Carbohydrate-Rod-Conjugates - Ternary Rod-Coil Block Molecules Forming Complex Liquid Crystal Structures

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Electronic Supporting Information

1. Additional Data

1.1. X-ray scattering

Conditions: Powder X-ray investigations were carried out with a Guinier film camera (Huber). Samples in glass capillaries (∅ 1 mm) in a temperature-controlled heating stage, quartz-monochromatized CuK_α radiation, 30 to 60 min exposure time, calibration with the powder pattern of $Pb(NO_3)_2$. Aligned samples were obtained on a glass plate. Alignment was achieved upon slow cooling (rate: 1 K min^{-1} – 0.01 K min^{-1}) of a small droplett of the sample. Alignment takes place at the sample–glass or at the sample–air interface, with domains fiber-like disordered around an axis perpendicular to the interface. The aligned samples were held on a temperature-controlled heating stage and the diffraction patterns were recorded with a 2D detector (HI-STAR, Siemens).

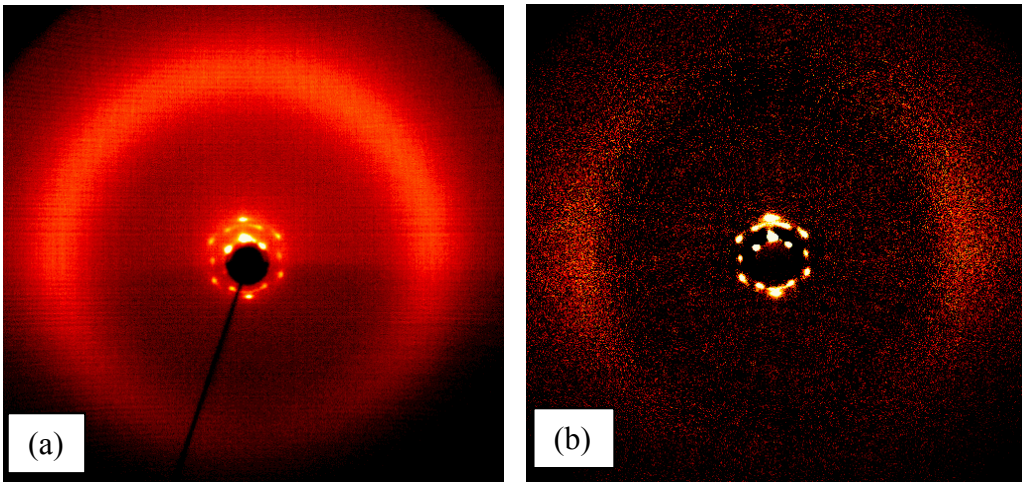


Figure S1. (a) 2D X-ray diffraction pattern of the Col_{hex} phase of **A6/6** at 80 °C. (b) Scattering intensity at 80 °C minus the scattering intensity of the isotropic liquid sample. The outer scattering shows a slight enhancement of the intensity near the equator of the pattern. This feature could be expected for the scattering of a fiber-like disordered sample with the terphenyl units perpendicular to the direction of the columns. The effect is rather week, since this scattering is isotropically distributed in the direction parallel to the columns and only the domains, where the columns are oriented perpendicular to the incident X-ray beam, contribute to the enhancement (a), but the anisotropy of the scattering becomes rather obvious when compared with that of an isotropic liquid sample (b), thus supporting the structure model of the Col_{hex} phase.

Table S1. X-ray data from Guinier powder patterns (θ_{obs}: experimental scattering angle; d_{obs}: experimental and d_{calc}: calculated d spacing; *hk/hkl*: assigned indices for 2D (Col_{squ}, Col_{hex})/3D (ChL_{hex}) phases, Parameter used: Lattice parameters or *d* values used to calculate d_{calc} with an error of the calculated parameters in the order of 0.1 nm)

Compound	*T* (°C)	Phase Plane group	θ_{obs} (°)	d_{obs} (nm)	*hk/ hkl*	d_{calc} (nm)	d_{obs}- d_{calc} (nm)	Parameter used (nm)
A4/2	105	Col_{hex}	1.375	3.23	10			a = 3.72
	90	Col_{squ}	0.875	5.05	11	5.01	0.04	a = 7.08
		*p*4*gm*	1.256	3.51	20	3.54	-0.03	
			1.404	3.14	21	3.17	-0.03	
			1.755	2.51	22	2.50	0.01	
			1.976	2.23	31	2.24	-0.01	
			2.239	1.97	32	1.96	0.01	
A4/3	90	Col_{hex}	1.250	3.53	10	3.51	0.02	a = 4.05
			2.184	2.02	11	2.03	-0.01	
			2.506	1.76	20	1.75	0.01	
	50	Col_{hex}	1.213	3.64	10	3.63	0.01	a = 4.19
			2.105	2.10	11	2.10	0.00	
			2.441	1.81	20	1.81	0.00	

Table S1. continued

Compound	*T* (°C)	Phase Plane group	θ_{obs} (°)	d_{obs} (nm)	*hk/ hkl*	d_{calc} (nm)	d_{obs}- d_{calc} (nm)	Parameter used (nm)
A4/4	85	Col_{hex}	1.213	3.64	10	3.66	-0.02	a = 4.23
			2.078	2.12	11	2.12	0.00	
			2.398	1.84	20	1.83	0.01	
	55	Col_{hex}	1.163	3.80	10	3.79	0.01	a = 4.38
			2.019	2.19	11	2.19	0.00	
			2.329	1.89	20	1.90	-0.01	
A6/3	100	Col_{squ}	0.800	5.56	11	5.56	0.00	a = 7.87
		*p*4*gm*	1.138	3.88	20	3.93	-0.05	
			1.275	3.46	21	3.52	-0.06	
			1.588	2.78	22	2.78	0.00	
			1.795	2.46	31	2.29	-0.03	
			2.031	2.18	32	2.18	0.00	
		Col_{squ}	1.237	3.57	10	3.57	0.00	a = 3.57
		*p*4*mm*	1.750	2.52	11	2.52	0.00	
			2.475	1.79	20	1.79	0.00	
A6/5	80	Col_{hex}	1.100	4.01	10	4.04	-0.03	a = 4.67
			1.880	2.35	11	2.34	0.01	
			2.179	2.03	20	2.02	0.01	
	70	Col_{squ}	0.750	5.89	11	5.87	0.02	a = 8.30
		*p*4*gm*	1.071	4.12	20	4.15	-0.03	
			1.197	3.69	21	3.71	-0.02	
			1.680	2.63	31	2.62	0.01	
			1.914	2.31	32	2.30	0.01	
			2.179	2.03	41	2.01	0.02	

Table S1. continued

Compound	T (°C)	Phase Plane group	θ_{obs} (°)	d_{obs} (nm)	*hk/ hkl*	d_{calc} (nm)	d_{obs}- d_{calc} (nm)	Parameter used (nm)
A6/6	80	Col_{hex}	1.050	4.21	10	4.20	0.01	a = 4.85
			1.817	2.43	11	2.42	0.01	
			2.108	2.09	20	2.10	-0.01	
A8/3	140	Col_{squ}	1.175	3.76	10	3.73	0.03	a = 3.73
		p4mm	1.672	2.64	11	2.64	0.00	
			2.375	1.86	20	1.86	0.00	
	25	Col_{squ}	1.113	3.97	10	3.79	0.00	a = 3.97
		p4mm	1.567	2.82	11	2.81	0.01	
			2.228	1.98	20	1.98	0.00	
A10/0	120	SmA	1.341	3.29	-	-	-	-
A10/1	100	ChL_{hex}	1.175	3.76	001	3.76	0.00	c = 3.76
			1.275	3.46	100	3.46	0.00	a = 4.00
			1.725	2.56	101	2.55	0.01	
A10/2	130	Col_{squ}	1.200	3.68	10	3.70	-0.02	a = 3.70
		p4mm	1.671	2.64	11	2.62	0.02	
			2.388	1.85	20	1.85	0.00	
	90	ChL_{hex}	1.195	3.76	001	3.77	-0.01	c = 3.77
			1.195	3.76	100	3.77	-0.01	a = 4.35
			1.658	2.66	101	2.66	0.00	
			2.337	1.89	002	1.89	0.00	

Table S1. continued

Compound	T (°C)	Phase Plane group	θ_{obs} (°)	d_{obs} (nm)	*hk/ hkl*	d_{calc} (nm)	d_{obs}- d_{calc} (nm)	Parameter used (nm)
A10/4	135	Col_{squ}	1.113	3.97	10	3.96	0.01	a = 3.96
		*p*4*mm*	1.571	2.81	11	2.80	0.01	
			2.235	1.97	20	1.98	-0.01	
	115	Col_{squ}	1.100	4.01	10	4.02	-0.01	a = 4.02
		*p*4*mm*	1.555	2.84	11	2.84	0.00	
			2.193	2.01	20	2.01	0.00	
A10*/3	100	Col_{squ}	1.188	3.72	10	3.72	0.00	a = 3.72
		*p*4*mm*	1.681	2.63	11	2.63	0.00	
			2.371	1.86	20	1.86	0.00	
	25	Col_{squ}	1.150	3.84	10	3.86	-0.02	a = 3.86
		*p*4*mm*	1.616	2.73	11	2.73	0.00	
			2.288	1.93	20	1.93	0.00	
A16/3	80	ChL_{hex}	0.950	4.65	001	4.65	0.00	c = 4.65
			1.075	4.11	100	4.11	0.00	a = 4.75
			1.425	3.10	101	3.08	0.02	
			1.875	2.36	110	2.38	-0.02	
			1.900	2.33	002	2.33	0.00	
A16/4	65	ChL_{hex}	0.925	4.72	001	4.70	0.02	c = 4.70
			1.025	4.31	100	4.36	-0.05	a = 5.04
			1.375	3.21	101	3.20	0.01	
			1.750	2.52	110	2.52	0.00	
			1.875	2.39	002	2.35	0.04	

Table S1. continued

Compound	*T* (°C)	Phase Plane group	θ_{obs} (°)	d_{obs} (nm)	*hk/ hkl*	d_{calc} (nm)	d_{obs}- d_{calc} (nm)	Parameter used (nm)
A6.16/3	130	Col_{squ}	1.075	4.11	10	4.11	0.00	a = 4.11
		*p*4*mm*	2.150	2.05	20	2.05	0.00	
	90	Col_{squ}	1.050	4.20	10	4.20	0.00	a = 4.20
		*p*4*mm*	1.484	2.97	11	2.97	0.00	
			2.100	2.10	20	2.10	0.00	
A6.16/4	130	Col_{squ}	1.050	4.21	10	4.20	0.01	a = 4.20
		*p*4*mm*	2.088	2.12	20	2.10	0.02	
	90	Col_{squ}	1.038	4.26	10	4.38	-0.12	a = 4.38
		*p*4*mm*	1.425	3.10	11	3.10	0.00	
			2.013	2.19	20	2.19	0.00	
A6.16/6	80	Col_{squ}	0.975	4.53	10	4.50	0.03	a = 4.50
		*p*4*mm*	1.392	3.17	11	3.18	-0.01	
			1.958	2.25	20	2.25	0.00	
A16.6/3	130	Col_{squ}	1.112	3.97	10	3.98	-0.01	a = 3.98
		*p*4*mm*	2.223	1.99	20	1.99	0.00	
	80	Col_{squ}	1.050	4.21	10	4.20	0.01	a = 4.20
		*p*4*mm*	1.488	2.97	11	2.97	0.00	
			2.113	2.09	20	2.10	-0.01	
A16.6/4	110	Col_{squ}	1.038	4.26	10	4.23	0.03	a = 4.23
		*p*4*mm*	1.475	2.99	20	2.99	0.00	
			2.098	2.10	11	2.11	-0.01	

