An efficient synthesis of the natural tetrahydrofuran pachastrissamine starting from D-ribo-phytosphingosine

Richard J. B. H. N. van den Berg, Thomas J. Boltje, Carlo P. Verhagen, Remy E. J. N. Litjens, Gijsbert

*A. van der Marel, and Herman S. Overkleeft**

Leiden Institute of Chemistry, Leiden University, P.O. Box 9502, 2300 RA Leiden, The Netherlands

h.s.overkleeft@chem.leidenuniv.nl

Supporting Information

Table of Contents	S-1
General Experimental Details	S-2
¹ H, HH-COSY and ¹³ C NMR spectra of 6	S-3
¹ H, HH-COSY and ¹³ C NMR spectra of 8	S-6
¹ H, HH-COSY and ¹³ C NMR spectra of 4	S-9
¹ H, HH-COSY and ¹³ C NMR spectra of 9	S-12
¹ H, HH-COSY and ¹³ C NMR spectra of 10	S-15
¹ H, HH-COSY and ¹³ C NMR spectra of 13	S-18
¹ H, HH-COSY and ¹³ C NMR spectra of 3	S-21
¹ H, HH-COSY and ¹³ C NMR spectra of 14	S- 24

General Experimental Details: Phytosphingosine [(2S,3S,4R)-2-amino-1,3,4-octadecanetriol] can be obtained for 1800 €/kg from Degussa Cosmoferm B.V., The Netherlands. For more information, contact Degussa Cosmoferm B.V., Wateringseweg 1, P.O. Box 386, 2600 AJ Delft, The Netherlands, Fax: +31(15)2794120, e-mail: info@cosmoferm.com. The ^1H and ^{13}C NMR spectra, ^1H - ^1H COSY experiment and hetero nuclear chemical shift correlations (HMQC) were run on a 300, 400, 500 or a 600 MHz NMR spectrometer. Chemical shifts (δ) are given in ppm relative to tetramethylsilane as internal standard. Coupling constants are given in Hz. Mass spectra were recorded with an electrospray interface apparatus and a Q-TOF apparatus. Optical rotations were measured on an automatic polarimeter. Dichloromethane (DCM) was boiled under reflux over P_2O_5 for 2h and distilled immediately prior to use. Solvents used for flash chromatography and TLC were of technical grade and distilled before use. Flash chromatography was performed on silica gel (0.04 – 0.063 mm). TLC-analysis was conducted on plates with 0.2 mm film thickness. Spots were visualized by UV-absorption (254 nm) were applicable and by spraying with 20% sulfuric acid in ethanol followed by charring at $\sim 150^\circ\text{C}$ or by spraying with a solution of $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot\text{H}_2\text{O}$ (25 g/l) and $(\text{NH}_4)_4\text{Ce}(\text{SO}_4)_4\cdot 2\text{H}_2\text{O}$ (10g/l) in 10% sulfuric acid in ethanol followed by charring at $\sim 150^\circ\text{C}$.

S-15

S-24