



Synthesis of C6^A-to-C6^A and C3^A-to-C3^A diamide linked γ -cyclodextrin dimers

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ABSTRACT

The syntheses of three new diamide-linked γ -cyclodextrin dimers joined by substitution at either a glucopyranose C6^A or C3^A carbon are reported. The syntheses involve the reaction of either C6^A or C3^A amino-substituted γ -cyclodextrin with bis(4-nitrophenyl)succinate to form succinamide linked γ -cyclodextrin dimers or reaction of C6^A azido-substituted γ -cyclodextrin with carbon dioxide to form a urea linked γ -cyclodextrin dimer.

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1. Introduction

Host–guest complexation by native and modified cyclodextrin (CD) hosts often alters the solubility, chemical reactivity and other properties of the ground and excited states of the guest species and is an area of substantial supramolecular chemical interest.^{1,2} Prominent in such studies are linked β CD dimers in which the linker is substituted onto two β CD at either the C6^A or C3^A carbon of a glucopyranose unit.^{3–9} The nature of the linker can significantly influence the stabilities of the host–guest complexes formed by the linked β CD dimer. In some cases their stability constants are substantially greater than those expected statistically on the basis of native β CD complex, which is evidence for cooperativity between the linked β CD in complexing the guest species.^{3–6,9} While guests such as adamantane-1-carboxylate³ and 6-(4-toluidino)naphthalene-2-sulfonate⁶ fit the annuli of β CD and linked β CD dimers closely, the γ CD annulus better accommodates larger guest exemplified by fullerenes¹⁰ and porphyrins.¹¹ In principle, understanding of the host–guest complexation of these and other large guests may be enhanced through studies of their complexation by linked γ CD dimers. Accordingly, we have synthesised two linked γ CD dimers linked through either two C3^A or two C6^A by succinamide and a third γ CD dimer linked through two C6^A by urea (Fig. 1) which to the best of our knowledge are the first reported examples of diamide-linked γ CD dimers.

2. Results and discussion

The modification of native CDs may be achieved through substitution of either a OH², OH³ or OH⁶ (Fig. 1) but their similarity and number (6, 7 and 8 of each in α CD, β CD and γ CD, respectively) renders selective modification challenging.¹² The OH⁶ are the most basic and usually the most nucleophilic, the OH² are the most acidic and the OH³ are sterically the most difficult to access. Substitution of OH⁶ in β CD is usually most readily achieved through 6^A-O-(4-methylbenzenesulfonyl)- β -cyclodextrin,¹³ while substitution of OH² and OH³ is often achieved through 2^A-O-(4-methylbenzenesulfonyl)- β -cyclodextrin.¹⁴ However, using the literature reaction of β CD and 4-toluenesulfonylchloride in aqueous solution¹³ for γ CD results in polysubstitution at C6 probably because of the larger γ CD annulus.¹⁵ Larger arenesulfonyl chlorides have been used to achieve mono C6^A substitution,¹⁶ but the yields are low and the cost of the arenesulfonyl chlorides is greater than that of 4-toluenesulfonylchloride.

A modification of the method of Murakami¹⁴ for the synthesis of 2^A-O-(4-methylbenzenesulfonyl)- β -cyclodextrin was selected as the simplest and most economical simultaneous route to 2^A- and 6^A-O-(4-methylbenzenesulfonyl)- γ -cyclodextrin although the yields are modest. The reaction of γ CD and 4-toluenesulfonylchloride in dimethylformamide (DMF) in the presence of dibutyltin oxide gave a mixture of 2^A- and 6^A-O-(4-methylbenzenesulfonyl)- γ -cyclodextrin (2 γ CDTs and 6 γ CDTs), which were readily separated on a Diaion HP-20 column in 5–10% yields of each.