Table S1. continued

Compound	T (°C)	Phase Plane group	θ_{obs} (°)	d_{obs} (nm)	*hk/ hkl*	d_{calc} (nm)	d_{obs}- d_{calc} (nm)	Parameter used (nm)
A16.6/6	95	Col_{squ}	1.000	4.42	10	4.42	0.00	a = 4.42
		*p*4*mm*	1.413	3.13	20	3.13	0.00	
			1.999	2.21	11	2.21	0.00	
B10/3	130	Col_{squ}	1.000	4.01	10	4.00	0.01	a = 4.00
		*p*4*mm*	1.558	2.83	11	2.83	0.00	
			2.211	2.00	20	2.00	0.00	
	90	Col_{squ}	1.101	4.01	10	4.08	-0.07	a = 4.08
		*p*4*mm*	1.519	2.90	11	2.88	0.02	
			2.160	2.04	20	2.04	0.00	
B10/4	150	Col_{squ}	1.100	4.01	10	4.01	0.00	a = 4.01
		*p*4*mm*	1.557	2.83	11	2.84	-0.01	
			2.194	2.01	20	2.01	0.00	
	100	Col_{squ}	1.075	4.11	10	4.17	-0.06	a = 4.17
		*p*4*mm*	1.490	2.96	11	2.95	0.01	
			2.120	2.08	20	2.08	0.00	

High-resolution X-ray

High-resolution small-angle powder diffraction experiments were recorded at Station 16.1 of the synchrotron at Daresbury, U.K.. Samples were held in evacuated 1 mm capillaries. A modified Linkam hot stage was used, with a hole for the capillary drilled through the silver heating block and mica windows attached to it on each side. A quadrant multiwire proportional detector was used. *Q* calibration and linearization were verified using several orders of layer reflections from a series of *n*-alkanes. Diffraction intensities were Lorentz and multiplicity corrected. Fourier reconstruction of the electron density is carried out using the general formula:

$$\rho(xyz) = \Sigma_{hkl} F(hkl) \exp[2\pi i(hx+ky+lz)] = \Sigma_{hkl} \sqrt{I(hkl)} \exp[2\pi i(hx+ky+lz) + i\phi(hkl)].$$

Here $\phi(hkl)$ is the phase of the (hkl) reflection and I is the corrected intensity. For columnar phases (2-dimensional order) the relation

$$\rho(xy) = \Sigma_{hk} F(hk) \exp[2\pi i(hx+ky)] = \Sigma_{hk} \sqrt{I(hk)} \exp[2\pi i(hx+ky) + i\phi(hk)]$$

was used.

1.2. Molecular models

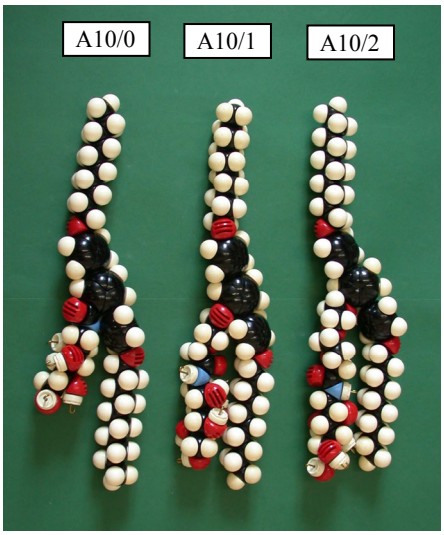
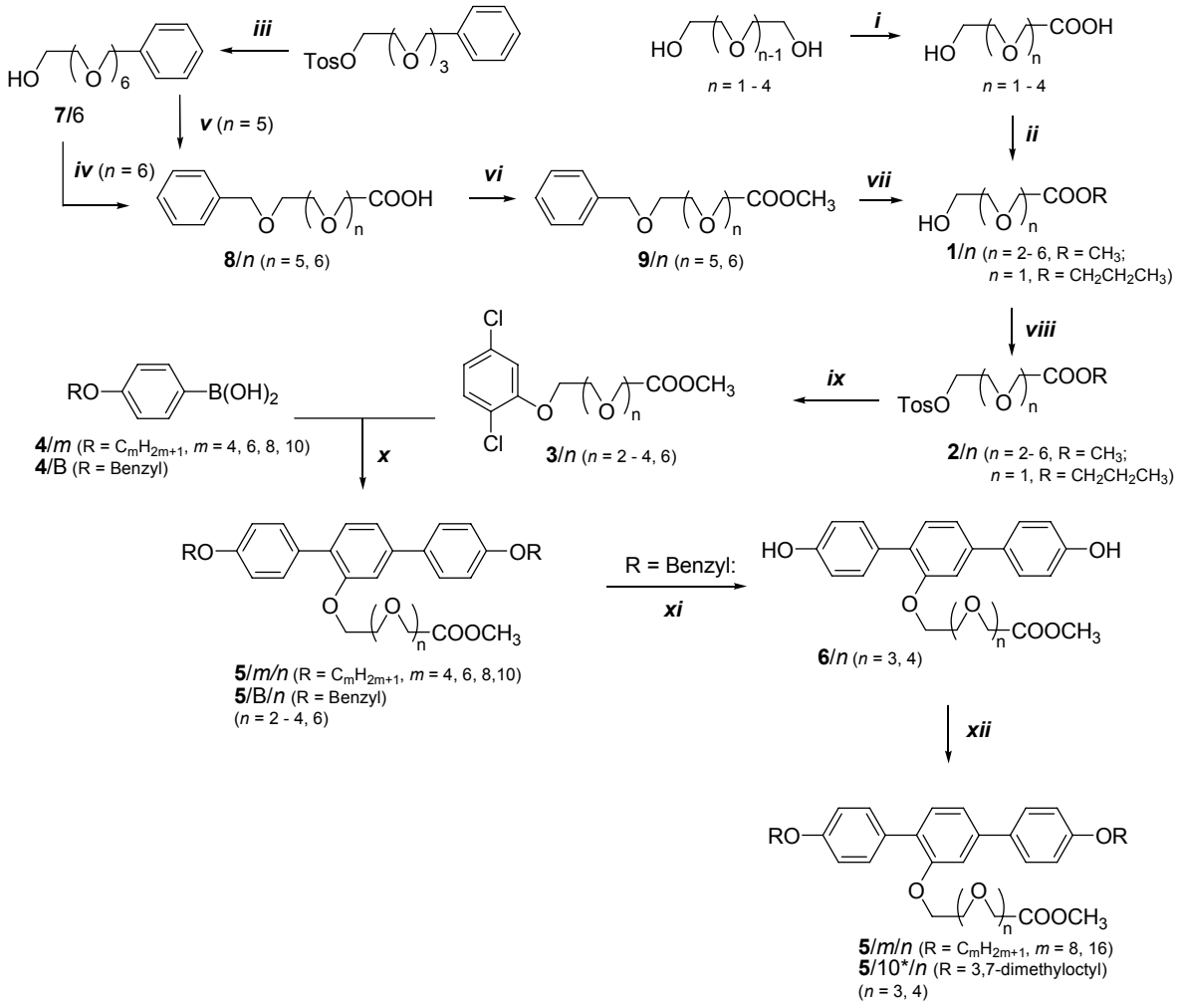


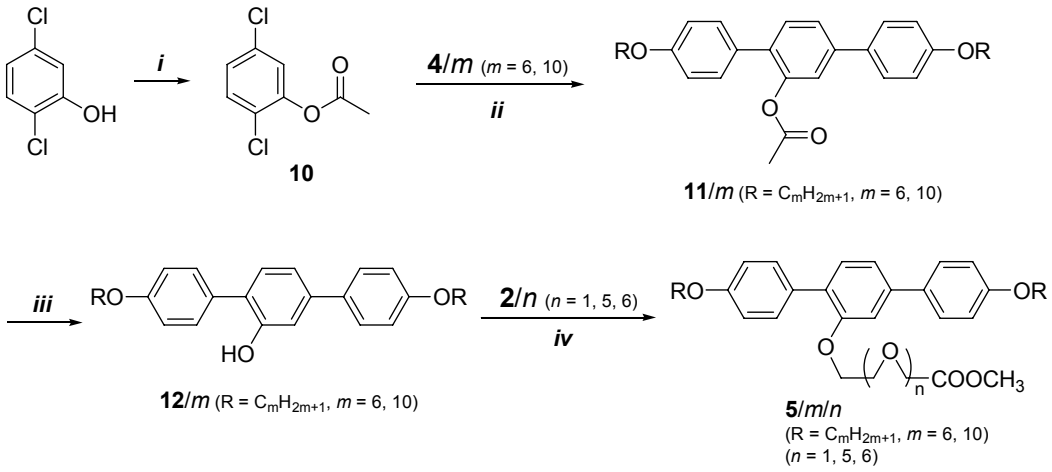
Figure S2. CPK models showing compounds **A10/0 - A10/2**.

2. Synthetic procedures and analytical data

2.1. Synthesis of the methyl ω-(4,4''-dialkoxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates (5/*m*/*n*)



Scheme S1. Synthesis of the esters **5**/*m*/*n*. *Reagents and conditions*: ***i***) 1. Na; 2. $ClCH_2COONa$, 100 °C, 10 h. 3, HCl (conc.); ***ii***) H_2SO_4, CH_3OH (or propanol), reflux, 10 h; ***iii***) $H(OCH_2CH_2)_3ONa$, 100 °C, 10 h; ***iv***) 1. NaH, THF, reflux 2 h; 2. $ClCH_2COONa$, reflux 8 h; 3. HCl (Conc.); ***v***) CrO_3, H_2SO_4, H_2O, acetone, 5 - 10 °C; ***vi***), H_2SO_4, CH_3OH, reflux, 10 h; ***vii***) Pd/C, H_2, ethylacetate, 40 °C, 24 h; ***viii***) tosylchloride, pyridine, 0 °C; ***ix***) 2,5-dichlorophenol, K_2CO_3, $(n\text{-}Bu)_4NI$, CH_3CN, reflux 8 h; ***x***) $Pd(OAc)_2$, 2-(di-*tert*-butylphosphino)biphenyl, KF, THF, r.t. 24 h; ***xi***) *Pearlman's* catalyst (palladium hydroxide on carbon), cyclohexene, methanol, reflux 8 h; ***xii***) 1-bromoalkane, K_2CO_3, $(n\text{-}Bu)_4NI$, CH_3CN, reflux 8 h.



Scheme S2. Synthesis of the esters **5**/*m*/*n*. *Reagents and conditions*: ***i***) DMAP, $(CH_3CO)_2O$, TEA, toluene, reflux, 10 h; ***ii***) $Pd(OAc)_2$, 2-(di-*tert*-butylphosphino)biphenyl, KF, THF; ***iii***) 1, NaOH (1 mol/L aq.), reflux 16 h, 2, HCl (Conc.); ***iv***) K_2CO_3, $(n\text{-}Bu)_4NI$, CH_3CN, reflux 8 h.

2.1.1. Methyl (or propyl) ω-hydroxy[oligo(oxyethylene)yl]acetates (1/*n*)

Genral procedure: The methyl (or propyl) ω-hydroxy[oligo(oxyethylene)yl]acetates (**1**/*n*) were synthesized according to ref. [1]. Metallic Na (15.4 g) was dissolved in the oligoethyleneglycol (2.7 mol) under an argon atmosphere, sodium chloroacetate (78 g, 0.67 mol) was added to the resulting solution at 100 °C, the mixture was then stirred at 100 °C for 10h. The excess oligoethyleneglycol was removed in vacuum. Water (100 mL), and 35 % hydrochloric acid (35 mL) were added to the residue, then the NaCl was removed by filtration. After evaporation of the water, methanol (or propanol for compound **1**/1) (600 mL) and sulphuric acid (10 mL) were added to the residue and the resulting mixture was refluxed for 10h. The solution was neutralized with saturated aqueous sodium carbonate solution and then the solvent was evaporated under reduced pressure. Water (400 mL) was added to the residue and the mixture was extracted with dichloromethane, the extraction procedure was repeated several times untill the last extraction contains no product as proven by TLC analysis. The combined organic phase was dried over Na_2SO_4, after filtration the solvent was evaporated in reduced pressure. The crude product was then purified by column chromatography on silica gel with $CHCl_3/CH_3OH$ as eluent or by flash distillation below 150 °C.

