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Note

Unexpected reaction of β-cyclodextrin tosylate with pyrrolidinones

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Abstract—Substitution reactions of 6^I-*O*-*p*-tolylsulfonylcyclomaltoheptaose with alkyl- and arylamines in 1-methyl-2-pyrrolidinone and various pyrrolidinones were investigated. An unexpected reaction of the tosyl group with pyrrolidinones was observed resulting in products deriving from nucleophilic attack by the lactam carbonyl oxygen and further opening of the heterocyclic ring. The new compounds have been fully characterized by ESIMS and NMR analyses.

Keywords: Monofunctionalized cyclodextrin derivatives; Primary tosylates; 1-Methyl-2-pyrrolidinone; ESIMS; NMR

The preparation of monofunctionalized cyclodextrin derivatives at the primary hydroxyls rim position has been extensively studied with the aim to obtain, among others, derivatives useful as biomimetic receptors and catalysts.[1] The most commonly used method[1a,2] for the selective introduction of a functional group at the primary hydroxyls rim involves activation of one hydroxyl group by sulfonylation. In this context our study was devoted to the synthesis of monofunctionalized cyclomaltoheptaose (β-cyclodextrin, βCD) derivatives bearing amino functionalities at the primary hydroxyls rim, starting from 6^I-*O*-*p*-tolylsulfonyl-cyclomaltoheptaose (**1**).[3]

A common strategy[4] involves S_N2 reactions with an amine in a polar solvent such as *N*,*N*-dimethylformamide (DMF) or 1-methyl-2-pyrrolidinone (NMP). By exploiting this methodology, we found some unexpected results regarding the reactivity of **1** with NMP which are discussed here.

Some authors[4a] reported the superiority of NMP over DMF for nucleophilic substitution of *p*-toluenesulfonates because of the possible contamination with N-formylated materials formed by transacylation between primary amino groups and DMF. Despite of this consideration, 6^I-(*o*-biphenylamino)-6^I-deoxycyclomaltoheptaose and 6^I-(1-naphthylamino)-6^I-deoxycyclomaltoheptaose, derivatives so far not reported, were obtained as the only reaction products using DMF as solvent. On the contrary, by performing their synthesis in NMP, an unexpected product, identified as **2**, was isolated along with the desired compounds after recrystallization from water (Scheme 1).

The treatment of **1** with NMP as the only reagent gave **2** in 61% yield, therefore proving that this compound arises from direct interaction of tosylate with the solvent and that its formation is not influenced by the presence of the amine.

The possible catalytic influence of cyclodextrin on lactam opening was excluded since only a stable complex, βCD/NMP (detected by ESIMS analysis), was observed when βCD and NMP were treated in the experimental conditions of substitution on **1**, with or without *p*-toluenesulfonic acid as initiator. The reaction between NMP and other tosylates confirmed this result. Thus, after the reaction of NMP with benzyl tosylate the formation of benzyl *N*-methyl-4-ammoniumbutanoate *p*-toluenesulfonate (**5**) was observed, proving the general characteristic of this interaction (Scheme 2).

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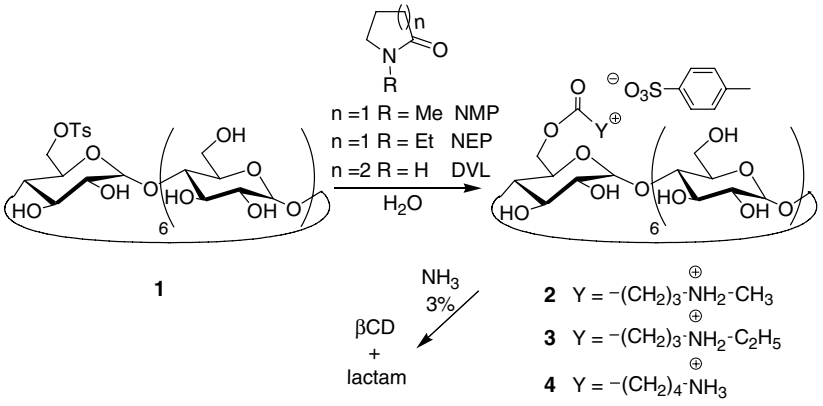
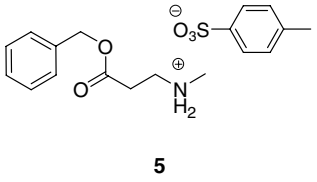


Scheme 1.



