Synthesis of 4-deoxy-L- (and D-) hexoses from chiral non-carbohydrate building blocks

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Experimental S-1

¹H NMR Spectra for compounds

Compound **2a** S-2
Compound **2b** S-3
Compound **4** S-4
Compound **5** S-5
Compound C-1 epimer of **5** S-6
Compound **6** S-7
Compound **7a** S-8
Compound **7b** S-9
Compound **8** S-10
Compound **9** S-11
Compound **10** S-12
Compound **11** S-13

¹³C NMR Spectra for compounds

Compound **2b** S-14
Compound **4** S-15
Compound **5** S-16
Compound **6** S-17
Compound **7a** S-18
Compound **7b** S-19
Compound **8** S-20
Compound **9** S-21
Compound **10** S-22
Compound **11** S-23

¹H-¹H COSY Spectra for compounds

Compound **7a** S-24
Compound **9** S-25

All moisture-sensitive reactions were performed under a nitrogen atmosphere using oven-dried glassware. Solvents were dried over standard drying agents³⁰ and freshly distilled prior to use. Reactions were monitored by TLC (precoated silica gel plate F₂₅₄, Merck). Column chromatography: Merck Kieselgel 60 (70-230 mesh). Melting points are uncorrected and were determined with a capillary apparatus. Optical rotations were measured at 25±2 °C in the stated solvent.

¹H and ¹³C spectra were recorded on NMR spectrometers operating at 200, 300, 400 or 500 MHz and 50, 75 100 or 125 MHz, respectively; solvent CDCl₃.

Wherever necessary, two-dimensional ¹H-¹H COSY experiments were carried out for complete signal assignments. Combustion analyses were performed using CHNS analyzer.

5

Lower R_f compound

C-1 epimer of **5**

Higher R_f compound

6

Lower R_f compound

6

STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

INOVA-500 "nmr500"

Relax. delay 1.000 sec

Acq. time 0.195 sec

Width 5250.0 Hz

2D Width 5250.0 Hz

4 repetitions

512 increments

OBSERVE H1, 499.7062174 MHz

DATA PROCESSING

Sine bell 0.096 sec

F1 DATA PROCESSING

Sine bell 0.096 sec

FT size 2048 x 2048

Total time 42 min, 58 sec

STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy

Solvent: CDC13

Temp. 25.0 C / 298.1 K

File: MAepoxOssidrCO
INOVA-500 "nmr500"

INOVA-500 "nmr500"

Relax. delay 1.000 sec

Acq. time 0.193 sec

Width 5304.7 Hz

2D Width 5304.7 Hz

4 repetitions

512 increments

OBSERVE H1, 499.7062174 MHz

DATA PROCESSING

Sine bell 0.097 sec

F1 DATA PROCESSING

Sine bell 0.048 sec

FT size 2048 x 2048

FT size 2048 x 2048
Total time 42 min, 52 sec