Propyl 5-hydroxy-3-oxapentanoate (**1**/1): Synthesized from ethylene glycol. Purified by column chromatography over silica gel 60 with $CHCl_3$/MeOH = 10/1 (V/V) as eluent; colorless liquid, yield 30 %. ^{1}H NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 4.07 (s, 2 H, OCH_2), 4.05 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.69-3.66 (m, 2 H, OCH_2), 3.62-3.59 (m, 2 H, OCH_2), 1.65-1.57 (m, 2 H, CH_2), 0.87 (t, ^3J(H,H) = 7.5, 3 H, CH_3).

Methyl 8-hydroxy-3,6-dioxaoctanoate (**1**/2): Synthesized from diethylene glycol. Purified by vacuum distillation, b.p. = 120 °C (8 Pa); colorless liquid, yield 24.5 %. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 4.12 (s, 2 H, OCH_2), 3.75-3.55 (m, 11 H, OCH_2, OCH_3).

Methyl 11-hydroxy-3,6,9-trioxaundecanoate (**1**/3): Synthesized from triethylene glycol. Purified by vacuum distillation, b.p. = 135 °C (3 Pa). Yield 12 %; colorless liquid; ^{1}H-NMR ($CDCl_3$, J/Hz, 200 MHz) δ = 4.06 (s, 2 H, OCH_2), 3.64-3.47 (m, 15 H, OCH_2, OCH_3).

Methyl 14-hydroxy-3,6,9,12-tetraoxatetradecanoate (**1**/4): Synthesized from tetraethylene glycol. Purified by column chromatography on silica gel 60 with $CHCl_3/CH_3OH$ = 10/1 (V/V) as Eluent to yield a colourless oil. Yield 19 %. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 4.13 (s, 2 H, OCH_2), 3.71-3.53 (m, 19 H, OCH_2, OCH_3).

Methyl 17-benzyloxy-3,6,9,12,15,-pentaoxaheptadecanoate **(1/5) and** ***methyl 20-benzyloxy-3,6,9,12,15,18-hexaoxaeicosanoate*** **(1/6)**

Hexaethyleneglycol monobenzyl ether (**7**/6): Under an argon atmosphere, metallic Na (5.0 g) was dissolved in the triethlyeneglycol (130 g, 0.87 mol). To the resulting solution, 8-[4-toluenesufonyloxy]-3,6-dioxaoctylbenzyl ether (78 g, 0.20 mol) was added dropwise. The resulting mixture was heated at 100 °C for 8 h, after that the reaction mixture was poured into 200 mL water and extracted with CH_2Cl_2 (3 × 150 mL), the combined organic phase was washed with water (2 × 100 mL) and brine (100 mL), dried over Na_2SO_4 and filtered. The solvent was removed in vacuum, the crude product was purified by column chromatography over silica gel 60 with $CHCl_3/CH_3OH$ = 10/1.5 (V/V) as eluent, which afforded a colorless liquid, yield 52 g (70 %). ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.40-7.22 (m, 5 H, Ar-H), 4.55 (s, 2 H, OCH_2), 3.73-3.57 (m, 24 H, OCH_2).

20-Benzyloxy-3,6,9,12,15,18-hexaoxaeicosanoic acid (**8**/6): In a two-necked flask equipped with a reflux condenser and a magnetic stirring bar, **7**/6 (52 g, 0.14 mol) was dissolved in dry THF (400 mL) under an argon atmosphere. After the addition of NaH (80 %, 4.5 g, 0.15 mol), the mixture was stirred at reflux for 2h. After cooling, sodium chloroacetate (27 g, 0.23 mol) was added and the reaction mixture was heated at reflux again for 10 h. Afterward, the solvent was evaporated in vacuum, and the residue was dissolved in water (100 mL), acidified with HCl (10 %) and extracted with diethyl ether (3 × 150 mL). The combined organic phase was washed with water (2 × 100 mL), and with brine (100 mL). After drying over Na_2SO_4 and filtration, the solvent was removed in vacuum, which afforded a yellow liquid, 25 g (41 %). The crude product was used for the next step without further purification. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.33-7.22 (m, 5 H, Ar-H), 4.55 (s, 2 H, OCH_2), 4.13-4.11 (m, 2 H, OCH_2), 3.73-3.60 (m, 24 H, OCH_2).

17-Benzyloxy-3,6,9,12,15-pentaoxaheptadecanoic acid (**8**/5): A solution of chromic trioxide (9.2 g, 92 mmol) in water (92 mL) and sulphuric acid (96 %, 14 mL) was added to a cooled (0°C) solution of **7**/6 (20.0 g, 54 mmol) in acetone (300 mL). The reaction mixture was stirred at 5 – 10 °C until the starting alcohol had been completely consumed (TLC, *ca*. 2 h) and then quenched with 2-propanol (30 mL). The resultant mixture was concentrated to a small volume and extracted with CH_2Cl_2 (3 × 100 mL). The combined organic extracts were washed with water (3 × 50 mL) and extracted with saturated aqueous $NaHCO_3$ solutions (3 × 100 mL). The combined alkaline extracts were acidified with hydrochloric acid (36 %) and extracted with CH_2Cl_2 (3 × 100 mL). The combined organic extracts were dried over Na_2SO_4, after filtration the solvent was evaporated in vacuum to afford a colorless liquid, 12.7 g (33 mmol), yield 61 %. The crude product was used for the next step without further purification. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400

MHz) δ = 7.33-7.22 (m, 5 H, Ar-H), 4.54 (s, 2 H, OCH_2), 4.14-4.11 (m, 2 H, OCH_2), 3.73-3.60 (m, 20 H, OCH_2).

Methyl 20-benzyloxy-3,6,9,12,15,18-hexaoxaeicosanoate (**9**/6): Dry methanol (150 mL), sulphuric acid (96 %, 3 mL, 0.06 mol) and **8**/6 (25 g, 58 mmol) were carefully mixed and heated to reflux for 10 h. After cool down, the solution was neutralized with saturated aqueous Na_2CO_3 solution, then the result mixture was concentrated to small volume and extracted by CH_2H_2 (3 × 100 mL). The combined organic phase was washed with water (2 × 100 mL) and brine (100 mL). After drying over Na_2SO_4 and filtration, the solvent was removed in vacuum. The crude product was purified by column chromatography over silica gel 60 with $CHCl_3/CH_3OH$ = 10/1 (V/V) as eluent which afforded a colorless liquid, yield 16.7 g (70 %). ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.33-7.22 (m, 5 H, Ar-H), 4.54 (s, 2 H, OCH_2), 4.13 (s, 2 H, OCH_2), 3.74-3.57 (m, 27 H, OCH_2, OCH_3).

Methyl 17-benzyloxy-3,6,9,12,15-pentaoxaheptadecanoate (**9**/5): Synthesized as described above from **8/5**, purified by column chromatography over silica gel 60 with $CHCl_3/CH_3OH$ = 10/1 (V/V) as eluent, colorless liquid, yield 75 %. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.33-7.22 (m, 5 H, Ar-H), 4.53 (s, 2 H, OCH_2), 4.13 (s, 2 H, OCH_2), 3.73-3.55 (m, 23 H, OCH_2, OCH_3).

Methyl 20-hydroxy-3,6,9,12,15,18-hexaoxaeicosanoate (**1**/6): **9**/6 (16.7 g, 40.6 mmol) was dissolved in ethyl acetate (20 mL), the resulting solution was treated with palladium (10 % on carbon) catalyst (0.4 g, 1 % by mol) and was hydrogenated (1.05×10^5 Pa) at 40 °C until the starting benzyl ether had been completely consumed as judged by TLC analysis (*ca*. 8 h). The catalyst was filtered off and all volatiles were removed in vacuum, which afforded a colorless liquid, yield 13.5 g (94 %). The crude product was used for the next step without further purifications. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 4.14 (s, 2 H, OCH_2), 3.72 (s, 3 H, OCH_3), 3.71-3.55 (m, 24 H, OCH_2).

Methyl 17-hydroxy-3,6,9,12,15-pentaoxaheptadecanoate (**1**/5): Synthesized as described above from **9**/5, colorless liquid, yield 97 %. The crude product was used for the next step without further purifications. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 4.14 (s, 2 H, OCH_2), 3.72 (s, 3 H, OCH_3), 3.71-3.55 (m, 20 H, OCH_2).

2.1.2. Methyl (or propyl) ω-tosyloxy[oligo(oxyethylene)yl]acetates (2/*n*)

General procedure: Under an Argon atmosphere, approporite compound **1**/*n* (24 mmol) was dissolved in dry pyridine (7.6 g, 96 mmol) at 0 °C, after the addition of *p*-tosylchloride (9.2 g, 48 mmol) the reaction mixture was stirred for another 2 h at 0 °C, and then was put into the refrigerator overnight. Afterwards, the reaction mixture was poured into ice water mixture, extracted with diethyl ether (3 × 50 mL), the combined organic phases were washed with hydrochloric acid (aq. 10 %, 2 ×20 mL), water (2 × 30 mL) and brine (50 mL), and was dried over Na_2SO_4. After filtration and evaporation of the solvent, if no other declaration, the crude product was used for next step without further purification.

Propyl 5-(4-toluenesulfonyloxy)-3-oxapentanoate (**2**/1): Synthesized from **1**/1 (1.2 g, 7.4 mmol), *p*-tosyl chloride (2.82 g, 14.8 mmol) and pyridine (2.34 g, 29.6 mmol). Purified by column chromatography over silica gel 60 with $CHCl_3$/MeOH = 10/1 (V/V) as eluent; colorless oil, yield 98.9 %. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.78 (d, ^3J(H,H) = 8.5, 2 H, Ar-H), 7.32 (d,

^3J(H,H) = 8.1, 2 H, Ar-H), 4.20-4.03 (m, 6 H, OCH_2), 3.78-3.73 (m, 2 H, OCH_2), 2.42 (s, 3 H, Ar-CH_3), 1.70- 1.56 (m, 2 H, CH_2), 0.91 (t, ^3J(H, H) = 7.4, 3 H, CH_3).

Methyl 8-(4-toluenesulfonyloxy)-3,6-dioxaoctanoate (**2**/2): Synthesized from **1**/2 (8.83 g, 49.6 mmol), *p*-tosyl chloride (19.0 g, 99.7 mmol) and pyridine (15.8 g, 199.4 mmol). Yield 16.4 g (99.5 %); yellow oil. ^{1}H-NMR ($CDCl_3$, J/Hz, 400 MHz) δ = 7.79 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 4.17-4.05 (m, 4 H, OCH_2), 3.76-3.62 (m, 6 H, OCH_2), 3.57 (s, 3 H, OCH_3), 2.43 (s, 3 H, CH_3).

Methyl 11-(4-toluenesulfonyloxy)-3,6,9-trioxaundecanoate (**2**/3): Synthesized from **1**/3 (5.4 g, 24 mmol), *p*-tosylchloride (9.2 g, 48 mmol) and pyridine (7.6 g, 96 mmol). Yield: 9.1 g (100 %); yellow oil. ^{1}H-NMR ($CDCl_3$, J/Hz, 400 MHz) δ = 7.79 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 7.32 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 4.18-4.05 (m, 4 H, OCH_2), 3.77-3.63 (m, 10 H, OCH_2), 3.57 (s, 3 H, OCH_3), 2.43 (s, 3 H, CH_3).

Methyl 14-(p-toluenesulfonyloxy)-3,6,9,12-tetraoxatetradecanoate (**2**/4): Synthesized from **1**/4 (15.5 g, 58.3 mmol), *p*-tosylchloride (22.2 g, 117 mmol) and pyridine (18.5 g, 234 mmol). Yield: 21.5 g (89 %); pale yellow oil. ^{1}H NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.77 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 7.31 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 4.16-4.09 (m, 4 H, OCH_2), 3.74-3.51 (m, 17 H, OCH_2, OCH_3), 2.42 (s, 3 H, Ar-CH_3).