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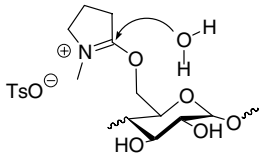
Scheme 2.

Good results were observed also with different pyrrolidinones. Thus by reacting **1** with 1-ethyl-2-pyrrolidinone (NEP) and δ-valerolactam (DVL), cyclodextrin derivatives **3** and **4** were isolated in 89% and 68% yields, respectively (Scheme 1). It is worth to note that the absence of nitrogen substituent decreases the reaction time (from 4 to 2 days).

Structural analysis by ESIMS and advanced NMR techniques showed that these compounds originate from pyrrolidinone ring opening involving a possible concerted mechanism, that implies the formation of the intermediate reported in Scheme 3, the nucleophilic attack of water being assisted from the tosylate moiety.

ROESY and DOSY measurements allowed to gain some insight about the interaction between cyclodextrin tosylate and NMP.

Selective 1D ROESY analysis of the reaction mixture revealed weak dipolar interaction between the methylic protons of pyrrolidinone and those of the tosyl group in **1**. From DOSY measurements, pure NMP showed a diffusion coefficient of 6.1×10^{-10} m^2/s which lowered to 5.6×10^{-10} m^2/s in the presence of **1**. These results suggest the formation of a transient NMP/**1** complex in which the lactam carbonyl is close enough to react with the tosylate, according to Scheme 3.



Scheme 3.

NMP opening before reaction with cyclodextrin tosylate has to be excluded since in the presence of β-cyclodextrin, pyrrolidinone hydrolysis, that generally requires drastic conditions,[5] was not observed. Moreover, the generation of an immonium ion in the reaction between primary chlorides and NMP has already been reported in the literature.[6]

Compound **1** can adsorb above 10% of water upon storage, which may explain the origin of water in the reaction mixture despite the use of dried materials.

Compounds **2**–**4** easily undergo alkaline (NH_3 3%, room temperature, few min) or neutral (water, room temperature, 1 week) hydrolysis to give βCD and the corresponding lactam (Scheme 1).

Structural characterization of all compounds was performed by ESIMS and NMR spectroscopy. The complete structural elucidation of compound **2** was obtained by combined 1D and 2D homo- and heteronuclear NMR (600 MHz, Me_2SO-d_6, 25 °C) measurements.

In comparative analysis of the homo- and heteronuclear scalar correlations in the gCOSY and gHSQC maps, the clusters due to the secondary and primary hydroxyls were recognized at 5.88–5.56 and 4.64–4.34 ppm (Fig. 1a), respectively, the ^{1}H–^{1}H and ^{1}H–^{13}C scalar correlations allowing the assignment of all ring protons.

The assignment of the proton signals of the monoderivatized unit was accomplished starting from the three well-resolved resonances at 4.29, 4.12 and 3.84 ppm. Of these, the doublet at 4.29 ppm and the double doublet at 4.12 ppm was assigned to the diastereotopic methylene protons H-6 as both of them correlate with the same carbon in the gHSQC map. They are also *J*-coupled to the double doublet at 3.84 ppm (H-5).

The 1D TOCSY spectrum (Fig. 1b) obtained by excitation of H-5 with a long mixing time (90 ms) allowed to extract the subspectrum corresponding to the separate

