Electronic Supplementary Material (ESI) for Journal of Materials Chemistry B.

Supporting Information

Synthesis of the Anionic Polyrotaxane Hydroxypropyl-β-cyclodextrin:Poly(decamethylenephosphate) and Evaluation of its Cholesterol Efflux Potential in Niemann-Pick C1 Cells

Kerstin Egele[1], *Shayak Samaddar*[2], *Nina Schneider*[3], *David Thompson*[2*], *Gerhard Wenz*[1*]

[1] Organic Macromolecular Chemistry, Saarland University, Saarbrücken, Germany

[2] Dept. of Chemistry, Purdue University, Lafayette, USA

[3] BASF SE, Ludwigshafen, Germany

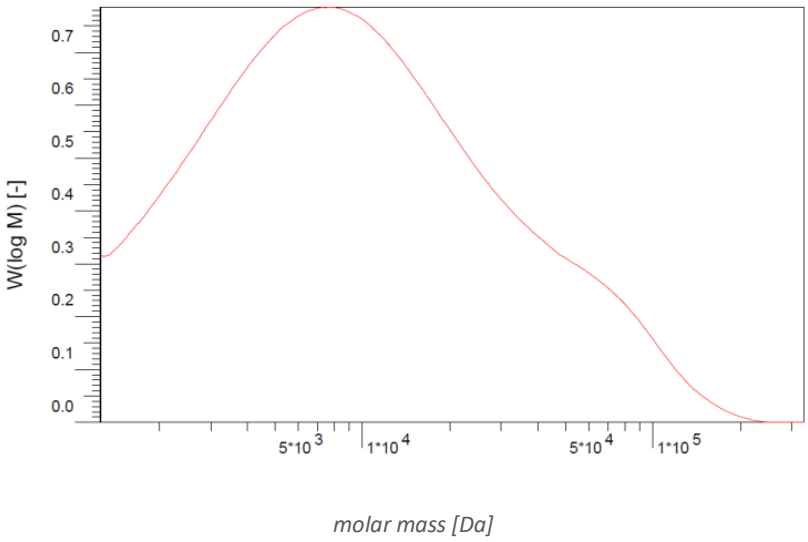


Figure S1A: Distribution of molar mass of poly(decamethylene phosphate) ***1*** *in 66 mM phosphate buffer pH 10 with 5 % ACN, calibration with pullulan.*

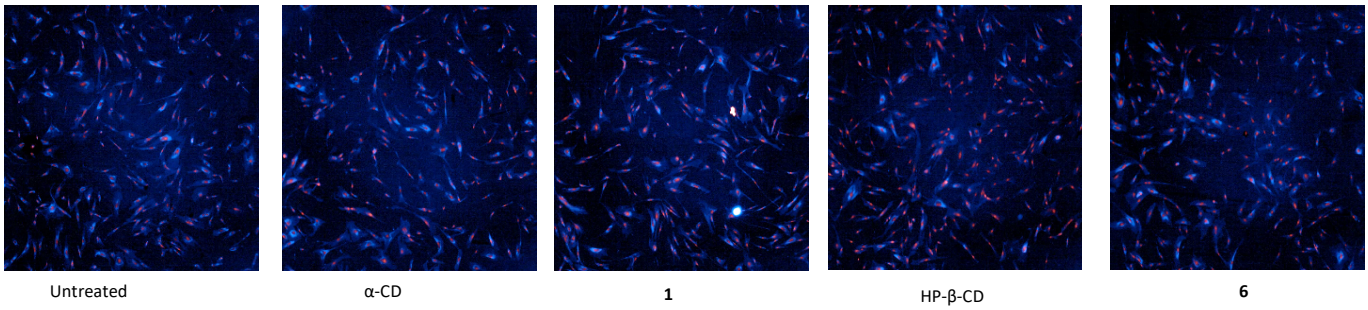
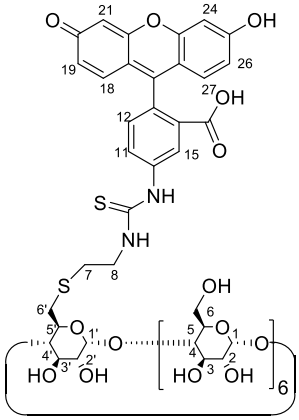


