile Cu(OTf)₂-catalyzed Preparation of Per-*O*-acetylated
Hexopyranoses with Stoichiometric Acetic Anhydride and
Sequential One-Pot Anomeric Substitution to Thioglycosides
under Solvent-free conditions**

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S11

Current Data Parameters
 NAME DCA-169a-b
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030711
 Time 1.39
 INSTRUM spect
 PROBD 5 mm tx12 1h/1
 PULPROG cosyrs
 TD 1024
 SOLVENT CDCl3
 NS 1
 DS 16
 SWH 4990.020 Hz
 FIDRES 4.873066 Hz
 AQ 0.1026548 sec
 RG 4096
 DM 100.200 usec
 DE 15.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 P1 8.10 usec
 SFO1 500.1321005 MHz
 NUC1 1H
 P11 0.00 dB
 D0 0.00000380 sec
 P16 1000.00 usec
 D16 0.00030000 sec
 P0 3.50 usec
 D13 0.00000300 sec
 INO 0.00020040 sec

F1 - Acquisition parameters
 NDO 256
 TD 1
 SFO1 500.1322 MHz
 FIDRES 19.492266 Hz
 SW 9.977 ppm
 FMODE undefined

F2 - Processing parameters
 SI 500.1300129 MHz
 SF 1024
 WDW QSIINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 500.1276329 MHz
 WDW QSIINE
 SSB 0
 LB 0.00 Hz
 GB 0

Current Data Parameters
 NAME DCA-169a-b
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030711
 Time 1.39
 INSTRUM spect
 PROBHD 5 mm tx12 1h/1
 PULPROG cosygs
 TD 1024
 SOLVENT CDCl3
 NS 1
 DS 16
 SMH 4990.020 Hz
 FIDRES 4.873066 Hz
 AQ 0.1026548 sec
 RG 4096
 DM 100.200 usec
 DE 15.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 P1 8.10 usec
 SF01 500.1321005 MHz
 NUC1 1H
 P11 0.00 dB
 D0 0.00000380 sec
 P16 1000.00 usec
 D16 0.00030000 sec
 P0 3.50 usec
 D13 0.00000300 sec
 INO 0.00020040 sec

F1 - Acquisition Parameters
 ND0 1
 TD 256
 SF01 500.1322 MHz
 FIDRES 19.492266 Hz
 SW 9.977 ppm
 FwMODE undefined

F2 - Processing Parameters
 SI 1024
 SF 500.1300129 MHz
 WDW QsINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

F1 - Processing Parameters
 SI 1024
 MC2 QF
 SF 500.1276329 MHz
 WDW QsINE
 SSB 0
 LB 0.00 Hz
 GB 0

Current Data Parameters
 NAME DCA-169a-b
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030711
 Time 2.10
 INSTRUM spect
 PROBHD 5 mm tx12 1h/1
 PULPROG noesygpcp
 TD 2048
 SOLVENT H2O
 NS 8
 DS 32
 SWH 4990.020 Hz
 FIDRES 2.436533 Hz
 AQ 0.2052596 sec
 RG 362
 DW 100.200 usec
 DE 15.50 usec
 TE 300.0 K
 D16 0.00015000 sec
 D8 0.80000001 sec
 P16 1000.00 usec
 d20 0.3988499 sec
 D1 1.50000000 sec
 P1 8.10 usec
 SFO1 500.1321005 MHz
 NUC1 1H
 P11 0.00 dB
 D0 0.0000300 sec
 GPX1 0.00 %
 GPY1 0.00 %
 GPZ1 40.00 %
 GPNAM1
 P2 16.20 usec
 GPX2 0.00 %
 GPY2 0.00 %
 GPZ2 -40.00 %
 GPNAM2
 INO 0.00010020 sec

F1 - Acquisition Parameters
 NDO 2
 TD 384
 SFO1 500.1314 MHz
 FIDRES 12.994843 Hz
 SW 9.977 ppm
 FMODE undefined