Methyl 17-[p-toluenesulfonyloxy]-3,6,9,12,15-pentaoxaheptadecanoate (**2**/5): Synthesized from **1**/5 (7.5 g, 25.5 mmol), p-tosyl chloride (9.7 g, 50.8 mmol) and pyridine (8.1 g, 102 mmol). Yield yellow oil 10.4 g (91 %). ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.76 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 7.30 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 4.16-4.11 (m, 4 H, OCH_2), 3.72-3.56 (m, 21 H, OCH_2, OCH_3), 2.41 (s, 3 H, CH_3).

Methyl 20-[p-toluenesulfonyloxy]-3,6,9,12,15,18-hexaoxaeicosanoate (**2**/6): Synthesized from **1**/6 (9.0 g, 25.4 mmol), p-tosyl chloride (9.7 g, 50.8 mmol) and pyridine (8.1 g, 102 mmol). Yield yellow oil 12.0 g (93 %). ^{1}H-NMR ($CDCl_3$, J/Hz, 200 MHz) δ = 7.78 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 7.32 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 4.16-4.11 (m, 4 H, OCH_2), 3.72-3.56 (m, 25 H, OCH_2, OCH_3), 2.43 (s, 3 H, CH_3).

2.1.3. Methyl ω-(2,5-dichlorophenyloxy)[oligo(oxyethylene)yl]acetates (**3**/*n*)

General procedure: Under an Argon atmosphere, **2**/*n* (24 mmol) and 2,5-dichlorophenol (4.3 g, 26 mmol) were dissolved in dry acetonitrile (100 mL). K_2CO_3 (8.3 g, 60 mmol) and (n-Bu)$_4$NI (20 mg) were added, and the mixture was stirred under reflux for 4-8 h. Afterwards, the solvent was evaporated in vacuum, and the residue was dissolved in water and diethyl ether, the water phase was extracted by diethyl ether (3 × 100 mL). The combined organic phase was washed with water (2 × 50 mL) and brine (50 mL). After drying over Na_2SO_4 and filtration, the solvent was removed, the crude product was purified by column chromatography on silica gel.

Methyl 8-(2,5-dichlorophenyloxy)-3,6-dioxaoctanoate (**3**/2): Synthesized from **2**/2 (16.4 g, 49.3 mmol), 2,5-dichlorophenol (8.4 g, 51.5 mmol), K_2CO_3 (17.0 g, 123 mmol), dry acetonitrile (100 mL) and (n-Bu)$_4$NI (50 mg). The crude product was purified by column chromatography over silica gel 60 with ethyl acetate/PE = 1/2 (V/V) as eluent. Yield 12.9 g (81 %); yellow oil. ^{1}H-NMR ($CDCl_3$, J/Hz, 400 MHz) δ = 7.24 (d, ^3J(H,H) = 8.3, 1 H, Ar-H), 6.93 (d, ^4J(H,H) = 2.3, 1

H, Ar-H), 6.86 (dd, ^3J(H,H) = 8.3, ^4J(H,H) = 2.3, 1 H, Ar-H), 4.15-4.09 (m, 4 H, OCH_2), 3.89 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.80-3.77 (m, 2 H, OCH_2), 3.73-3.54 (m, 5 H, OCH_2, OCH_3).

Methyl 11-(2,5-dichlorophenyloxy)-3,6,9-trioxaundecanoate (**3**/3): Synthesized from **2**/3 (9.1 g, 24 mmol), 2,5-dichlorophenol (4.3 g, 26 mmol), acetonitrile (100 mL), K_2CO_3 (8.3 g, 60 mmol) and $(n\text{-}Bu)_4NI$ (20 mg). The crude product was purified by column chromatography on silica gel with $CHCl_3/CH_3OH$ = 10/1 (V/V) as eluent. Yield 5.6 g (63.5 %); pale yellow oil. ^{1}H-NMR ($CDCl_3$, J/Hz, 200 MHz) δ = 7.24 (d, ^3J(H,H) = 8.3, 1 H, Ar-H), 6.93 (d, ^4J(H,H) = 2.3, 1 H, Ar-H), 6.86 (dd, ^3J(H,H) = 8.3, ^4J(H,H) = 2.3, 1 H, Ar-H), 4.18-4.13 (m, 4 H, OCH_2), 3.88 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.77-3.63 (m, 11 H, OCH_2, OCH_3).

Methyl 14-(2,5-dichlorophenyloxy)-3,6,9,12-tetraoxatetradecanoate (**3**/4): Synthesized from **2**/4 (21.5 g, 51.2 mmol), 2,5-dichlorophenol (8.6 g, 52.7 mmol), acetonitrile (150 mL), K_2CO_3 (25 g, 0.18 mol) and $(n\text{-}Bu)_4NI$ (50 mg). The crude product was purified by column chromatography on silica gel with $CHCl_3/CH_3OH$ = 10/1 (V/V) as eluent to yield a pale yellow oil. Yield 15.0 g (71 %). ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.23 (d, ^3J(H,H) = 8.4, 1 H, Ar-H), 6.92 (d, ^4J(H,H) = 2.2, 1 H, Ar-H), 6.84 (dd, ^3J(H,H) = 8.4, ^4J(H,H) = 2.2, 1 H, Ar-H), 4.17-4.12 (m, 4 H, OCH_2), 3.86 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.75-3.63 (m, 15 H, OCH_2, OCH_3).

Methyl 20-(2,5-dichlorophenyloxy)-3,6,9,12,15,18-hexaoxaeicosanoate (**3**/6): Synthesized from **2**/6 (3.5 g, 6.9 mmol), 2,5-dichlorophenol (1.3 g, 8.0 mmol), K_2CO_3 (2.4 g, 17.3 mmol), acetonitrile (100 mL) and $(n\text{-}Bu)_4NI$ (10 mg). Tthe crude product was purified by column chromatography over silica gel 60 with $CHCl_3$/MeOH = 10/1.5 (V/V) as eluent. Yield 2.52 g (73 %); yellow oil. ^{1}H-NMR ($CDCl_3$, J/Hz, 400 MHz) δ = 7.24 (d, ^3J(H,H) = 8.5, 1 H, Ar-H), 6.93 (d, ^4J(H,H) = 2.3, 1 H, Ar-H), 6.85 (dd, ^3J(H,H) = 8.5, ^4J(H,H) = 2.3, 1 H, Ar-H), 4.16-4.12 (m, 4 H, OCH_2), 3.88 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.75-3.62 (m, 23 H, OCH_2, OCH_3).

2.1.4. Methyl ω-(4,4''-dialkoxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates (5/*m*/*n*) and methyl ω-(4,4''-dibenzyloxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates (5/B/*n*)

2.1.4.1 Procedure A (C-C-coupling) [2]

An oven-dried Schlenk flask was evacuated and backfilled with argon and charged with $Pd(OAc)_2$ (15 mg, 0.067 mmol), 2-(di-*tert*-butylphosphino)biphenyl (45 mg, 0.15 mmol), 4-alkoxybenzeneboronic acid (9.4 mmol) and dry KF (2.02 g, 35 mmol). The flask was evacuated and backfilled with argon, then dry THF (6 mL) and **3**/*n* (3.2 mmol) were added by syringe through a rubber septum. The reaction mixture was stirred at room temperature until the starting dichloro compound **3**/*n* had been completely consumed as judged by TLC analysis (*ca*. 24 h). The reaction mixture was diluted with diethyl ether and poured into a separatory funnel. The mixture was washed with aqueous NaOH (1 M, 50 mL), and the aqueous phase was extracted with diethyl ether (3 × 50 mL), and the combined organic phase was washed with brine (50 mL), dried over Na_2SO_4, filtered, and concentrated in vacuum. The crude product was purified by column chromatography on silica gel.

Methyl 8-(4,4''-dibutyloxy-p-terphenyl-2'-yloxy)-3,6-dioxaoctanoate (**5**/4/2): Synthesize from **3**/2 (1.38 g, 4.3 mmol) and 4-butyloxybenzeneboronic acid (2.5 g, 13 mmol), using $Pd(OAc)_2$ (34 mg, 0.15 mmol), 2-(di-*tert*-butylphosphino)biphenyl (91 mg, 0.30 mmol) and KF (2.6 g, 45 mmol). The crude product was purified by column chromatography over silica gel 60 with

$CHCl_3/CH_3OH$ = 10/0.1 (V/V) as eluent. Yield 2.4 g (100 %); colorless solid; m.p. = 56 °C. ^{1}H-NMR ($CDCl_3$, J/Hz, 200 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.34 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.11 (s, 2 H, OCH_2), 4.00 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.78-3.65 (m, 7 H, OCH_2, OCH_3), 1.81-1.74 (m, 4 H, CH_2), 1.55-1.47 (m, 4 H, CH_2), 1.00-0.95 (m, 6 H, CH_3).

Methyl 11-(4,4''-dibutyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**5**/4/3): Synthesized from **3**/3 (1.89 g, 5.1 mmol) and 4-butyloxybenzeneboronic acid (3.0 g, 15 mmol), using $Pd(OAc)_2$ (29 mg, 0.13 mmol), 2-(di-*tert*-butylphosphino)biphenyl (77 mg, 0.26 mmol) and KF (2.3 g, 40 mmol). The crude product was purified by column chromatography over silica gel 60 with $CHCl_3$/ CH_3OH = 10/0.1 (V/V) as eluent. Yield 3.1 g (100 %); colorless solid; m.p. = 49 °C. ^{1}H-NMR ($CDCl_3$, J/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 4.00 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.71-3.56 (m, 11 H, OCH_2, OCH_3), 1.82-1.73 (m, 4 H, CH_2), 1.57-1.45 (m, 4 H, CH_2), 1.00-0.95 (m, 6 H, CH_3).

Methyl 14-(4,4''-dibutyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxa-tetradecanoate (**5**/4/4): Synthesized from **3**/4 (1.96 g, 4.8 mmol) and 4-butyloxybenzeneboronic acid (2.8 g, 14 mmol), using $Pd(OAc)_2$ (21 mg, 0.094 mmol), 2-(di-*tert*-butylphosphino)biphenyl (57 mg, 0.19 mmol) and KF (1.6 g, 28 mmol). The crude product was purified by column chromatography over silica gel 60 with $CHCl_3/CH_3OH$ = 10/0.3 (V/V) as eluent. Yield 2.6 g (85 %); colorless solid; m.p. = 30 °C. ^{1}H-NMR ($CDCl_3$, J/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.8, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.8, ^4J(H,H) = 1.5, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 4.00 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.77-3.58 (m, 15 H, OCH_2, OCH_3),), 1.80-1.75 (m, 4 H, CH_2), 1.53-1.42 (m, 4 H, CH_2), 1.00-0.95 (m, 6 H, CH_3).

Methyl 11-(4,4''-dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**5**/6/3): Synthesized from **3**/3 (1.98 g, 5.4 mmol) and 4-hexyloxybenzeneboronic acid (3.0 g, 14 mmol), using $Pd(OAc)_2$ (38 mg, 0.17 mmol), 2-(di-*tert*-butylphosphino)biphenyl (102 mg, 0.34 mmol) and KF (3.0 g, 52 mmol). The crude product was purified by column chromatography over silica gel 60 with $CHCl_3/CH_3OH$ = 10/0.1 (V/V) as eluent. Yield 3.2 g (100 %); colorless solid; m.p. = 44 °C. ^{1}H-NMR ($CDCl_3$, J/Hz, 200 MHz) δ = 7.52 (d, ^3J(H,H) = 8.9, 4 H, Ar-H), 7.34 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.79(t, ^3J(H,H) = 5.0, 2 H, OCH_2,), 3.7 (s, 3 H, OCH_3), 3.68-3.58 (m, 8 H, OCH_2), 1.79-1.72 (m, 4 H, CH_2), 1.48-1.24 (m, 12 H, CH_2), 0.94-0.87 (m, 6 H, CH_3).