6^A-Azido-6^A-deoxy- γ -cyclodextrin, (6 γ CDN₃) was prepared as described in the literature,¹⁶ through reaction of 6 γ CDTs with sodium azide in DMF, and 66 γ CD₂ur was prepared in 53% yield by reacting 6 γ CDN₃ in CO₂ saturated DMF in the presence of

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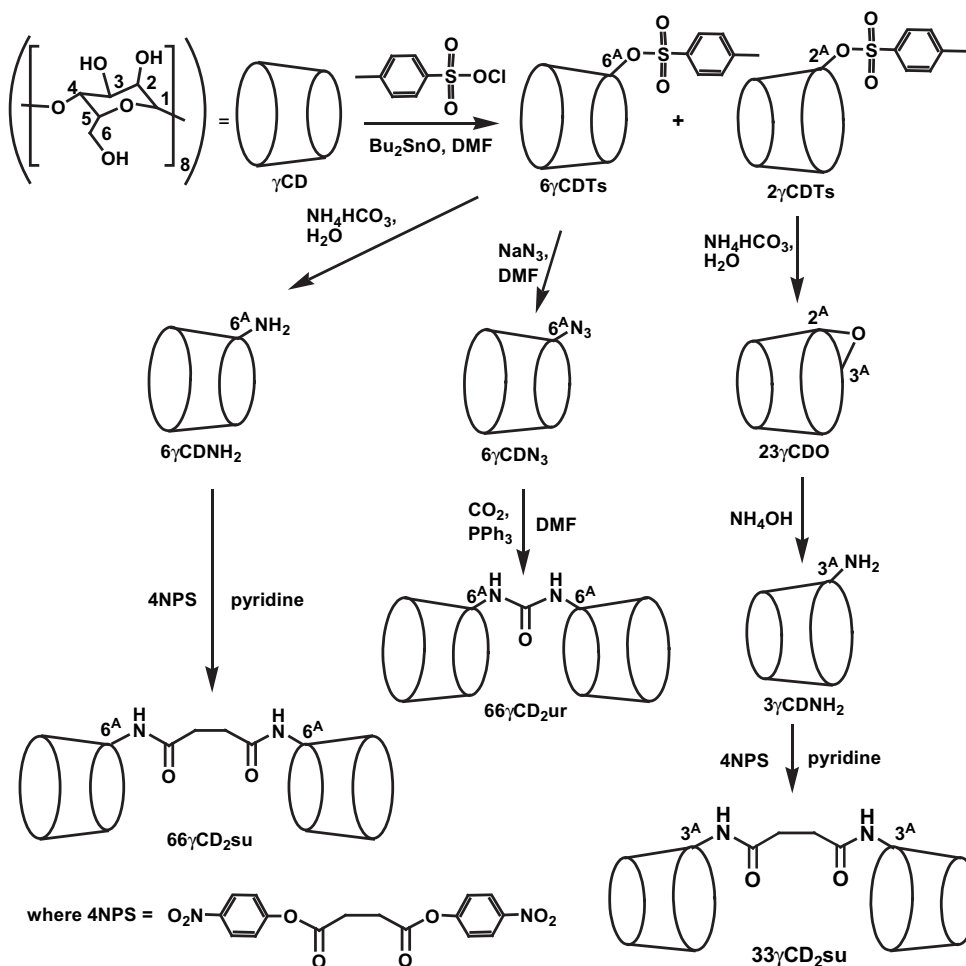


Figure 1. Synthetic routes from γ CD to the diamide-linked γ CD dimers: 66 γ CD₂ur, 33 γ CD₂su and 66 γ CD₂su.

triphenylphosphine. The preparation of 33 γ CD₂ur was not attempted as previous studies show that linking C3^A to C3^A of two CDs through the short urea linker is severely inhibited by steric crowding of the adjacent CDs and the inversion at C3^A in both CDs.⁶

Bis-4-nitrophenyl succinate^{6,17} required in the preparation of 33 γ CD₂su and 66 γ CD₂su was synthesised by reaction of succinyl chloride with 4-nitrophenol in a similar manner to that described in the literature.

