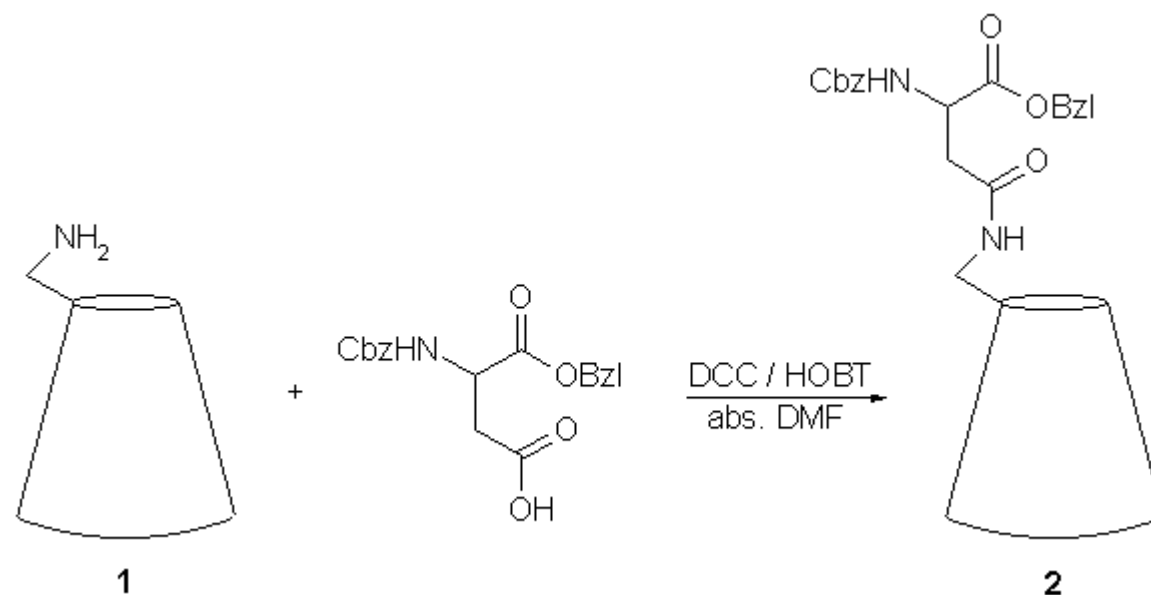


6-(N-Benzylmethoxycarbonyl-asparagyl-O-benzylester)-amido-6-deoxy-b-cyclodextrine**Thorsten Graf and Burkhard Koenig***

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Received: 31 March 2003 / Accepted: 24 June 2003 / Published: 28 June 2003



6-Amino-6-deoxy- β -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)]⁺, 1473.7 (100%) [M+H]⁺, 1495.6 (33%) [M+Na]⁺.

UV/Vis (MeOH/H₂O 1:1) λ_{max} [nm] (lg ϵ): 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),

59.8 (-, CD-C6, HSQC), 59.9 (-, CD-C6, HSQC), 59.9 (-, CD-C6, HSQC), 60.0 (-, CD-C6, HSQC), 60.1 (-, CD-C6, HSQC), 65.5 (-, CH₂-Ar, HSQC), 66.1 (-, CH₂-Ar, HSQC), 69.8 (+, CD-CH), 71.9 (+, CD-CH), 71.9 (+, CD-CH), 72.0 (+, CD-CH), 72.1 (+, CD-CH), 72.2 (+, CD-CH), 72.3 (+, CD-CH), 72.4 (+, CD-CH), 72.5 (+, CD-CH), 72.9 (+, CD-CH), 72.9 (+, CD-CH), 73.0 (+, CD-CH), 73.1 (+, CD-CH), 81.2 (+, CD-CH), 81.3 (+, CD-CH), 81.4 (+, CD-CH), 81.5 (+, CD-CH), 81.6 (+, CD-CH), 81.8 (+, CD-CH), 83.2 (+, CD-CH), 101.7 (+, CD-C1, HSQC), 101.8 (+, CD-C1, HSQC), 102.0 (+, CD-C1, HSQC), 102.1 (+, CD-C1, HSQC), 102.2 (+, CD-C1, HSQC), 127.7 (+, Ar-CH, HSQC), 127.8 (+, Ar-CH, HSQC), 127.9 (+, Ar-CH, HSQC), 128.0 (+, Ar-CH, HSQC), 128.3 (+, Ar-CH, HSQC), 128.4 (+, Ar-CH, HSQC), 135.8 (C_{quart}, Ar-C, HMBC), 136.8 (C_{quart}, Ar-C, HMBC), 155.8 (C_{quart}, C5, HMBC), 169.0 (C_{quart}, C3, HMBC), 171.4 (C_{quart}, C4, HMBC).

Acknowledgment: We thank the Wacker-Chemie GmbH Burghausen, Germany, for their support.

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