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(4R,2'R,3'R,5'R)-3-[3,5-Bis(tert-butylidemethylsilyloxy)-1-oxo-2-(phenylmethoxy)hexyl]-4-(phenylmethyl)-2-oxazolidinone

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To a solution of alcohol 1 (225 mg, 0.43 mmol) [1] in dry N,N-dimethylformamide (0.9 ml) at 0°C under an atmosphere of nitrogen was added imidazole (72 mg, 1.1 mmol) and tert-butyldimethylsilyl chloride (67 mg, 0.45 mmol). The resultant solution was allowed to reach room temperature and stirred overnight. The reaction mixture was poured into ether (17 ml), washed with water (3 x 4 ml), brine (4 ml) then dried over sodium sulfate. Removal of the solvent at reduced pressure afforded a pale yellow oil that was purified by flash chromatography using light petroleum-ethyl acetate (4:1) as eluent to give the title compound 2 (246 mg, 89%) as a colourless oil.

[a]D -41.37 (c 0.614, CHCl₃).

IR (cm⁻¹, neat): 2956-2856s, 1789s, 1709s, 1386, 1110.

1H NMR (400 MHz, CDCl₃): 0.01, 0.02, 0.06, 0.09 (12H, s, 2 x SiMe₂), 0.84, 0.88 (18H, s, 2 x Bu'), 1.13 (3H, d, J₆',₅' 6.0 Hz, H₆'), 1.71-1.77 (1H, m, H₄A), 1.87-2.04 (1H, m, H₄B), 2.55 (1H, dd, J₉,₈ 13.4 and J₉ 9.9 Hz, CHC₆₂Ph), 3.15 (1H, dd, J₉,₈ 13.4 and J₉ 3.2 Hz, CHC₆₂Ph), 4.06-4.15 (4H, m, H₅, H₃', H₅'), 4.55-4.60 (1H, d, J₉,₈ 11.5 Hz, OC₆₂Ph), 4.64 (1H, d, J₉,₈ 11.5 Hz, OC₆₂Ph), 5.83 (1H, d, J₂,₃' 6.6 Hz, H₃'), 7.19-7.41 (10H, m, Ph).

13C NMR (100 MHz, CDCl₃): -4.8, -4.7, -4.6, -4.5 (CH₃, 2 x SiMe₂), 17.8, 18.0 (quat., 2 x CMe₃), 24.3 (CH₃, C₆'), 25.7, 25.9 (CH₃, 2 x CMe₃), 37.7 (CH₂, CH₂Ph), 45.5 (CH₂, C₄'), 55.6 (CH, C₄), 65.6 (CH, C₅'), 66.1 (CH₂, C₅), 70.8 (CH, C₃'), 73.2 (CH₂, OCH₂Ph), 80.7 (CH, C₂'), 127.3, 127.9, 128.3, 128.4, 128.9, 129.4 [CH, 2 x Ph (last 4 peaks coincidental)], 135.2 (quat., CHCH₂Ph), 137.5 (quat., OCH₂Ph), 153.0 (quat., C₂), 172.1 (quat., C₁').

Cl-MS (LSIMS, NBA matrix): 642 (MH⁺, 3%), 584 (M-C₄H₁₀, 13), 510 (M-C₆H₁₆O₃Si, 9), 418 (10), 286 (14), 215 (10) and 159 (C₈H₁₉O₃Si, 100).

Anal. calc. for C₃₅H₅₅NO₆Si₂ M⁺ (EI), 641.3568; found M⁺, 641.3561.

References

Sample availability: available from the authors.