The first step in the synthesis of previously unreported (2^A,3^A)-3^A-amino-3^A-deoxy- γ -cyclodextrin (3 γ CDNH₂) was achieved through reaction of 2 γ CDTs with aqueous ammonium bicarbonate to give 2^A,3^A-manno-epoxide- γ -cyclodextrin (23 γ CDO), which was isolated through chromatography on Diaion HP-20 and BioRex 70 (H⁺) in 78.6% yield. The product was then reacted with ammonium hydroxide (25%, 60 cm³) at 60 °C for 4 h to give 3 γ CDNH₂ after chromatographic separation on BioRex 70 (H⁺) in 56.7% yield.¹³ C and ¹H NMR spectra indicate that the C2^A and C3^A carbons of a glycopyranose unit in γ CD moiety are inverted to form an altropyranose ring during the synthesis, as is also the case for 3 β CDNH₂.^{18,19} The new 6^A-amino-6^A-deoxy- γ -cyclodextrin (6 γ CDNH₂) was converted from 6 γ CDTs by treatment with ammonium hydroxide over five days and was isolated through chromatography on BioRex 70 (H⁺) in 18% yield.

The 33 γ CD₂su and 66 γ CD₂su linked γ CD dimers were synthesised through a general method with yields of 75.7 and 91.6%, respectively. The succinate diester was stirred with 2.5 equiv of either 3 γ CDNH₂ or 6 γ CDNH₂ in pyridine for 48 h at room temperature to give 33 γ CD₂su and 66 γ CD₂su, which were separated on BioRex 70 (H⁺).

3. Conclusion

The preparative methods described herein for 33 γ CD₂su, 66 γ CD₂su and 66 γ CD₂ur are both facile and economic such that multigram quantities may be prepared. The preparative methodology for the first two are potentially applicable to a range linked γ -cyclodextrin dimers of varying diamide linker length. The products are chemically stable with no degradation observed in samples stored at –5 to 0 °C for several years. In aqueous solution the diamide linkers are stable over a wide pH range.

In preliminary complexation studies 600 MHz 2D ¹H NOESY NMR spectra of 10^{–3} mol dm^{–3} sodium 6-(4-toluidino)naphthalenesulfonate, NaTNS, and either equimolar 66 γ CD₂ur, 33 γ CD₂su, or 66 γ CD₂su in D₂O at pD 7.0 and 25 °C reveal strong cross-peaks arising from dipolar interactions between the H^{3,5,6} protons of the linked γ CD annuli and the aromatic protons of TNS[–] consistent with complexation of this guest by the diamide-linked γ CD dimers. Quantitative studies of the complexation the substantially larger porphyrins and also of complexation in hydrophobe substituted poly(acrylate)s²⁰ are in progress.

4. Experimental

4.1. General

Proton and ¹³C NMR spectra were recorded with a Varian Gemini ACP-300 spectrometer operating at 300.145 MHz (¹H) or 75.4 MHz (¹³C). 2D ¹H NOESY NMR spectra were recorded with

a Varian-Inova 600 spectrometer operating at 599.957 MHz using a standard pulse sequence with a mixing time of 0.3 s. Thin layer chromatography (TLC) was carried out on Merck Kieselgel 60 F₂₅₄ on aluminium-backed sheets. For analysis of cyclodextrin derivatives, plates were developed with 7:7:5:4 v/v ethyl acetate/propan-2-ol/ammonium hydroxide/water and the compounds were visualised by drying the plate and then dipping it into a 1% sulfuric acid in ethanol solution and heating it with a heat-gun. To visualise amino bearing cyclodextrins plates were dried prior to dipping into 0.5% ninhydrin in ethanol and heated with a heat-gun, before dipping in 1% sulfuric acid in ethanol. For the preparations described below R_c represents the R_f of a substituted cyclodextrin relative to the R_f of the parent cyclodextrin, $R_f(\gamma\text{CD})=0.42$. Melting points were measured using a Kofler hot-stage apparatus viewed through a Reichert microscope and are uncorrected. As cyclodextrin derivatives decompose without melting above 180 °C, melting points were not determined. Elemental analyses were performed by the Microanalytical Service of the Chemistry Department, University of Otago, Dunedin, New Zealand. Mass spectrometry was carried on a Micromass Q-TOF2 instrument. Samples were dissolved in HPLC grade methanol, Milli-Q water or a mixture of the two at a concentration of 0.5 mg cm⁻³.

