SUPPLEMENTARY MATERIAL TO
Diastereoselective addition of alkenylchromium(III) reagents to Garner’s aldehyde. The Nozaki–Hiyama–Kishi coupling approach to sphingosines and ceramides

ZORANA FERJANČIĆ1*, RADOMIR MATOVIĆ2 and FILIP BIHELOVIĆ1**

1Faculty of Chemistry, University of Belgrade, Studentski trg 16, P. O. Box 158, 11000 Belgrade, Serbia and 2ICTM – Center for Chemistry, Njegoševa 12, 11000 Belgrade, Serbia

PHYSICAL AND SPECTRAL DATA FOR COMPOUNDS 10 AND 12–14

(S)-2,2-Dimethyl-3-octanoyloxazolidine-4-carboxaldehyde (10). Yield: 87 %; Colorless oil; IR (film, cm⁻¹): 3399, 2955, 2930, 1826, 1738, 1657, 1409, 1372, 1252, 1216, 1157, 1069; ¹H-NMR (500 MHz, DMSO-d₆, δ / ppm): 9.61 (1H, s), 4.81 (1H, d, J = 6.5 Hz), 4.32 (1H, dd, J = 1.0 and 10.0 Hz), 4.07 (1H, dd, J = 6.5 and 9.5 Hz), 2.21 (1H, dt, J = 7.5 and 16.0 Hz), 1.95 (1H, dt, J = 7.5 and 16.0 Hz), 1.48 (3H, s), 1.45 (3H, s), 1.45–1.41 (2H, m), 1.29–1.21 (8H, m), 0.86 (3H, t, J = 7.0 Hz); ¹³C-NMR (125 MHz, DMSO-d₆, δ / ppm): 200.04 (C), 169.94 (C), 95.41 (C), 65.54 (CH), 63.47 (CH₂), 35.02 (CH₂), 31.64 (CH₂), 28.99 (2×CH₂), 25.80 (CH₃), 24.68 (CH₂), 23.50 (CH₃), 22.51 (CH₂), 14.37 (CH₃); HRMS (ESI-TOF high acc) Calcd. for C₁₄H₂₆NO₃ (MH⁺): 256.1907. Found: 256.1906. [α]D²⁰ (c=0.64, CHCl₃): –38.9.

(S)-Methyl 3-hydroxy-2-octanamidopropanoate (12). Yield: 85 %; Colorless, viscous oil; IR (film, cm⁻¹): 3370, 2956, 2930, 2858, 1751, 1655, 1543, 1463, 1440, 1223, 1084; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 6.55 (1H, d, J = 7.5 Hz), 4.67 (1H, dt, J = 4.0 and 7.5 Hz), 3.98–3.96 (1H, m), 3.91–3.88 (1H, m), 3.79 (3H, s), 3.13 (1H, bs), 2.26 (2H, t, J = 7.5 Hz), 1.67–1.61 (2H, m), 1.32–1.16 (8H, m), 0.88 (3H, t, J = 7.0 Hz); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 173.84 (C), 171.07 (C), 63.35 (CH₂), 54.58 (CH), 52.66 (CH₃), 36.44 (CH₂), 31.61 (CH₂), 29.13 (CH₃), 28.93 (CH₂), 25.51 (CH₂), 22.54 (CH₂), 13.99 (CH₃); HRMS (ESI-TOF high acc): Calcd. for C₁₄H₂₃NO₄Na (MNa⁺): 268.1519. Found: 268.1509; [α]D²⁰ (c=0.48, CHCl₃): +23.9.

(S)-Methyl 2,2-dimethyl-3-octanoyloxazolidine-4-carboxylate (13). Yield: 69 %; Colorless oil; IR (film, cm⁻¹): 2956, 2931, 2857, 1755, 1650, 1461, 1411, 1368, 1256, 1210, 1079, 1056; ¹H-NMR (500 MHz, DMSO-d₆, 60 °C, δ / ppm):
4.77 (1H, br, J = 4.0 Hz), 4.13 (2H, m), 3.73 (3H, s), 2.25 (1H, dt, J = 7.5 and 16.0 Hz), 2.00 (1H, dt, J = 7.0 and 15.5 Hz), 1.55 (3H, s), 1.49 (1H, br, J = 6.5 Hz), 1.46 (3H, s), 1.30-1.25 (8H, m), 0.87 (3H, t, J = 7.0 Hz); 13C-NMR (125 MHz, DMSO-d6, δ / ppm): 171.01 (C), 169.15 (C), 94.89 (C), 66.20 (CH2), 58.60 (CH), 55.12 (CH3), 34.27 (CH2), 30.80 (CH2), 28.20 (CH2), 28.12 (CH2), 24.85 (CH3), 23.79 (CH2), 23.10 (CH3), 13.46 (CH3); HRMS (ESI-TOF high acc) Calcd. for C15H28NO4 (MH+): 286.2013. Found: 286.2004; [α]20

(R)-1-(4-(Hydroxymethyl)-2,2-dimethylazolidin-3-yl)-1-octanone (14). Yield: 75 %; Colorless, viscous oil; IR (film, cm-1): 3420, 2956, 2931, 2873, 1627, 1464, 1423, 1372, 1251, 1209, 1079, 1059; 1H-NMR (500 MHz, DMSO-d6, δ / ppm): 5.05 (1H, dd, J = 5.0 and 6.5 Hz), 3.92-3.84 (3H, m), 3.42-3.39 (1H, m), 2.38-2.23 (1H, m), 1.53-1.47 (2H, m), 1.47 (3H, s), 1.41 (3H, s), 1.30-1.21 (8H, m), 0.86 (3H, t, J = 7.0 Hz); 13C-NMR (125 MHz, DMSO-d6, δ / ppm): 169.31 (C), 93.81 (C), 64.87 (CH2), 61.44 (CH2), 58.50 (CH), 34.35 (CH2), 31.22 (CH2), 28.65 (CH2), 28.60 (CH2), 26.75 (CH3), 24.71 (CH2), 22.74 (CH2), 22.07 (CH2), 13.94 (CH3); HRMS (ESI-TOF high acc) Calcd. for C14H28NO3 (MH+): 258.2064. Found: 258.2053; [α]20

(c=0.6, CHCl3): -9.3. 

Available on line at www.shd.org.rs/JSCS/

(CC) 2014 SCS. All rights reserved.