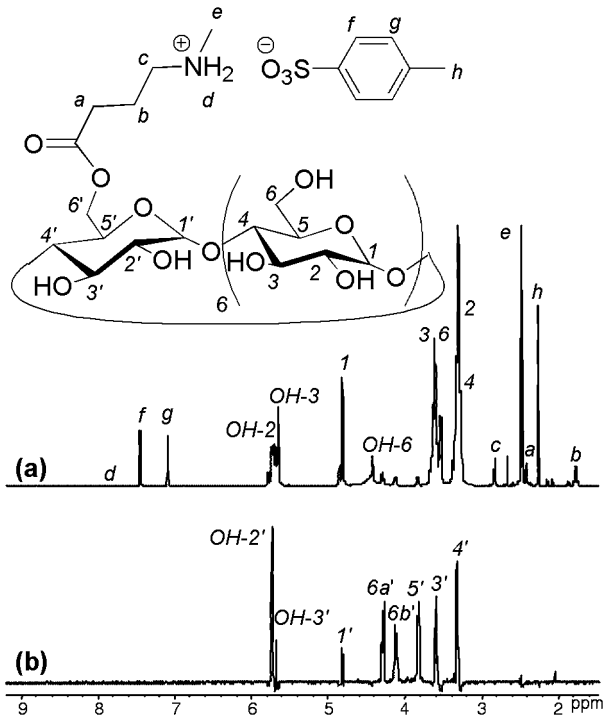


Figure 1. (a) ^{1}HNMR (600 MHz, Me_2SO-d_6, 25 °C) spectrum of **2**. (b) 1D TOCSY spectrum (600 MHz, Me_2SO-d_6, 25 °C, mix 90 ms) by excitation of the H-5′ resonance of **2**.

spin system of the 6^{I}-O-substituted glucopyranose ring allowing the complete attribution also of the resonances overlapped by the more intense signals of the underivatized units.

The ^{1}H nuclei of the tosylate and 4-(*N*-methylammonium)butanoyl moieties generate well-separated resonances which are easily attributed as reported in Section 1. The assignment of the methylene carbons and of the aromatic protons was performed on the basis of the dipolar interactions generated in the ROESY map from the methyl protons bound to the nitrogen and the aromatic ring, respectively. No dipolar interactions between the protons of the aromatic ring and the internal H-3 and H-5 protons of **2** were detected suggesting that no inclusion of the tosylate moiety into the cavity of the macrocycle occurred.

The presence of a carbonyl directly bound to O-6 moiety is clearly demonstrated by measuring in the gHMBC map the long range ^{1}H–^{13}C correlation between H-6 protons and the carbonyl resonance at 172 ppm (Fig. 2).

In conclusion, the use of NMP as solvent in S_N2 reactions revealed a particular, so far unknown reactivity, of **1** towards lactams. When weak nucleophiles are used, a parallel reaction between tosylate and NMP significantly competes with the conventional bimolecular substitution to lead to the unexpected product **2** deriving from pyrrolidone ring opening. The presence of traces of water in the reaction medium probably assists the formation of **2** (Scheme 3).

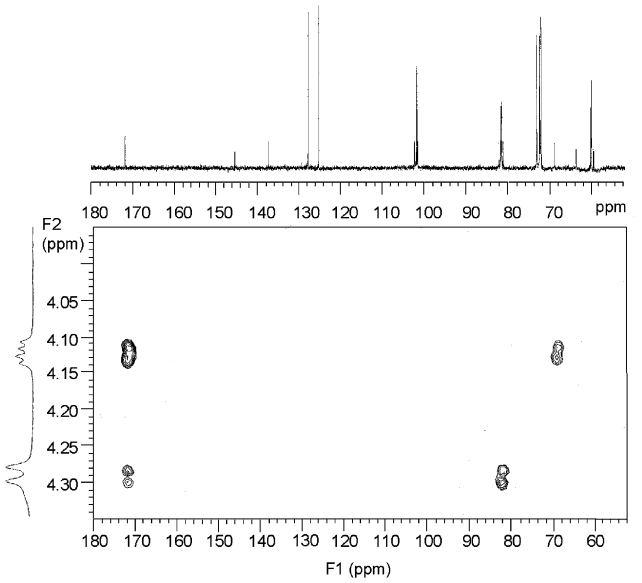


Figure 2. gHMBC spectrum (600 MHz, Me_2SO-d_6, 25 °C) of **2**: spectral region corresponding to the diastereotopic methylene protons H-6′.

If the use of pyrrolidinones as solvent in the nucleophilic displacement on **1** is limited by this parasite reaction, such a reaction might become of interest for the preparation of new cyclodextrin derivatives bearing polar substituents.

1. Experimental

1.1. Materials and methods

All chemicals were purified prior to use by standard methods.[7] β-Cyclodextrin (Fluka.) was dried (12 h) at 110 °C/0.1 mm Hg, in the presence of P_2O_5.