Bio-Rex 70 resin was purchased from Bio-Rad Laboratories, Inc, CA and converted to the acid form using 3 mol dm⁻³ hydrochloric acid. γ -Cyclodextrin (Nihon Shokuhin Kako Co.), *N,N'*-dicyclohexylcarbodiimide (DCC) (Merck), 4-toluenesulfonylchloride (Aldrich) and 4-nitrophenol (BDH) were used without further purification. All other reagents and solvents were of good quality reagent grade. 6^A-Azido-6^A-deoxy- γ -cyclodextrin¹⁶ and bis-4-nitrophenyl succinate^{6,17} were prepared by literature methods.

4.2. Synthesis

4.2.1. 2^A-O-(4-Methylbenzenesulfonyl)- γ -cyclodextrin, 2 γ CDTs and its 6^A-analogue, 6 γ CDTs. γ -Cyclodextrin (51.9 g, 40.0 mmol) was added to anhydrous DMF (150 cm³) and the mixture was stirred for two hours at 0 °C under dry nitrogen until dissolution was complete. After heating to 100 °C, dibutyltin oxide (25.1 g, 100.9 mmol) was added and stirred for another 2 h. The mixture was then cooled to 0 °C, triethylamine (12.2 g, 120.6 mmol) was added, followed by dropwise addition of 4-toluenesulfonylchloride (20 g, 105 mmol) in DMF (50 cm³). The mixture was stirred for 2 h before another portion of 4-toluenesulfonylchloride (9.7 g, 50.9 mmol) in DMF (20 cm³) was added dropwise. The resultant solution was stirred for a further 10 h at room temperature and then concentrated to a yellow syrup. This was added to 2 dm³ of vigorously stirred acetone and stirring was continued for 30 min. The precipitate formed was collected by filtration, washed with acetone and diethylether and dried under vacuum to give 62 g of crude product, which was recrystallised from ca. 200 ml water. The precipitate was collected and dried under vacuum to give ca. 9 g of crude 6 γ CDTs, while the filtrate was evaporated to dryness to give ca. 47 g of crude 2 γ CDTs.

The crude 2 γ CDTs was dissolved in water (1 dm³) and loaded onto a Diaion HP-20 column (5×30 cm). After flushing with ca. 3 dm³ of water, followed by 10–15% aqueous methanol solvent gradient elution of unreacted γ CD, 2 γ CDTs was eluted with 20–25% aqueous methanol (ca. 400 cm³ fractions). The fractions containing the product were combined, the methanol was removed and the product was dried under vacuum to give the 2 γ CDTs as a white powder. Yield: 4.37 g (7.5%). TLC: $R_c=1.67$. ¹H NMR δ_H (DMSO-*d*₆): 7.83, 7.47 (ABq, $J=8.2$ Hz, 4H, ArH), 5.91–5.69 (m, 15H, OH², OH³), 4.88 (s, 8H, H1), 4.30–3.30 (m, 56H, H2–6, OH⁶), 2.41 (s, 3H, Ar-CH₃). ¹³C NMR, δ_C (DMSO-*d*₆): 133.4, 129.9, 128.2, 125.7 (Ar-C); 101.9–101.1 (C1^{B-H}), 97.3 (C1^A); 82.3–78.2 (C4); 73.1–69.2 (C2, C3, C5); 60.2 (C6); 40.9–38.4 (DMSO); 21.3 (Ar-CH₃).