Methyl 11-(4,4''-dibenzyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate **(5**/B/3**)**: Synthesized from **3**/3 (2.69 g, 7.34 mmol), and 4-benzyloxybenzeneboronic acid (5.02 g, 22.0 mmol) using $Pd(OAc)_2$ (43.6 mg, 0.19 mmol), 2-(di-*tert*-butylphosphino)biphenyl (128 mg, 0.43 mmol) and

KF (3.3 g, 57.0 mmol). The crude product was purified by column chromatography over silica gel 60 with ethyl acetate/PE = 1/1 (V/V) as eluent. Yield 4.7 g (97 %); colorless solid; m.p. = 72 °C. ^{1}H-NMR ($CDCl_3$, J/Hz, 200 MHz) δ = 7.55-7.28 (m, 15 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.04 (d, ^3J(H,H) = 8.6, 2 H, Ar-H), 7.00 (d, ^3J(H,H) = 8.5, 2 H, Ar-H), 5.11 (s, 2 H, OCH_2), 5.10 (s, 2 H, OCH_2), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.11 (s, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2),), 3.70-3.57 (m, 11 H, OCH_2, OCH_3).

Methyl 14-(4,4"-dibenzyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**5**/B/4). Synthesized **3***/4* (3.24 g, 7.88 mmol) and 4-benzyloxybenzeneboronic acid (5.39 g, 23.7 mmol) using $Pd(OAc)_2$ (53.8 mg, 0.24 mmol), 2-(di-*tert*-butylphosphino)-biphenyl (143 mg, 0.48 mmol) and KF (4.18 g, 72 mmol). Yield 5.0 g (90 %); mp. = 71 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.54 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.53 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.45 (d, ^3J(H,H) = 7.7, 4 H, Ar-H), 7.25-7.41 (m, 7 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.04 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.00 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 5.11 (s, 2 H, OCH_2COO), 5.09 (s, 2 H, OCH_2), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.11 (s, 2 H, OCH_2COO), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.70 (s, 3 H, OCH_3), 3.68-3.59 (m, 12 H, OCH_2).

Methyl 11-(4,4''-dioctyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**5**/8/3): Synthesized from **3**/3 (0.587 g, 1.6 mmol) and 4-octyloxybenzeneboronic acid (1.0 g, 4.0 mmol), using $Pd(OAc)_2$ (14.5 mg, 0.065 mmol), 2-(di-*tert*-butylphosphino)biphenyl (38.6 mg, 0.129 mmol) and KF (1.1 g, 19 mmol). The crude product was purified by column chromatography over silica gel 60 with ethyl acetate/n-hexane = 1/2 (V/V) as eluent. Yield 0.71 g (62.5 %); colorless solid; m.p. = 43 °C. ^{1}H-NMR ($CDCl_3$, J/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.71-3.58 (m, 11 H, OCH_2, OCH_3), 1.83-1.73 (m, 4 H, CH_2), 1.57-1.24 (m, 20 H, CH_2), 0.89-0.86 (m, 6 H, CH_3).

Methyl 8-(4,4''-didecyloxy-p-terphenyl-2'-yloxy)-3,6-dioxaoctanoate (**5**/10/2): Synthesized from **3**/2 (0.917 g, 3.29 mmol) and 4-decyloxybenzeneboronic acid (2.37 g, 8.5 mmol), using $Pd(OAc)_2$ (20 mg, 0.089 mmol), 2-(di-*tert*-butylphosphino)biphenyl (53 mg, 0.18 mmol), KF (1.55 g, 26.7 mmol) and dry THF (10 mL). The crude product was purified by column chromatography over silica gel 60 with $CHCl_3$/MeOH = 10/0.1 (V/V) as eluent. Yield 1.9 g (80 %); colorless waxy solid; transitions/°C: Cr 60 SmA 72 Iso. ^{1}H-NMR ($CDCl_3$, J/Hz, 200 MHz) δ = 7.52 (d, ^3J(H,H) = 8.4, 4 H, Ar-H), 7.34 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.8, 1 H, Ar-H), 7.14 (d, ^4J(H,H) = 1.8, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.11 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.73-3.61 (m, 7 H, OCH_2, OCH_3), 1.88-1.73 (m, 4 H, CH_2), 1.53-1.18 (m, 28 H, CH_2), 0.90-0.84 (m, 6 H, CH_3).

Methyl 11-(4,4"-didecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**5**/10/3): Synthesized from **3**/3 (1.16 g, 3.2 mmol) and 4-decyloxybenzeneboronic acid (2.6 g, 9.4 mmol), using $Pd(OAc)_2$ (15 mg, 0.067 mmol), 2-(di-*tert*-butylphosphino)biphenyl (45 mg, 0.15 mmol),

and KF (2.02 g, 35 mmol). The crude product was purified by column chromatography on silica gel with $CHCl_3/CH_3OH$ = 10/0.1 (V/V) as eluent. Yield: 2.1 g (86 %); colorless waxy solid; transitions/°C: Cr 52 (SmA 52) Iso. ^{1}H-NMR ($CDCl_3$, J/Hz, 200 MHz) δ = 7.54-7.47 (m, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.8, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.8, 1 H, Ar-H), 6.97-6.89 (m, 4 H, Ar-H), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.11 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 4 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.69-3.56 (m, 11 H, OCH_2, OCH_3), 1.88-1.73 (m, 4 H, CH_2), 1.53-1.18 (m, 28 H, CH_2), 0.90-0.84 (m, 6 H, CH_3).

Methyl 14-(4,4"-didecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**5**/10/4): Synthesized from **3**/4 (1.18 g, 2.9 mmol) and 4-decyloxybenzeneboronic acid (2.7 g, 9.7 mmol), using $Pd(OAc)_2$ (20 mg, 0.089 mmol), 2-(di-*tert*-butylphosphino)biphenyl (53 mg, 0.18 mmol), and dry KF (1.55 g, 26.8 mmol). The crude product was purified by column chromatography on silica gel with $CHCl_3/CH_3OH$ (or ethyl acetate/PE) as eluent. Yield 1.1 g (48 %); transition temperatures: Cr 47 (SmA 45) Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.8, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.8, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.8, ^4J(H,H) = 1.8, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.8, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.71-3.56 (m, 15 H, OCH_2, OCH_3), 1.82-1.75 (m, 4 H, CH_2), 1.57-1.31 (m, 4 H, CH_2), 1.27-1.10 (m, 24 H, CH_2), 0.87 (t, ^3J(H,H) = 6.8, 6 H, CH_3).

Methyl 20-(4,4''-didecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoate (**5**/10/6): Synthesized from **3**/6 (0.60 g, 1.2 mmol), $Pd(OAc)_2$ (15 mg, 0.067 mmol), 2-(di-*tert*-butylphosphino)biphenyl (45 mg, 0.15 mmol), 4-decyloxybenzeneboronic acid (1.01 g, 3.6 mmol), KF (2.0 g, 34 mmol) and THF (4 mL). The crude product was purified by column chromatography over silica gel 60 with $CHCl_3$/MeOH = 10/0.3 (V/V) as eluent. Yield 0.97 g (90 %); yellow oil. ^{1}H-NMR ($CDCl_3$, J/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.1, 2 H, OCH_2), 4.14 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.1, 2 H, OCH_2), 3.72 (s, 3 H, OCH_3), 3.71-3.53 (m, 20 H, OCH_2), 1.82-1.75 (m, 4 H, CH_2), 1.50-1.42 (m, 4 H, CH_2), 1.37-1.22 (m, 24 H, CH_2), 0.89-0.85 (m, 6 H, CH_3).

2.1.4.2. Procedure B (debenzylation of methyl ω-(4,4''-dibenzyloxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates, follwed by etherification)

2.1.4.2.1. Methyl ω-(4,4''-dihydroxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates (**6**/*n*):

Methyl 11-(4,4''-dihydroxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**6**/3): The benzyl ether **5**/B/3 (4.5 g, 6.8 mmol), methanol (70 mL) was treated with cyclohexene (60 mL) and *Pearlman's* catalyst (0.9 g) under reflux until the starting benzyl ether had been completely consumed as judged by TLC analysis. The catalyst was filtered off and all volatiles were removed in vacuum, the crude product was purified by column chromatography over silica gel 60, eluent: $CHCl_3/CH_3OH$ = 10/0.5 (V/V); yield 3.1 g (94.5 %); colorless solid; m.p. = 58 °C. ^{1}H-NMR ($CDCl_3$, J/Hz, 200 MHz) δ = 7.48 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.46 (d, ^3J(H,H) = 8.7,

2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.16 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.09 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.18-4.05 (m, 4 H, OCH_2), 3.79-3.53 (m, 13 H, OCH_2, OCH_3).

Methyl 14-(4,4"-dihydroxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**6**/4): **5**/B/4 (3.5 g, 5.0 mmol) dissolved in methanol (70 mL) was treated with cyclohexene (70 mL) and Pearlman's catalyst (0.7 g) under reflux until the starting benzyl ether had been completely consumed as judged by TLC analysis. The catalyst was filtered off and all volatiles were removed in vacuum, the crude product was purified by column chromatography over silica gel with $CHCl_3/CH_3OH$ = 10/0.5 (V/V) as eluent. Yield 2.3 g (88 %); mp. = 68 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.47 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.44 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.32 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.15 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.08 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.88 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.17 (s, 2 H, OCH_2COO), 4.13 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.78-3.57 (m, 17 H, OCH_2, OCH_3).

2.1.4.2.2. Methyl ω-(4,4''-dialkoxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates (**5**/*m*/*n*):

General procedure: Under an Argon atmosphere, the diphenol **6**/*n* (55 mmol) was dissolved in dry acetonitrile (150 mL), K_2CO_3 (34.5 g, 0.25mol), the alkyl bromide (0.1 mol), and Bu_4NI (50 mg) were added, and the mixture was stirred under reflux for 4 - 8 h. Afterwards, the solvent was evaporated in vacuum, and the residue was dissolved in water and diethyl ether, the water phase was extracted by diethyl ether (3 × 100 mL). The combined organic phase was washed with water and brine. After drying over Na_2SO_4 and filtration, the solvent was removed, the crude product was purified by column chromatography over silica gel 60.

Methyl 11-[4,4''-bis(3,7-dimethyloctyloxy)-p-terphenyl-2'-yloxy]-3,6,9-trioxaundecanoate (**5**/10*/3): Synthesized from **6**/3 (0.6 g, 1.2 mmol) and 1-bromo-3,7-dimethyloctane (2.74 g, 12.4 mmol) in dry acetonitrile (50 mL), K_2CO_3 (0.86 g, 6.2 mmol), and $(n\text{-}Bu)_4NI$ (20 mg). Eluent; $CHCl_3/CH_3OH$ = 10/0.1 (V/V); yield 0.7 g (76 %); yellow oil. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.34 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.13 (s, 2 H, OCH_2), 4.04-3.96 (m, 4 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.70-3.58 (m, 11 H, OCH_2, OCH_3), 1.85-1.77 (m, 2 H, CH), 1.67-1.61 (m, 2 H, CH), 1.60-1.43 (m, 4 H, CH_2), 1.37-1.07 (m, 12 H, CH_2), 0.93-0.90 (m, 6 H, CH_3), 0.89-0.70 (m, 12 H, CH_3).

Methyl 14-[4,4''-bis(3,7-dimethyloctyloxy)-p-terphenyl-2'-yloxy]-3,6,9,12-tetraoxatetradecanoate (**5**/10*/4): Synthesized from **6**/4 (0.9 g, 1.7 mmol) and 1-bromo-3,7-dimethyloctane (1.9 g, 8.7 mmol) in dry acetonitrile (50 mL), K_2CO_3 (1.2 g, 8.7 mmol) and $(n\text{-}Bu)_4NI$ (20 mg). Eluent: ethyl acetate/n-hexane = 1/2 (V/V); yield 1.15 g (82 %); yellow oil. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.34 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 4.08-3.94 (m, 4 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.71 (s, 3 H, OCH_3), 3.70-3.55 (m, 12 H, CH_2), 1.88-1.79 (m, 2 H, CH), 1.69-1.61 (m, 2 H, CH), 1.60-1.43 (m, 4 H, CH_2), 1.37-1.07 (m, 12 H, CH_2), 0.96-0.90 (m, 6 H, CH_3), 0.89-0.70 (m, 12 H, CH_3).