NMR measurements were performed on Varian VXR-300 and INOVA 600 spectrometers operating at 300 and 600 and 75 and 150 MHz for ^{1}H and ^{13}C, respectively. The temperature was controlled to ±0.1 °C. ^{1}H NMR chemical shifts are referenced to TMS as external standard. The 2D NMR spectra were obtained by using standard sequences. Proton 1D TOCSY and proton 1D ROESY spectra were recorded using selective pulses. The selective 1D TOCSY spectra were acquired with 256 scans in 32K data points with a 2 s relaxation delay and a mixing time ranging from 20 to 120 ms. The selective 1D ROESY spectra were acquired with 3520 scans in 32 K data points with a 10 s relaxation delay and a mixing time of 0.9 s. DOSY experiments were carried out by using a stimulated echo sequence with self-compensating gradient schemes, a spectral width of 5660 Hz and 32K data points. Typically, a value of 200 ms was used for *D*, 1.0 ms for *d*,

and g was varied in 20 steps (four transients each) to obtain an approximately 90–95% decrease in the resonance intensity at the largest gradient amplitudes.

HRMS analyses were performed on a Bruker Daltonics MicroTOF equipped with an ESI source by infusion of cyclodextrin in 1:1 MeOH–5 mM aq ammonium acetate solns.

Electrospray mass spectra were acquired using a Perkin–Elmer Sciex API III$^+$ triple quadrupole mass spectrometer (Sciex, Concord, Ont., Canada) equipped with a source for atmospheric pressure ionization and an articulated ionspray interface. The analysis was carried out by flow injection using 1:1 MeOH–5 mM aq ammonium acetate as mobile phase at 50 μL/min. The ESI spectra were obtained under the following conditions: ionspray voltage, 5.5 kV; orifice voltage, 60 V if not otherwise stated; scan range, m/z 500–1500; scan time, 4.73 s; no interscan delay; unit-mass resolution. ESI product ions were produced by collision-induced dissociation (CID) under the following experimental conditions: collision energy, 30 eV if not otherwise stated; collision gas thickness (CGT), 250×10^{13} mol/cm^2; scan range was variable, depending on the m/z value of the selected precursor ion.

LC–MS analysis was performed on a Column Pecosphere, C18, 3 μm 30 × 4.6 mm, at a flow rate of 1 mL/min under the following linear gradient (A = MeCN, B = water): isocratic A = 50% (20 min).

TLC was carried out on silica gel plates (Macherey-Nagel Alugram Sil G/UV$_{254}$ 0.2 mm) using the following eluents: (A) 2:2:1 *i*-PrOH–AcOEt–water; (B) 3:1 MeOH–NH_3. Compounds were visualized by spraying a soln of *p*-anisaldehyde (2.5 mL) in EtOH 95% (93 mL), AcOH (1 mL) and H_2SO_4 95–97% (3.4 mL) or by examination under UV light.

Purification of 6^I-*O*-*p*-tolylsulfonyl-β-cyclodextrin (**1**)[3a] (aliquots of 400 mg) was accomplished by reverse-phase chromatography using solid phase extraction (SPE) cartridges SPE-C_{18} Supelco-ENVI, 12 mL, 2 g (eluent: 50 mL MeOH 10% in water, 250 mL MeOH 28% in water, 60 mL MeOH).

Melting points were determined using a Kofler hot-stage apparatus and are uncorrected.

1.2. General procedure for the reaction of 1 with lactams

Tosylate **1** (50 mg, 0.039 mmol) was dissolved in dry NMP (1.5 mL) and the soln was stirred at 90 °C for 48–96 h under N_2. The mixture was then cooled to room temperature and poured into acetone (25 mL). The precipitate was filtered off and the solid was dried (115 °C/0.005 mmHg, P_2O_5, 6 h) to give the product as crystalline solid.