The crude 6 γ CDTs was dissolved in water (500–700 cm³) and loaded onto a Diaion HP-20 column (3×25 cm). After flushing with water (ca. 1 dm³), followed by 10–20% aqueous methanol solvent gradient elution of unreacted γ CD, 6 γ CDTs was eluted with 30–40% aqueous methanol (ca. 250 cm³ fractions). The fractions containing the product were combined and evaporated to dryness under vacuum to give 6 γ CDTs as a white powder. Yield: 2.47 g (4.25%). TLC: $R_c=1.40$. ¹H NMR, δ_H (DMSO-*d*₆): 7.78, 7.46 (ABq, $J=8.3$ Hz, 4H, ArH), 6.05–5.35 (m, 16H, OH², OH³), 5.09–4.81 (m, 8H, H1), 4.31–3.20 (m, 55H, H2–6, OH⁶), 2.42 (s, 3H, Ar-CH₃). ¹³C NMR, δ_C (DMSO-*d*₆): 133.2, 130.7, 128.8, 126.2 (Ar-C); 102.9–101.8 (C1^A); 81.6–80.8 (C4); 73.6–69.7 (C2, C3, C5); 60.7 (C6); 40.9–38.4 (DMSO); 21.8 (Ar-CH₃).

4.2.2. 2^A,3^A-Manno-epoxide- γ -cyclodextrin, 23 γ CDO. A solution of 2^A-O-(4-methylbenzenesulfonyl)- γ -cyclodextrin (4 g, 2.76 mmol) in aqueous ammonium bicarbonate (10%, 125 cm³) was stirred at 60 °C for 3 h. The solvent was removed under vacuum and the residue was redissolved in water, followed by evaporation to dryness (this procedure was repeated three times). This crude product was dissolved in water (20 cm³) and added drop-wise to vigorously stirred acetone (500 cm³). The precipitate formed was collected by filtration and washed with acetone and diethylether to give 4 g of crude product. The crude material was dissolved in water (125 cm³) and loaded onto a Diaion HP-20 column (3×20 cm). The column was washed with water (1 dm³) and 10% aqueous methanol and the washings were evaporated under vacuum to give the product as a white powder, which contained traces of 2 γ CDNH₂ by-product. The pure 2^A,3^A-manno-epoxide was run through a column (4.5×4.5 cm) of BioRex 70 (H⁺), 100–200 mesh (BioRad) to remove 2 γ CDNH₂. The fractions containing the product were combined and evaporated to dryness under vacuum to give as a white powder. Yield: 2.77 g (78.6%). TLC: $R_c=1.15$. ¹H NMR, δ_H (D₂O): 5.27 (s, 1H, H1^A-epoxide), 5.13–5.07 (m, 7H, H1), 3.95–3.59 (m, 47H, H2–6), 3.49 (d, 1H, H2^A-epoxide). ¹³C NMR, δ_C (D₂O): 104.5–103.7 (C1), 83.7–83.0 (C4), 75.7–72.1 (C2^{B-H}, C3^{B-H}, C5), 63.6 (C6^{B-H}), 62.9 (C6^A), 57.1 (C2^A), 52.2 (C3^A).

4.2.3. 3^A-Amino-3^A-deoxy-(2^AS,3^AS)- γ -cyclodextrin, 3 γ CDNH₂. 2^A,3^A-Mannoepoxide- γ -cyclodextrin (2.6 g, 2.03 mmol) was dissolved in aqueous ammonium hydroxide (25%, 60 cm³) and the solution was stirred at 60 °C for 4 h. The mixture was then evaporated to dryness and the residual was dissolved in aqueous ammonium hydroxide (28%, 20 cm³) and added to acetone (500 cm³). The precipitate was collected, washed with acetone and diethylether and dried under vacuum to obtain 2.74 g of the crude product. This was dissolved in water (20 cm³) and loaded onto a column (4.5×4.5 cm) of BioRex 70 (H⁺), 100–200 mesh (BioRad). After flushing with water (ca. 500 cm³), the 3 γ CDNH₂ product was eluted with 1 mol dm⁻³ aqueous ammonium hydroxide (ca. 100 cm³ fractions). Fractions containing the product were combined and evaporated to dryness under vacuum (removal of excess ammonia was achieved by dissolving the residue in water and evaporating to dryness three times) to afford 3 γ CDNH₂ as a white powder. Yield: 1.49 g (56.7%). TLC: $R_c=0.76$. ¹H NMR, δ_H (D₂O): 5.22 (d, 2H, H1^A), 5.16–4.93 (m, 6H, H1), 4.20 (m, 1H, H2^A), 4.00–3.56 (m, 46H, H2–6), 3.10 (d, 1H, H3^A). ¹³C NMR, δ_C (D₂O): 103.1–99.7 (C1), 80.9–80.2 (C4), 79.2–71.1 (C2, C3^{B-H}, C5), 60.8–59.8 (C6), 52.2 (C3^A).