Methyl 11-(4,4''-dihexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**5**/16/3): Synthesized from **6**/3 (2.2 g, 4.6 mmol) and 1-bromohexadecane (4.2 g, 14 mmol) in dry acetonitrile (100 mL), K_2CO_3 (3.1 g, 23 mmol) and $(n\text{-}Bu)_4NI$ (20 mg). Eluent: $CHCl_3/CH_3OH$ = 10/0.1 (V/V); yield 3.9 g (92 %); colorless solid; transitions/°C: Cr 67 SmA 73 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.52 (d, ^3J(H,H) = 8.5, 4 H, Ar-H), 7.34 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 6.90 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.4, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 4.00 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.4, 2 H, OCH_2), 3.70 (s, 3 H, OCH_3), 3.66-3.61 (m, 8 H, OCH_2), 1.88-1.72 (m, 4 H, CH_2), 1.52-1.20 (m, 52 H, CH_2), 0.86 (t, ^3J(H,H) = 6.4, 6 H, CH_3).

Methyl 14-(4,4''-dihexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**5**/16/4): Synthesized from **6**/4 (2.3 g, 4.4 mmol) and 1-bromohexadecane (5.3 g, 17 mmol) in dry acetonitrile (100 mL), K_2CO_3 (3.0 g, 22 mmol) and Bu_4NI (20 mg). The crude product was purified by column chromatography on silica gel with ethyl acetate/n-hexane = 2/5 (V/V) as eluent. Yield 1.3 g (75 %); transition temperatures: Cr 62 SmA 65 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2COO), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.76-3.55 (m, 15 H, OCH_2, OCH_3), 1.83-1.73 (m, 4 H, CH_2), 1.57-1.16 (m, 52 H, CH_2), 0.86 (t, ^3J(H,H) = 6.4, 6 H, CH_3).

2.1.4.3. Procedure C (C-C-coupling of 2,5-dichlorophenylacetate to give 4,4''-dialkyloxy-2'-hydroxyterphenyls, followed by etherification)

2.1.4.3.1. 2,5-Dichlorophenylacetate (**10**)

2,5-Dichlorophenol (16.3 g, 0.10 mol) was dissolved in toluene (200 mL). Acetic acid anhydride (12.2 g, 0.12 mol), triethylamine (13.1 g, 0.13 mol) and DMAP (0.12 g, 1 mmol) were added and the mixture was heated to reflux for 8 h. After cooling down, the reaction mixture was extracted with diethyl ether. The organic phase was washed with water, dried over Na_2SO_4 and filtered. The solvent was removed in vacuum and the crude product was purified by vacuum distillation (b.p. = 59 °C, 60 Pa) to afford a colorless oil, 17.5 g (85 mmol), yield 85 %. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.33 (d, ^3J(H,H) = 9.3, 1 H, Ar-H), 7.23-7.10 (m, 2 H, Ar-H), 2.32 (s, 3 H, CH_3).

2.1.4.3.2. 4,4''-Dialkoxy-p-terphenyl-2'-ylacetates (**11**/*m*)

4,4''-Dihexyloxy-p-terphenyl-2'-ylacetate (**11**/6): Synthesized in the similar way as **5**/10/3 from **10** (1.15 g, 5.6 mmol), $Pd(OAc)_2$ (39 mg, 0.17 mmol), 2-(di-*tert*-butylphosphino)biphenyl (104 mg, 0.35 mmol), 4-hexyloxybenzeneboronic acid (3.0 g, 14 mmol), KF (3.0 g, 52 mmol) and THF (6 mL). Eluent: $CHCl_3$; yield 2.5 g (91 %); colorless solid; transitions/°C: Cr (N 66) 72 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.45-7.41 (m, 2 H, Ar-H), 7.36 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.27 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t,

^3J(H,H) = 6.5, 2 H, OCH_2), 2.11 (s, 3 H, CH_3), 1.86-1.72 (m, 4 H, CH_2), 1.52-1.25 (m, 12 H, CH_2), 0.94-0.87 (m, 6 H, CH_3).

4,4''-Didecyloxy-p-terphenyl-2'-ylacetate (**11**/10): Synthesized in the similar way as **5**/10/3 from **10** (0.65 g, 3.17 mmol), $Pd(OAc)_2$ (43 mg, 0.19 mmol), 2-(di-*tert*-butylphosphino)biphenyl (113 mg, 0.38 mmol), 4-decyloxybenzeneboronic acid (2.20 g, 7.93 mmol), KF (3.31 g, 57 mmol) and THF (7 mL). Eluent: $CHCl_3$; yield 1.55 g (81.5 %); colorless solid; transitions/°C: Cr 77 SmA 126 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.47 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.41 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.37 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.28 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.5, 2 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 2.11 (s, 3 H, CH_3), 1.86-1.72 (m, 4 H, CH_2), 1.52-1.25 (m, 28 H, CH_2), 0.94-0.87 (m, 6 H, CH_3).

2.1.4.3.3. 4,4''-Dialkyloxy-p-terphenyl-2'-ols (**12**/*m*)

General procedure: The appropriate 4,4''-Dialkoxy-p-terphenyl-2'-ylacetate **(11**/*m***)** (5 mmol) and NaOH (aq. 1M, 50 mL) were heated to reflux for 16 h. After cooling to r.t., the reaction mixture was acidified by HCl (conc.). Then the mixture was extracted with diethyl ether (3 × 50 mL). The combined organic phase was washed with water (2 × 50 mL) and brine (50 mL). After drying over Na_2SO_4 and filtration, the solvent was evaporated under vacuum and the crude product was purified by column chromatography over silica gel.

4,4''-Dihexyloxy-p-terphenyl-2'-ol (**12**/6): Synthesized from **11**/6 (2.5 g, 5.1 mmol), NaOH (aq. 1M, 50 mL) and HCl (Conc. 10 mL). Eluent: $CHCl_3$; yield 1.8 g (79 %); colorless solid; transitions/°C: Cr 110 SmA 140 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.53 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.39 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.26-7.22 (m, 1 H, Ar-H), 7.19-7.13 (m, 2 H, Ar-H), 7.00 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 5.22 (s, 1 H, OH), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 1.86-1.72 (m, 4 H, CH_2), 1.46-1.24 (m, 12 H, CH_2), 0.90 (t, ^3J(H,H) = 6.6, 6 H, CH_3).

4,4''-Didecyloxy-p-terphenyl-2'-ol (**12**/10): Synthesized from **11**/10 (3.6 g, 6.0 mmol), NaOH (aq. 1M, 50 mL) and HCl (Conc. 10 mL). Eluent: $CHCl_3$; yield 2.5 g (75 %); colorless solid; transitions/°C: Cr 90 SmA 158 Iso. ^{1}H-NMR (acetone-d_6, *J*/Hz, 200 MHz) δ = 8.25 (s, 1 H, OH), 7.63-7.52 (m, 4 H, Ar-H), 7.38-7.25 (m, 1 H, Ar-H), 7.22-7.08 (m, 2 H, Ar-H), 7.06-6.88 (m, 4 H, Ar-H), 4.10-3.92 (m, 4 H, OCH_2), 1.86-1.65 (m, 4 H, CH_2), 1.61-1.10 (m, 28 H, CH_2), 0.94-0.87 (m, 6 H, CH_3).

2.1.4.3.4. Methyl (or propyl) ω-(4,4''-dialkoxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates (**5**/*m*/*n*)

General procedure: The appropriate 4,4''-dialkoxy-p-terphenyl-2'-ol (**12**/*m*) (1 mmol) was dissolved in dry acetonitrile (30 mL), K_2CO_3 (0.35 g, 2.5 mmol), the tosylate **2**/*n* (1.5 mmol), and Bu_4NI (10 mg) were added, and the mixture was stirred under reflux for 4-8h. Afterwards, the solvent was evaporated in vacuum, and the residue was dissolved in water and diethyl ether, the water phase was extracted by diethyl ether (3 × 50 mL). The combined organic phase was washed with water (3 × 50 mL) and brine(50 mL). After drying over Na_2SO_4 and filtration, the solvent was removed, the crude product was purified by column chromatography on silica gel 60.

Methyl 14-(4,4''-dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**5**/6/4): See ref. [3].

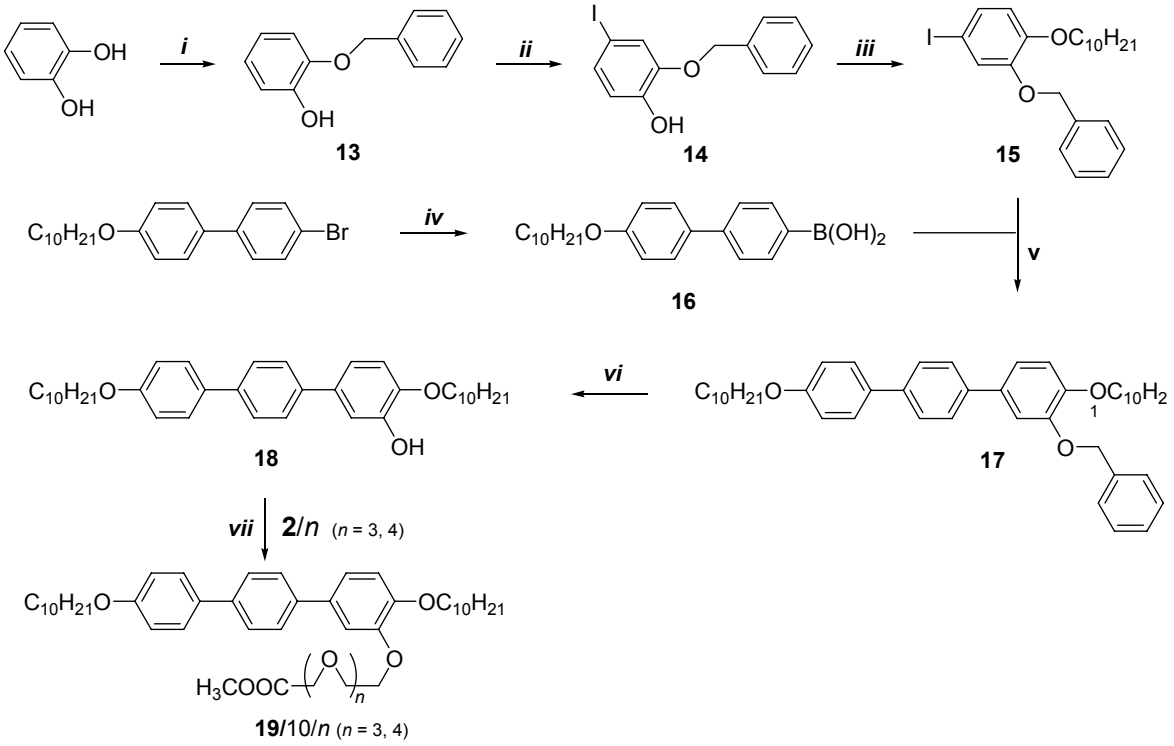
Methyl 17-(4,4''-dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15-pentaoxaheptadecanoate (**5**/6/5), Synthesized form **12**/6 (1.0 g, 2.2 mmol), dry acetonitrile (60 mL), K_2CO_3 (1.2 g, 8.7 mmol), **2**/5 (1.5 g, 3.3 mmol) and Bu_4NI (20 mg). Eluent: ethyl acetate/n-hexane = 1/2 (V/V); yield 1.2 g (74 %); yellow oil. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.34 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.2-3.6 (m, 29 H, OCH_2, OCH_3), 1.81-1.75 (m, 4 H, CH_2), 1.50-1.43 (m, 4 H, CH_2), 1.39-1.22 (m, 4 H, CH_2), 0.92-0.87 (m, 6 H, CH_3).