F2 - Processing Parameters
 SI 1024
 SF 500.1300129 MHz
 MDM QSIINE
 SSB 3
 LB 0.00 Hz
 GB 0
 PC 0.40

F1 - Processing Parameters
 SI 1024
 MC2 TPP1
 SF 500.1268467 MHz
 WDM QSIINE
 SSB 3
 LB 0.00 Hz
 GB 0

Current Data Parameters
 NAME DCA-169a-b
 EXPNO 11
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20030711
 Time 19.37
 INSTRUM spect
 PROCESSOR 1
 FIDRES 5 mm 1x12 1Hz
 PULPROG zgpg30
 TD 16384
 SFO 500.132463 MHz
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 531.765 Hz
 FIDRES 0.336539 Hz
 AQ 1.484404 sec
 RG 90.5
 DE 90.600 usec
 TE 280.0 K
 P1 3.000000 sec
 P2 3.00 usec
 SFO1 500.132463 MHz
 NUC1 1H
 PL1 -2.00 dB
 F2 - Processing Parameters
 SI 16384
 SF 500.130136 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

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S18

Current Data Parameters
 NAME DCA-159a-b
 EXPNO 11
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20030711
 Time 19.37
 INSTRUM spect
 PULPROG zgpg30
 FIDRES 0.463
 TD 16384
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 5318.744 Hz
 FIDRES 0.4634 Hz
 RG 1.484404 sec
 AG 90.5
 DW 90.600 usec
 DE 15.50 usec
 TE 280.0 K
 T1 3.0000000 sec
 P1 3.0000000 sec
 SFO1 500.132463 MHz
 NUC1 1H
 PL1 -2.00 dB
 F2 - Processing parameters
 SFO2 500.130136 MHz
 SE 1.00
 KW 0
 SSB 0
 LB 0
 GB 0
 PC 1.00

14b
1H-1H COSY
(expanded)

Current Data Parameters
NAME DCA-169a-b
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030711
Time 19.37
INSTRUM spect
PROBHD 5 mm txd2 1h
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 8
DS 2
SWH 5318.76 Hz
AQ 0.336839 Hz
RG 1.484404 sec
DE 90.5
DM 90.600 usec
TE 300.2 K
D1 3.0000000 sec
P1 3.00 usec
SFO1 500.1322463 MHz
NUC1 1H
PL1 -2.00 dB
F2 - Processing parameters
SI 16384
SF 500.1300136 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Current Data Parameters
 NAME DCA-169a-b
 EXPNO 14
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030711
 Time 20.09
 INSTRUM spect
 PROBD 5 mm tx12 1h/1
 PULPROG noesygpcp
 TD 2648
 SOLVENT H2O
 NS 8
 DS 32
 SMH 4990.020 Hz
 FIDRES 2.436533 Hz
 AQ 0.2052596 sec
 RG 406.4
 DE 100.200 usec
 TE 15.50 usec
 D8 0.00015000 sec
 D16 0.80000001 sec
 P16 1000.00 usec
 d20 0.39884999 sec
 D1 1.50000000 sec
 P1 8.10 usec
 SF01 500.1321506 MHz
 NUC1 1H
 PL1 0.00 dB
 D0 0.00000300 sec
 GPX1 0.00 ‡
 GPY1 0.00 ‡
 GPZ1 40.00 ‡
 GPNAM1 sine.100
 P2 16.20 usec
 GPX2 0.00 ‡
 GPY2 0.00 ‡
 GPZ2 -40.00 ‡
 GPNAM2 sine.100
 INO 0.00010020 sec

F1 - Acquisition Parameters
 MD0 2
 TD 384
 SF01 500.1314 MHz
 FIDRES 12.994843 Hz
 SW 9.977 ppm
 FnmODE undefined

F2 - Processing Parameters
 SI 1024
 SF 500.1300136 MHz
 WDW QSINE
 SSB 3
 LB 0.00 Hz
 GB 0
 PC 1.00

F1 - Processing Parameters
 SI 1024
 MC2 TPPI
 SF 500.1267972 MHz
 WDW QSINE
 SSB 3
 LB 0.00 Hz
 GB 0