4.2.4. 6^A-Amino-6^A-deoxy-cyclodextrin, 6 γ CDNH₂. 6^A-O-(4-Methylbenzenesulfonyl)- γ -cyclodextrin, 6 γ CDTs (3.4 g, 2.3 mmol) was dissolved in ammonium hydroxide (28%, 250 cm³) at 0 °C. The reaction vessel was closed and left in the dark with occasional stirring for five days. The ammonium hydroxide was removed under reduced pressure, after which water was added (100 cm³) and removed under reduced pressure. The remaining solid was dissolved in ammonium hydroxide (28%, 20 cm³) and the solution added

drop-wise to vigorously stirring acetone (450 cm³) and stirred for 30 min. The resulting precipitate was dried under vacuum to give crude 6 γ CDNH₂ as a cream powder. This was dissolved in water (20 cm³) and loaded onto a BioRex 70 (H⁺) column. The column was washed with water (400 cm³), and the title compound was eluted with 0.05–0.1 mol dm⁻³ aqueous ammonium carbonate (4 $\times$ 100 cm³ fractions). The 6 γ CDNH₂ containing fractions were evaporated to dryness under reduced pressure, and the residue freeze dried to give 6 γ CDNH₂ as a white solid. Yield: 0.55 g (18%). TLC: R_f=0.90. ¹H NMR, δ (D₂O) 5.37 (s, 2H, H1^A), 5.10 (m, 14H, H1^{B-H}), 3.93–3.58 (m, 48H, H2–6). ¹³C NMR, δ (D₂O): 103.72–101.77 (C1); 80.50–79.0 (C4); 73.89–71.88 (C2, C3, C5); 60.33 (C6); 41.05 (C6^A).

4.2.5. *N,N'*-Bis(6^A-deoxy- γ -cyclodextrin-6^A-yl)urea, 66 γ CD₂ur. 6^A-Azido-6^A-deoxy- γ -cyclodextrin (1 g, 0.75 mmol) was dissolved in DMF (20 cm³) and the solution was saturated with dry CO₂ and stirred at room temperature for 15 min. Triphenylphosphine (297 mg, 1.134 mmol) in DMF (5 cm³) was added dropwise and the reaction mixture was saturated with CO₂ and stirred for 27 h after which it was added drop-wise to vigorously stirred acetone. The resulting precipitate was collected by centrifugation, washed acetone and diethylether, dried under vacuum and freeze dried. Yield: 520 mg (53%). TLC: R_f=0.40. ¹H NMR, δ _H(D₂O): 5.09–5.07 (m, 16H, H1); 3.9–3.3 (m, 96H, H²-H⁶). ¹³C NMR: δ _C(D₂O) 163.1 (amide C=O), 104.5 (C1), 85.5–83.0 (C4), 76.4–73 (C2, C3, C5), 63.0 (C6^{B-H}), 43.2 (C6^A). Mass spectrum *m/z*: 1332.8 (M+Na)²⁺. Elemental analysis: C₉₇H₁₆₀N₂O₇₉·21H₂O: C, 38.8; H, 6.7; N, 1.0. Found: C, 38.6; H, 6.4; N, 1.2.

