Supporting Information

Synthesis of the C-Linked Disaccharide α -D-Man-(1 $\rightarrow$ 4)-D-Man Employing a SmI₂-Mediated C-Glycosylation Step: Enroute to Cyclic C-Oligosaccharides

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¹ H NMR spectra for compounds 4 , 5 , 13-16 , 18-20 and ¹³ C NMR spectra for 4 , 5 , 13-16 , 18 , 19 (17 pages)	S2-S18
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S2

Pulse Sequence: s2pul
 Solvent: CDCl₃
 Ambient temperature
 GEMINI-200 "gem200"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.985 sec
 Width 2999.6 Hz
 16 repetitions
 OBSERVE H1, 199.9750206 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 49 sec

S3

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-200 "gem200"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 35.0 degrees
 Acq. time 1.498 sec
 Width 11816.7 Hz
 1216 repetitions
 OBSERVE C13, 50.2837894 MHz
 DECOUPLE H1, 199.9760737 MHz
 Power 30 dB,
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 3 hr, 33 min, 50 sec

13

55

Chemical structure of a bicyclic compound, likely a diazole derivative, showing a diazole ring fused to a bicyclic system. The structure includes a diazole ring (N=N) and a methoxy group (OMe). The compound is labeled with a circled number 14.

Filename: --

SS

LM 4/8 f2

exp4 std13c

SAMPLE DEC. & VT
 date Nov 28 2001 dfrq 199.976
 solvent CDC13 dn H1
 file ACQUISITION exp 0
 sfreq 50.289 dm yvy
 tn 50.289 dm 9900
 at 1.438 lb PROCESSING 1.00
 np 35392 wfile ft
 sw 11816.7 7500 proc 65536
 bs 64 fn
 pw 10.5 werr react
 dl 1.000 wexp procplot
 tof 766.8 wbs
 nt 1024 wnt
 ct 128
 alock n
 gain not used
 FLAGS
 l1 n
 l2 n
 l3 n
 l4 y
 DISPLAY 966.8
 sp 9101.5
 wp 2147
 vs 0
 sc 0
 wc 265
 hzmm 34.35
 ls 500.00
 rf1 251.4
 rf2 0
 rfp 10
 th 100.000
 ins
 ai no ph

S6

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-200, "gem200"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.995 sec
Width 2999.6 Hz
32 repetitions
OBSERVE H1, 199.9750222 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 38 sec

S7

Pulse Sequence: s2pul
 Solvent: CDCl₃
 Ambient temperature
 GEMINI-200 gem200
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 35.0 degrees
 Acq. time 1.498 sec
 Width 11816.7 Hz
 256 repetitions
 OBSERVE C13, 50.2837600 MHz
 DECOUPLE H1, 199.9760737 MHz
 Power 30 dB,
 continuously on
 VOLTAGE modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 65536
 Total time 43 min, 47 sec

58

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: Lise/1m4-31-H_22Feb2002
File: PROTON

Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
Mercury-40008 "Kaj400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6389.8 Hz
16 repetitions
OBSERVE H1, 399.8724709 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 50 sec

89

exp7 std13c
 SAMPLE DEC. & VT
 date Feb 20 2002 dfrq 199.976
 solvent CDC13 dn HI
 file exp 0
 ACQUISITION dm yyy
 sfrq 50.289 dnm 9900 w
 tn C13 dmf
 at 1.498 lb PROCESSING
 np 35392 wtfile 1.00
 sw 11816.7 wfile
 fb 7500 proc ft
 bs 64 fn 85536
 pt 10.5
 dt 1.000 werr react
 tof 766.8 wexp procpilot
 nt 1024 wbs
 ct 128 wnt
 alock not used
 gain
 FLAGS
 ll n
 ln n
 dp y
 DISPLAY
 sp 1013.3
 wp 7051.7
 vs 1687
 sc 0
 wc 265
 hzmm 26.61
 ls 500.00
 rfl 251.4
 rfp 0
 th 127
 ins 100.000
 al no ph

S10

Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 GEMINI-200 "gem200"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.995 sec
 Width 2999.6 Hz
 32 repetitions
 OBSERVE H1, 199.9750246 MHz
 DATA PROCESSING
 FT size 65536
 Total time 1 min, 38 sec

SM

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-200 "gem200"
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 35.0 degrees
 Acq time 1.458 sec
 Width 11816.7 Hz
 640 repetitions
 OBSERVE C13, 50.2837591 MHz
 DECOUPLE H1, 199.9760737 MHz
 Power 30 dB, continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 43 min, 47 sec

S12

Pulse Sequence: s2pu]
 Solvent: CDCl3
 Ambient temperature
 GEMINI-200 "gem200"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.995 sec
 Width 2399.6 Hz
 128 repetitions
 OBSERVE F1 199.9750237 MHz
 DATA PROCESSING
 FT size 65536
 Total time 6 min, 35 sec

8/13

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: jm4-44-7-gem200"
 DEMINI-200 "gem200"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 35.0 degrees
 Acq. time 1.498 sec
 Width 11816.7 Hz
 16960 repetitions
 OBSERVE C13, 50.2837557 MHz
 DECOUPLE H1, 199.9760737 MHz
 Power 30 dB
 Continuously on
 WALTZ16 modulated
 DATA PROCESSING
 FT size 65536
 Total time 14 hr, 15 min, 16 sec

S14

Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory: Lise/1m4-45-400MHz_14Mar2002
 File: PROTON

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 Mercury-40088 "Kaj400"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.398 sec
 Width 6389.8 Hz
 32 repetitions
 OBSERVE H1, 399.8724617 MHz
 DATA PROCESSING
 FT size 65536
 Total time 1 min, 40 sec

SIS

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-200 "gem200"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 35.0 degrees
 Acq. time 1.498 sec
 Width 11816.7 Hz
 20508 repetitions
 OBSERVE C13, 50.2837591 MHz
 DECOUPLE H1, 199.9760737 MHz
 Power 30 dB
 Continuously on
 WALTZ16 modulated
 DATA PROCESSING
 FI size 65536
 Total time 24 hr, 56 min, 41 sec

S16

Archive directory: /export/home/vnari/vnarsys/data
 Sample directory: Lise/LM4-S1-H_02Apr2002
 File: PROTON

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 Mercury-400BB "Kaj400"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.998 sec
 Width 6389.8 Hz
 32 repetitions
 OBSERVE H1 399.8724619 MHz
 DATA PROCESSING
 FT size 65536
 Total time 1 min, 40 sec

817

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-200 "gem200"
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 35.0 degrees
 Acq time 1.498 sec
 Vd: 11816.7 Hz
 107308 repetitions
 OBSERVE C13, 50.2837591 MHz
 DECOUPLE H1, 199.9760737 MHz
 Power 30 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 85 hr, 31 min, 30 sec

S18

Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory: Lise/lm4-55-fraktion1-H_04Apr2002
 File: PROTON

Pulse Sequence: szpu1

Solvent: CDCl₃
 Ambient temperature
 Mercury-400BB "ka3400"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.998 sec
 Width 6389.8 Hz

32 repetitions
 OBSERVE H1, 399.8724503 MHz
 DATA PROCESSING
 FT size 65536
 Total time 1 min, 40 sec