Methyl 20-(4,4''-dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoate (**5**/6/6): Synthesized form **12**/6 (1.0 g, 2.2 mmol), dry acetonitrile (60 mL), K_2CO_3 (1.2 g, 8.7 mmol), **2**/6 (1.7 g, 3.3 mmol) and Bu_4NI (20 mg). Eluent: ethyl acetate/n-hexane = 1/2 (V/V); yield 1.1 g (64 %); yellow oil. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.1, 2 H, OCH_2), 4.13 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.1, 2 H, OCH_2), 3.72 (s, 3 H, OCH_3), 3.71-3.35 (m, 20 H, OCH_2), 1.81-1.75 (m, 4 H, CH_2), 1.50-1.43 (m, 4 H, CH_2), 1.39-1.22 (m, 4 H, CH_2), 0.92-0.87 (m, 6 H, CH_3).

Methyl 14-(4,4''-dioctyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**5**/8/4). See ref. [3].

Propyl 5-(4,4''-didecyloxy-p-terphenyl-2'-yloxy)-3-oxapentanoate (**5**/10/1): Synthesized form **12**/10 (0.40 g, 0.72 mmol), dry acetonitrile (30 mL), K_2CO_3 (0.25 g, 1.8 mmol), **2**/1 (0.35 g, 1.1 mmol) and Bu_4NI (10 mg). Eluent: ethyl acetate/n-hexane = 1/3 (V/V); yield 0.35 g (69 %); colorless solid; transitions/°C: Cr 63 SmA 73 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.39 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.36 (d, ^3J(H,H) = 7.7, 1 H, Ar-H), 7.20 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.00 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.20-4.17 (m, 2 H, OCH_2), 4.02-3.95 (m, 6 H, OCH_2), 3.87 (t, ^3J(H,H) = 4.8, 2 H, OCH_2), 3.78-3.75 (m, 2 H, OCH_2), 1.84-1.75 (m, 4 H, CH_2), 1.69-1.60 (m, 2H, CH_2), 1.50-1.42 (m, 4 H, CH_2), 1.40-1.20 (m, 24 H, CH_2), 0.96-0.82 (m, 9 H, CH_3).

2.2. Synthesis of the methyl ω-(4,4''-didecyloxy-p-terphenyl-3-yloxy)[oligo(oxyethylene)-1-yl]acetates (19/10/*n*)



Scheme S3. Synthesis of the methyl ω-(4,4''-didecyloxy-p-terphenyl-3-yloxy)[oligo(oxyethylene)-1-yl]acetates (**19**/10/*n*). *Reagents and conditions*: ***i***) methanol, NaOH, benzyl chloride, reflux, 2 h; ***ii***) methanol, NaI, NaOH, NaOCl (4 %), 0-3°C; ***iii***) 1-bromodecane, K_2CO_3, $(n\text{-}Bu)_4NI$, CH_3CN, reflux 8 h; ***iv***) 1. THF, n-BuLi, -78°C; 2. $B(OMe)_3$, -78°C; 3. HCl (10%); ***v***) $Pd(PPh_3)_4$, DME, $NaHCO_3$ (saturated aq.), reflux, 8 h; ***vi***) *Pearlman's* catalyst (palladium hydroxide on carbon), cyclohexene, methanol, reflux 8 h; ***vii***) K_2CO_3, $(n\text{-}Bu)_4NI$, CH_3CN, reflux 8 h.

2.2.1. 2-Benzyloxyphenol (**13**)

Catechol (33 g, 0.30 mol) was dissolved in dry methanol (300 mL), NaOH (12 g, 0.30 mol) dissolved in dry methanol (100 mL) was added dropwise at 0-3 °C, after that, benzyl chloride (38 g, 0.30 mol) dissolved in dry methanol (100 mL) was added dropwise, the reaction mixture was heated to reflux for 2 h, cooled, acidified with concentrated HCl (ca. 10 mL), extracted with diethyl ether (3 × 150 mL) and washed with water (3 × 150 mL) and brine (100 mL). After drying over Na_2SO_4 and filtration, the solvent was removed in vacuum and the crude product was purified with column chromatography on silica gel with $CHCl_3$ as eluent and vacuum distillation (b.p. = 108-120 °C at 5Pa) which afforded a colorless oil, 19.3 g (96.4 mmol), yield 32 %. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.45-7.32 (m, 5 H, Ar-H), 6.98-6.80 (m, 4 H, Ar-H), 5.68 (s, 1 H, OH), 5.07 (s, 2 H, OCH_2).

2.2.2. 2-Benzyloxy-4-iodophenol (**14**)

13 (19 g, 95 mmol) was dissolved in methanol (500 mL), NaI (14.3 g, 95 mmol) and NaOH (3.8 g, 95 mmol) was added, and the solution was cooled to 0 °C. Aqueous sodium hypochlorite (4 %, 0.1mol) was added dropwise over 75min at 0-3 °C, as each drop hit the solution, a red color appeared and faded instantly. The resulting mixture was stirred for 1 h at 0-2 °C, and then

aqueous sodium thiosulfate solution (10 %, 20 mL) was added, the mixture was adjusted to pH = 7 by using 5 % aqueous HCl and the product was extracted by diethyl ether (3 × 100 mL). The combined organic phases were washed with water (3 × 100 mL) and brine (100 mL). After drying over Na_2SO_4 and filtration, the solvent was removed in vacuum and the residue was purified by column chromatography ($CHCl_3$ as eluent) and recrystalization (diethyl ether/n-hexane) which afforded a colorless crystal, 10.9 g (33.4 mmol), yield 35 %; m.p. = 70 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.43-7.36 (m, 5 H, Ar-H), 7.20 (d, ^4J(H,H) = 2.1, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 8.1, ^4J(H,H) = 2.1 1 H, Ar-H), 6.69 (d, ^3J(H,H) = 8.1, 1 H, Ar-H), 5.60 (s, 1 H, OH), 5.05 (s, 2 H, OCH_2).

2.2.3. 1-Benzyloxy-2-decyloxy-5-iodobenzene (**15**)

Under an Argon atmosphere, **14** (8.0 g, 25 mmol) was dissolved in dry acetonitrile (100 mL), K_2CO_3 (8.7 g, 63 mmol), the 1-bromodecane (5.7 g, 26 mmol), and Bu_4NI (20 mg) were added, and the mixture was stirred under reflux for 4-8h. Afterwards, the solvent was evaporated in vacuum, and the residue was dissolved in water (50 mL) and diethyl ether (50 mL), the water phase was extracted by diethyl ether (3 × 50 mL). The combined organic phase was washed with water (2 × 50 mL) and brine (50 mL). After drying over Na_2SO_4 and filtration, the solvent was removed, the crude product was purified by column chromatography on silica gel with ethyl acetate/PE (1/100 V/V) as eluent which afforded a colorless oil, 10.4 g (22.3 mmol), yield 89.6 %; m.p. = 40 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.43-7.18 (m, 7 H, Ar-H), 6.63 (d, ^3J(H,H) = 8.7, 1 H, Ar-H), 5.06 (s, 2 H, OCH_2), 3.96 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 1.82-1.71 (m, 2 H, CH_2), 1.51-1.25 (m, 14 H, CH_2), 0.86 (t, ^3J(H,H) = 6.6, 3 H, CH_3).

2.2.4. 3-Benzyloxy-4,4''-didecyloxy-p-terphenyl (**17**)

In a two-necked flask equipped with a reflux condenser and a magnetic stirring bar, $Pd(PPh_3)_4$ (0.1 g, 0.09 mmol) was added under an argon atmosphere to a mixture consisting of the **15** (0.80 g, 1.7 mmol), 4'-decyloxybiphenyl-4-boronic acid (**16**) (0.67 g, 1.9 mmol), dimethoxyethane (30 mL), and saturated $NaHCO_3/H_2O$ solution (30 mL). The resulting mixture was stirred at reflux temperature for 5 h. After cooling, the solvent was evaporated and the residue was dissolved in diethyl ether. The organic phase was separated, and was washed with water. After drying over Na_2SO_4 and filtration, the solvent was removed, the crude product was purified by column chromatography on silica gel 60 with $CHCl_3$ as eluent which afforded 0.72 g (65 %); colorless solid; m.p. = 90 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.65-7.25 (m, 11 H, Ar-H), 7.22-7.16 (m, 2 H, Ar-H), 6.98-6.95 (m, 3 H, Ar-H), 5.19 (s, 2 H, OCH_2), 4.06 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 1.88-1,76 (m, 4 H, CH_2), 1.58-1.27 (m, 28 H, CH_2), 0.88 (t, ^3J(H,H) = 6.4, 6 H, CH_3).

2.2.5. 4,4''-Didecyloxy-p-terphenyl-3-ol (**18**)

17 (1.0 g, 1.5 mmol), dissolved in methanol (35 mL) was treated with cyclohexene (30 mL) and *Pearlman's* catalyst (30 mg, 20 %) under reflux until the starting benzyl ether had been completely consumed as judged by TLC analysis. The catalyst was filtered off and all volatiles were removed in vacuum, the crude product was purified by column chromatography over silica gel 60 with $CHCl_3$ as eluent which afforded 0.66 g (79 %); colorless solid; transitions/°C: Cr 132 SmA 162 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.58 (s, 4 H, Ar-H), 7.54 (d, ^3J(H,H) = 8.5,

2 H, Ar-H), 7.22 (d, ^4J(H,H) = 2.3, 1 H, Ar-H), 7.09 (dd, ^3J(H,H) = 8.5, ^4J(H,H) = 2.3, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.5, 1 H, Ar-H), 5.68 (s, 1 H, OH), 4.07 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 1.86-1,76 (m, 4 H, CH_2), 1.50-1.43 (m, 4 H, CH_2), 1.42-1.20 (m, 24 H, CH_2), 0.88 (t, ^3J(H,H) = 6.7, 6 H, CH_3).

2.2.6. Methyl ω-(4,4''-didecyloxy-p-terphenyl-3-yloxy)[oligo- (oxyethylene)ylacetates (**19**/10/*n*)

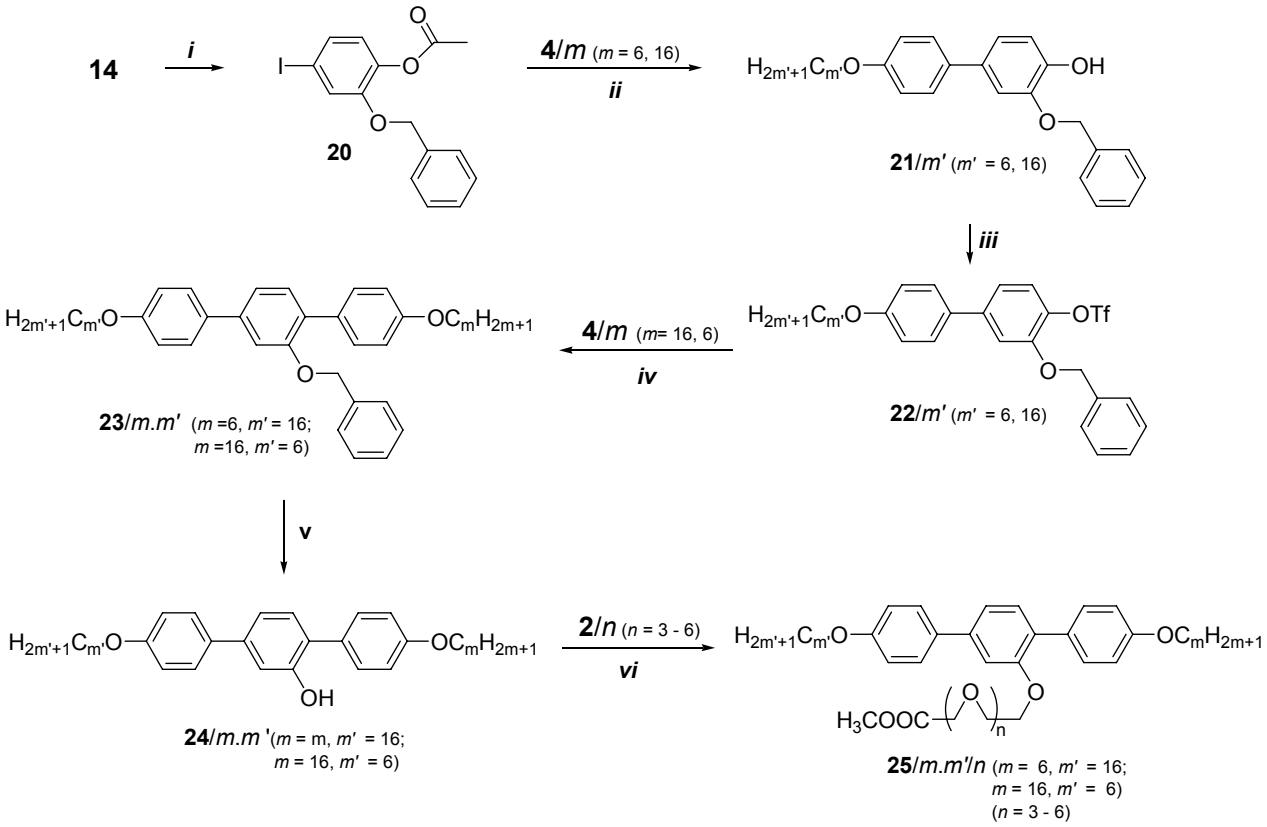
General procedure:

