6-(N-Benzylmethoxycarbonyl-asparagyl-O-benzylester)-amido-6-deoxy-b-cyclodextrine

Thorsten Graf and Burkhard Koenig*

Department of Organic Chemistry, Faculty of Science IV, University of Regensburg, Universitätsstraße 31, 93051 Regensburg, Germany
Fax (+49) 941 943 171; E-mail: burkhard.koenig@chemie.uni-regensburg.de

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6-Amino-6-deoxy-b-cyclodextrine (1) was prepared according to the literature [1,2] in a three step synthesis. A solution of 1 (400 mg, 0.35 mmol) in 10 ml of abs. DMF was added dropwise to a solution of DCC (81 mg, 0.39 mmol), HOBT (53 mg, 0.39 mmol) and Cbz-Asp-OBzl (2) (126 mg, 0.35 mmol) in 30 ml of abs. DMF under nitrogen atmosphere at 0°C within half an hour. The reaction mixture was stirred at room temp. for 18 h, the solvent was removed at 40°C under reduced pressure. The resulting residual was stirred in 45 ml of acetone for 3 h. The crude product was filtered, washed with acetone and dried under reduced pressure to yield 514 mg (99%) of the title compound (2) as a colorless powder. The crude product was purified by preparative HPLC on a reverse phase column (column: Phenomenex; Luna 10 C18; solvent gradient: water/acetonitrile; from 0% CH3CN to 95%; flow rate: 10.5 ml/min; retention time of product: 8.2 min; detection: UV absorption 214 and 195 nm).

MP: thermal decomposition above 250°C.

MS (+p ESI, DMSO/MeOH + 10 mmol/l NH4OAc): 1382.8 (15%) [M+H - (CH2-Ph)]+1, 1473.7 (100%) [M+H]+, 1495.6 (33%) [M+Na]+.

UV/Vis (MeOH/H2O 1:1) lmax [nm] (lg e): 252.1 (3.551), 257.6 (3.662), 262.9 (3.634), 268.0 (3.536), 281.5 (3.308), 306.9 (3.417).

1H-NMR (600 MHz, DMSO-D6): 2.55 - 2.71 (m, 2H, H2, H2'), 3.25 - 3.75 (m, 42H, 7xCD-H2, 7xCD-H3, 7xCD-H4, 7xCD-H5, 7xCD-H6, 7xCD-H6'), 4.36 - 4.52 (m, 7H, 6xCD-6OH, H1), 4.79 - 4.87 (m, 6H, 6xCD-H1), 5.00 - 5.13 (m, 5H, 1xCD-H1, 2xCH2-Ph), 5.61 - 5.82 (m, 14H, 7xCD-2OH, 7xCD-3OH), 7.30 - 7.39 (m, 10H, Ar-H), 7.52 (d, 3J = 8.1 Hz, 1H, NH), 7.75 (m, 1H, CD-NH).

13C-NMR (150 MHz, DMSO-D6): 36.7 (-, C2, C2', HSQC), 50.7 (+, C1, HSQC), 59.7 (-, CD-C6, HSQC),
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References


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