1.2.1. 6^I-*O*-(*N*-Methyl-4-aminobutanoyl)cyclomaltoheptaose *p*-toluenesulfonate (2). (33 mg, 61%); mp 225–230 °C (slow decomposition); HPLC: t_R 4.09 min; TLC (eluent B): R_f 0.18; ^{1}H NMR (600 MHz, Me_2SO-d_6): δ 1.79 (q, 2H, J 7.2 Hz, –CH_2–), 2.28 (s, 3H, CH_3^{ar}), 2.44 (t, 2H, J 7.2 Hz, –OCOCH_2–), 2.51 (s, 3H, CH_3^{am}), 2.86 (t, 2H, J 7.2 Hz, –CH_2NH$_2$), 3.24–3.50 (m overlap to water signal, H-6), 3.50–3.70 (m, 28H, H-2–5), 3.84 (br t, 1H, H-6′), 4.13 (br t, 1H, H-6′), 4.29 (br d, J 11.1 Hz 1H, OH-6), 4.48 (t, J 6.3 Hz, 5H, OH-6), 4.81–4.91 (m, 7H, H-1), 5.66–5.87 (m, 14H, OH-2, OH-3), 7.1 (d, J 7.8 Hz, 2H, H–C_{ar}-o), 7.48 (t, J 7.8 Hz, 2H, H–C_{ar}-m); ^{13}C NMR (150 MHz, Me_2SO-d_6): 21.5 (CH_3^{ar}), 21.7 (–CH$_2$–), 30.8 (–OCO-CH$_2$–), 33.4 (CH_3^{am}), 48.4 (–CH$_2$NH$_2$), 60.2, 60.6 (C-6), 72.7, 73.0, 73.1, 73.7 (C-2, C-3, C-5), 81.8, 82.2, 82.3, 82.5, 82.9 (C-4) 102.4, 102.5, 102.6, 103.4 (C-1), 126.2 (C_{ar}-o), 128.7 (C_{ar}-m), 138.3 (C_{ar}-q), 146.4 (C_{ar}-q), 172.7 (*C*O); ESIMS: m/z 1234 [M+H$^+$]; MS/MS (m/z 1234): 1234 (100%), 1072 (5.2%), 910 (6%), 748 (8%), 586 (14%), 424 (20.2%), 262 (10.4%), 100 (5.2%); HRESIMS: Calcd for $C_{47}H_{79}NO_{36}$+H$^+$ 1234.4455, found m/z 1234.4408.

1.2.2. 6^I-*O*-(5-Aminovaleroyl)cyclomaltoheptaose *p*-toluenesulfonate (3). (37 mg, 68%); mp 232 °C (slow decomposition); HPLC: t_R 3.26 min; TLC (eluent A): R_f 0.11; ^{1}H NMR (300 MHz, Me_2SO-d_6): δ 1.63 (m, 4H, –CH_2CH_2–), 2.10 (t, J 6.6 Hz, 2H, –OCOCH_2–), 2.28 (s, 3H, CH_3), 3.10 (m, 2H, –C$H_2NH_3^+$), 3.20–3.50 (m overlap to water signal, H-6), 3.50–3.65 (m, 28H, H-2-5), 4.46 (t, J 5.4 Hz, 6H, OH-6), 4.83 (br s, 7H, H-1), 5.60–5.75 (m, 14H, OH-2, OH-3), 7.16 (d, J 7.8 Hz, 2H, H–C_{ar}-o), 7.38 (br s, 3H, NH_3^+), 7.47 (d, J 7.8 Hz, 2H, H–C_{ar}-m); ^{13}C NMR (75 MHz, Me_2SO-d_6): 21.4 (CH_3^{ar}), 22.7 (–OCOCH$_2$CH_2–), 32.1 (–OCO*C*H$_2$–), 41.9 (–*C*H$_2NH_3^+$), 60.7 (C-6), 72.7, 73.1, 73.7 (C-2, C-3, C-5), 82.3 (C-4), 102.7 (C-1), 126.2 (C_{ar}-o), 128.7 (C_{ar}-m), 138.2 (C_{ar}-q), 146.4 (C_{ar}-q), 170.8 (*C*O); ESIMS: m/z 1234 [M+H$^+$]; MS/MS (m/z 1234, OR = 100 V): 1234 (100%), 1072 (4%), 910 (5.4%), 748 (6.1%), 586 (10.1%), 424 (13.7%), 325 (23.8%), 262 (9%), 163 (6.5%); HRESIMS: Calcd for $C_{47}H_{79}NO_{36}$+H$^+$ 1234.4455, found m/z 1234.4399.