*Figure S 2: Filipin staining of primary fibroblasts after treatment with α-CD, poly-(decamethylenephosphate) **1**, HP-β-CD and **6** for 24 h (25 µM equivalent HP-β-CD dose. Nuclei were stained with NuclearRed. Cells were imaged with a 5 x objective.*

Synthesis of FTIC-tagged CD 7

FTIC-tagged CD **7** was prepared according to Kräuter *et al.*[1] starting from Mono-[6-deoxy-6-(2-aminoethylsulfanyl)]-β-CD which has been synthesized before according to Steffen *et al.*[2]



Mono-[6-deoxy-6-(2-aminoethylsulfanyl)]-β-CD (1.00 g, $8.1 \cdot 10^{-4}$ mol, 1.0 eq.) were dissolved in 18 mL anhydrous pyridine. A solution of fluorescein-5-isothiocyanate (Isomer I) (634 mg, $1.62 \cdot 10^{-3}$ mol, 2.0 eq.) in 18 mL pyridine was added over 20 min under N_2. The reaction mixture was heated unter stirring to 60 °C. After 3 h, the solvent was evaporated in vacuum at 60 °C. The residue was dissolved in 15 mL N, N-dimethylacetamide and precipitated in 200 mL cold acetone. The mixture was stirred overnight, then the precipitate was collected by centrifugation. The residue was redissolved in 100 ml water and stirred overnight. The crude product was purified by ultrafiltration using a 500 Da regenerate cellulose membrane against a 0.01 M NH_4OH solution and H_2O in sequence before freeze drying. A yellow, fluffy solid (816 mg, $5.15 \cdot 10^{-4}$ mol, 64 %) was recovered. ^{1}H-NMR: δ/ppm (DMSO-d_6): 9.24 (bs, 1H, COOH), 8.11 (bs, 1H, Ar-OH), 7.54-6.97 (m, 5H, H-11/12/15, NH), 6.83-6.46 (m, 6H, H-18/19/21/24/26/27), 6.36-5.25 (bs, 20 H, OH), 5.09-4.71 (m, 7H, H-1/1'), 4.10-3.45 (m, 26 H, H-3/3'/5/5'/6), 3.44-3.21 (m, 14H, H-2/2'/4/4'), 3.15-2.91 (m, 3H, H-6'a/H-8), 2.91-2.64 (m, 3H, H-6'b/H-7).

(1) Krauter, I.; Herrmann, W.; Wenz, G. *J. Incl. Phenom. Mol. Recognit. Chem.* **1996**, *25* (1–3), 93.

(2) Steffen, A.; Thiele, C.; Tietze, S.; Strassnig, C.; Kämper, A.; Lengauer, T.; Wenz, G.; Apostolakis, J.; Kamper, A.; Lengauer, T.; Wenz, G.; Apostolakis, J. *Chem. Eur. J.* **2007**, *13*, 6801.

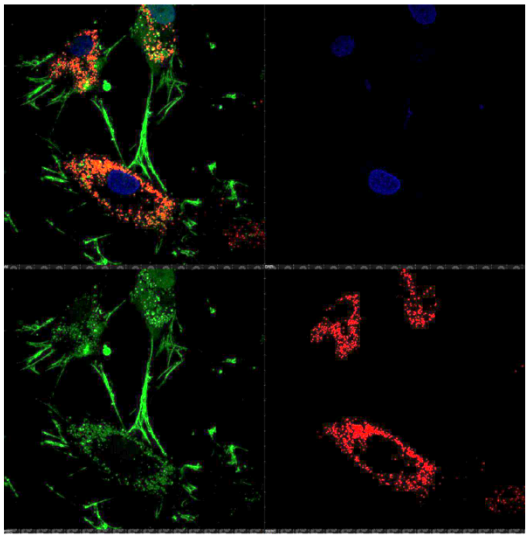


Figure S 3: NPC1-deficient cells treated with 25μM CD-equivalent FITC tagged polyrotaxane ***6a*** *in Opti-MEM media. (24 h* ***6a*** *treatment). We proposed extra cellular matrix association as the fluorescent signal appears in the characteristic shape of fibronectin structure.*