4.3. General procedure for the preparation of the succinamide linked γ CD dimers

Either (2^AS,3^AS)-3^A-amino-3^A-deoxy- γ -cyclodextrin or 6^A-amino-6^A-deoxy- γ -cyclodextrin (~1 mmol) was dissolved in pyridine (20 cm³) and stirred at room temperature for 15 min. Bis-4-nitrophenyl succinate (0.4 equiv) was added to this solution in two or more portions over a period of 1 h. The reaction mixture was then stirred for 48 h at room temperature before being added dropwise to diethylether (200 cm³) with vigorous stirring. The resultant precipitate was collected by centrifugation, washed with acetone and diethylether and dried under vacuum. The product was dissolved in H₂O and run down a BioRex 70 (H⁺) column to remove either excess (2^AS,3^AS)-3^A-amino-3^A-deoxy- γ -cyclodextrin or 6^A-amino-6^A-deoxy- γ -cyclodextrin. The white solid products were obtained by freeze drying followed by further drying over phosphorous pentoxide.

4.3.1. *N,N'*-Bis((2^AS,3^AS)-3^A-deoxy- γ -cyclodextrin-3^A-yl) succinamide, 33 γ CD₂su. The title compound was prepared by treatment of the 3 γ CDNH₂ (1.39 g, 1.08 mmol) with bis(4-nitrophenyl) succinate (155 mg, 0.43 mmol). After the general work-up and purification procedure, the title compound was obtained as a white solid. Yield: 0.87 g (75.7%). TLC: R_f=0.60. ¹H NMR, δ _H(D₂O): 5.39–4.94 (m, 16H, H1), 4.26–3.59 (m, 96H, H2–6), 2.6 (s, 4H, CH₂). ¹³C NMR, δ _C(D₂O): 177.7 (C=O), 105.8, 104.2, 102.3 (C1), 81.9, 82.7, 83.1 (C4), 72.3, 73.9,

74.4, 74.9, 74.9, 75.2, 75.6 (C2,3,5), 62.4, 62.9 (C6), 53.6 (C3^A), 33.6 (CH₂). Mass spectrum *m/z*: 1359.9 (M+Na)²⁺. Elemental analysis: C₁₀₀H₁₆₄N₂O₈₀·19H₂O: C, 39.81; H, 6.75; N, 0.93. Found: C, 39.54; H, 6.39; N, 0.89.

4.3.2. *N,N'*-Bis(6^A-deoxy- γ -cyclodextrin-6^A-yl) succinamide, 66 γ CD₂su. The title compound was prepared by treatment of the 6 γ CDNH₂ (1.58 g, 1.21 mmol) with bis(4-nitrophenyl) succinate (176 mg, 0.49 mmol). After the general work-up and purification procedure, the title compound was obtained as a white solid. Yield: 1.2 g (91.6%). TLC: R_f=0.45. ¹H NMR, δ _H(D₂O): 5.11 (m, 16H, H1), 3.42–3.93 (m, 96H, H2–H6), 2.59 (m, 4H, CH₂). ¹³C NMR, δ _C(D₂O): 177.5 (C=O), 104.3 (C1), 85.7, 83.1 (C4), 75.6, 75.0, 74.4, 72.8 (C2,3,5), 62.9 (C6^{B-H}), 42.8 (C6^A), 33.7 (CH₂). Mass spectrum *m/z*: 1359.9 (M+Na)²⁺. Elemental analysis: C₁₀₀H₁₆₄N₂O₈₀·20H₂O: C, 39.58; H, 6.78; N, 0.92. Found: C, 39.48; H, 6.40; N, 0.91.

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Supplementary data

Supplementary 1D ¹H and ¹³C NMR and 2D ¹H NOESY NMR spectra may be found, in the online version, at [doi:10.1016/j.tet.2010.02.005](https://doi.org/10.1016/j.tet.2010.02.005).

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