1.2.3. 6^I-*O*-(*N*-Ethyl-4-aminobutanoyl)cyclomaltoheptaose *p*-toluenesulfonate (4). (44 mg, 80%); mp 204–207 °C (slow decomposition); HPLC: t_R 4.75 min; TLC (eluent A): R_f 0.10; ^{1}H NMR (300 MHz, Me_2SO-d_6): δ 1.15 (t, 3H, J 7.2 Hz, CH_3^{Et}), 1.80 (q, J 7.8 Hz, 2H, –CH_2–), 2.28 (s, 3H, CH_3^{ar}), 2.46 (t overlap to Me_2SO signal, J 7.8 Hz, 2H, –OCOCH_2–), 2.91 (m, 4H, –C$H_2NH_2^+$CH_2CH$_3$), 3.24–3.42 (m overlap to water signal, H-6), 3.47–3.70 (m, 28H, H-2–5), 3.85 (br dd, 1H, H-6′), 4.15 (dd, J 5.4 Hz, 1H, H-6′), 4.27–4.38 (m, 1H, OH-6), 4.44–4.54 (m, 5H, OH-6), 5.80–5.88 (m, 7H, H-1), 5.66–5.83 (m, 14H, OH-2, OH-3), 7.11 (d, J 8.1 Hz, 2H, H–C_{ar}-m), 7.466 (d, J 8.1 Hz, 2H,

H–C_{ar}-o), 8.21 (br s, 2H, NH_2); ^{13}C NMR (75 MHz, Me_2SO-d_6): δ 11.7 (CH_3^{Et}), 21.4 (CH_3^{ar}), 21.8 (–*C*H_2–), 30.9 (–OCO*C*H_2–), 42.7 (CH_2^{Et}), 46.2 (–*C*$H_2NH_2^+CH_2CH_3$), 60.7 (C-6), 72.8, 73.1, 73.7 (C-2, C-3, C-5), 81.8, 82.2, 82.3, 82.5, 82.9 (C-4) 102.4, 102.7, 103.0, 103.21 (C-1), 126.2 (C_{ar}-o), 128.7 (C_{ar}-m), 138.2 (C_{ar}-q), 146.5 (C_{ar}-q), 172.7 (*C*O). ESIMS: *m*/*z* 1248 [M+H^+]; MS/MS (*m*/*z* 1248, OR = 100 V): 1248 (100%), 1086 (0.6%), 924 (3%), 762 (1.5%), 600 (4.7%), 438 (6%), 276 (3.2%), 163 (1.4%), 114 (1.2%); HRE-SIMS: Calcd for $C_{55}H_{897}NO_{39}+H^+$ 1248.4611, found *m*/*z* 1248.4575.

1.3. Benzyl *N*-methyl-4-aminobutanoate *p*-toluene-sulfonate (5)

Benzyl tosylate[8] (150 mg, 0.572 mmol) was dissolved in dry NMP (8 mL) and the soln was stirred at 90 °C for 24 h under N_2. The solvent was removed by distillation 'bulb to bulb' under diminished pressure, and the yellow oily residue was dried under diminished pressure (0.001 mmHg) to give **5** (160 mg) as an oil (0.45 mmol, 79%); TLC (eluent A): R_f 0.10; ^1H NMR (300 MHz, D_2O): δ 1.80 (m, 2H, –C*H*$_2$–), 2.20 (s, 3H, CH_3^{Tos}), 2.25 (m, 2H, –OCOC*H*$_2$–), 2.50 (s, 3H, CH_3^{am}), 2.82 (m, 2H, –C*H*$_2NH_2$), 4.99 (s, 2H, CH_2^{Bn}), 7.09 (d, *J* 7.8 Hz, 2H, H–C_{Tos}-o), 7.19 (s, 6H, H–C_{Bn}), 7.45 (t, *J* .8 Hz, 2H, H–C_{Tos}-m). ESIMS: *m*/*z* 208 [M+H^+], 230 [M+Na^+]; MS/MS (*m*/*z* 208, OR = 60 V): *m*/*z* 208 (15%), 91 (100%).

Supplementary data

ROESY experiment on NMP/**1** mixture are reported in Figure S1. DOSY experiment on NMP in the free state and in the presence of **1** are presented in Figure S2. ^1H and ^{13}C NMR data of compounds **2**–**4** are available in Figures S3–S8. Synthetic procedures and physical data of 6^I-(*o*-biphenylamino)-6^I-deoxycyclomaltoheptaose and 6^I-(1-naphtylamino)-6^I-deoxycyclomaltoheptaose are described in S9. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.carres.2006.05.015.

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