Under an Argon atmosphere, **18** (2 mmol) was dissolved in dry acetonitrile (50 mL), K_2CO_3 (0.7 g, 5 mmol), the tosylate **2**/*n* (3 mmol), and Bu_4NI (10 mg) were added, and the mixture was stirred under reflux for 4-8 h. Afterwards, the solvent was evaporated in vacuum, and the residue was dissolved in water and diethyl ether, the water phase was extracted by diethyl ether (3 × 100 mL). The combined organic phase was washed with water and brine. After drying over Na_2SO_4 and filtration, the solvent was removed, the crude product was purified by column chromatography on silica gel 60.

Methyl 11-(4,4''-didecyloxy-p-terphenyl-3-yloxy)-3,6,9-trioxaundecanoate (**19**/10/3): Synthesized from **18** (0.6 g, 1.1 mmol), **2**/3 (0.64 g, 1.7 mmol), dry acetonitrile (30 mL), K_2CO_3 (0.39 g, 2.8 mmol) and $(n\text{-}Bu)_4NI$ (10 mg). Eluent: ethyl acetate/n-hexane = 2/5 (V/V); yield 0.7 g (83.5 %); colorless solid; transitions/°C: Cr 72 SmA 95 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.57 (s, 4 H, Ar-H), 7.53 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.24-7.14 (m, 2 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.93 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 4.24 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.13 (s, 2 H, OCH_2), 4.02 (t, ^3J(H,H) = 6.2, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.3, 2 H, OCH_2), 3.89 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.78-3.64 (m, 11 H, OCH_2, OCH_3), 1.85-1,72 (m, 4 H, CH_2), 1.45-1.26 (m, 28 H, CH_2), 0.87 (t, ^3J(H,H) = 6.5, 6 H, CH_3).

Methyl 14-(4,4''-didecyloxy-p-terphenyl-3-yloxy)-3,6,9,12-tetraoxa-tetradecanoate (**19**/10/4): Synthesized from **18** (0.6 g, 1.1 mmol), **2**/4 (0.71 g, 1.7 mmol), dry acetonitrile (30 mL), K_2CO_3 (0.39 g, 2.8 mmol) and $(n\text{-}Bu)_4NI$ (10 mg). Eluent: ethyl acetate/n-hexane = 2/5 (V/V); yield 0.7 g (79 %); colorless solid; transitions/°C: Cr 60 SmA 86 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.57 (s, 4 H, Ar-H), 7.53 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.24-7.14 (m, 2 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.93 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 4.24 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.13 (s, 2 H, OCH_2), 4.02 (t, ^3J(H,H) = 6.1, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.1, 2 H, OCH_2), 3.83 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.80-3.63 (m, 15 H, OCH_2, OCH_3), 1.85-1,71 (m, 4 H, CH_2), 1.45-1.15 (m, 28 H, CH_2), 0.87 (t, ^3J(H,H) = 6.6, 6 H, CH_3).

2.3. Synthesis of the methyl ω-(4, 4''-dialkyloxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates with different alkyl chain length



Scheme S4. Synthesis of the methyl ω-(4, 4''-alkyloxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates with different alkyl chain lengths. *Reagents and conditions*: ***i***) DMAP, $(CH_3CO)_2O$, TEA, toluene, reflux, 8 h; ***ii***) $Pd(PPh_3)_4$, DME, $NaHCO_3$ (saturated aq.), reflux, 8 h; ***iii***) pyridine, $(CF_3SO_2)_2O$, 0 °C 10 min, r.t. 24 h; ***iv***) $Pd(PPh_3)_4$, toluene, Na_2CO_3, reflux, 8 h; ***v***) *Pearlman's* catalyst (palladium hydroxide on carbon), cyclohexene, methanol, reflux 8 h; ***vi***) K_2CO_3, $(n\text{-}Bu)_4NI$, CH_3CN, reflux 8 h.

2.3.1. 2-Benzyloxy-4-iodophenyl acetate (**20**)

14 (19.5 g, 59.8 mmol) was dissolved in toluene (400 mL), acetic anhydride (9.15 g, 89.7 mmol), triethylamine (9.66 g, 95.7 mmol) and DMAP (0.12 g, 1 mmol) were added and the mixture was heated to reflux for 8 h. After cooling down, the reaction mixture was extracted with diethyl ether (3 × 100 mL) and the combined organic phase was washed with water (2 × 100 mL). After drying over Na_2SO_4 and filtration, the solvent was removed in vacuum and the crude product was purified by column chromatography on silica gel 60 with $CHCl_3$ as eluent which afforded a colorless solid, 18.4 g (68.6 mmol), yield 84 %; m.p. = 90 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.37-7.26 (m, 7 H, Ar-H), 6.77 (d, ^3J(H,H) = 8.3, 1 H, Ar-H), 5.03 (s, 2 H, OCH_2), 2.23 (s, 3 H, CH_3).

2.3.2. 3-Benzyloxy-4'-alkoxybiphenyl-4-ols (**21**/*m'*):

General procedure: In a two-necked flask equipped with a reflux condenser and a magnetic stirring bar, $Pd(PPh_3)_4$ (0.2 g, 5mol %) was added under an argon atmosphere to a mixture consisting **20** (1.25 g, 3.4 mmol), the boronic acid **4**/*m* (3.5 mmol), dimethoxyethane (30 mL), and saturated $NaHCO_3/H_2O$ solution (20 mL). The mixture was stirred at reflux temperature for 8 h. After cooling, the solvent was evaporated and the residue was dissolved in diethyl ether (150 mL). The organic phase was separated, and was washed with water (3 × 50 mL). After drying over Na_2SO_4 and filtration, the solvent was removed, the crude product was purified by column chromatography on silica gel 60 and repeated crystallization from appropriate solvent (in most cases, the acetate group was removed after the coupling reaction, if not the case, hydrolysis was carried out by using NaOH in ethanol).

3-Benzyloxy-4'-hexyloxybiphenyl-4-ol (**21**/6): Synthesized from **20** (11.0 g, 30 mmol), **4**/6 (7.33, 33 mmol), $Pd(PPh_3)_4$ (1.73g, 1.5 mmol), DME (85 mL) and saturated $NaHCO_3/H_2O$ (85 mL). Eluent: $CHCl_3$; repeated crystallization from acetone; yield 8.2 g (73 %), colorless solid; m.p. = 122 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.45-7.34 (m, 7 H, Ar-H), 7.10 (d, ^4J(H,H) = 2.1, 1 H, Ar-H), 7.05 (dd, ^3J(H,H) = 8.1, ^4J(H,H) = 2.1, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.3, 1 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 5.60 (s, 1 H, OH), 5.15 (s, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 1.82-1.74 (m, 2 H, CH_2), 1.58-1.40 (m, 2 H, CH_2), 1.39-1.25 (m, 4 H, CH_2), 0.91 (t, ^3J(H,H) = 6.5, 3 H, CH_3).

3-Benzyloxy-4'-hexadecyloxybiphenyl-4-ol (**21**/16): Synthesized from **20** (4.5 g, 12.2 mmol), **4**/16 (4.9, 13.5 mmol), $Pd(PPh_3)_4$ (0.7 g, 0.61 mmol), DME (60 mL) and saturated $NaHCO_3/H_2O$ (60 mL). Eluent $CHCl_3$; repeated crystallization from acetone; yield 3.75 g (60 %); colorless solid; m.p. = 115 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.45-7.33 (m, 7 H, Ar-H), 7.10 (d, ^4J(H,H) = 1.9, 1 H, Ar-H), 7.05 (dd, ^3J(H,H) = 8.3, ^4J(H,H) = 1.9, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.3, 1 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 5.60 (s, 1 H, OH), 5.15 (s, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 1.81-1.74 (m, 2 H, CH_2), 1.53-1.41 (m, 2 H, CH_2), 1.33-1.20 (m, 24 H, CH_2), 0.87 (t, ^3J(H,H) = 6.6, 3 H, CH_3).

2.3.3. 3-Benzyloxy-4'-alkoxybiphenyl-4-yltrifluoromethanesulfonates (**22**/*m'*)

General procedure: To a solution of the phenol **21**/*m'* (5.0 mmol) in dry pyridine (3 mL) at 0 °C, trifluoromethanesulfonic anhydride (1.6 g, 5.5 mmol) was slowly added. The resulting mixture was stirred at 0 °C for 5min, then allowed to warm to r.t. and stirred at this temperature for 24 h. The resulting mixture was poured into water and extracted with diethyl ether (3 × 50 mL). The diethyl ether extract was washed sequentially with water (2 × 50 mL), 10 % aq HCl (30 mL), water (2 × 50 mL) and brine (30 mL), after dried over Na_2SO_4 and concentrated. The crude product was purified by flash column chromatography on silica gel 60 with $CHCl_3$ as eluent.

3-benzyloxy-4'-hexyloxybiphenyl-4-yltrifluoromethanesulfonate (**22**/6): Synthesized from **21**/6 (7.1 g, 18.9 mmol), pyridine (10 mL) and trifluoromethanesulfonic anhydride (5.9 g, 21.0 mmol). Eluent: $CHCl_3$; yield 9.5 g (99 %); yellow oil. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.47-7.45 (m, 2 H, Ar-H), 7.40-7.37 (m, 4 H, Ar-H), 7.35-7.30 (m, 1 H, Ar-H), 7.24 (d, ^3J(H,H) = 8.3, 1 H, Ar-H), 7.19 (d, ^4J(H,H) = 2.1, 1 H, Ar-H), 7.10 (dd, ^3J(H,H) = 8.3, ^4J(H,H) = 2.1, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 5.21 (s, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2),

1.82-1.75 (m, 2 H, CH_2), 1.50-1.43 (m, 2 H, CH_2), 1.38-1.24 (m, 4 H, CH_2), 0.90 (t, ^3J(H,H) = 6.5, 3 H, CH_3). ^{19}F NMR ($CDCl_3$, 188 MHz) δ = -74.44 (s, 3F, CF_3).

3-benzyloxy-4'-hexadecyloxybiphenyl-4-yltrifluoromethanesulfonate (**22**/16): Synthesized from **21**/16 (4.6 g, 8.9 mmol), dry pyridine (10 mL) and trifluoromethanesulfonic anhydride (2.8 g, 9.8 mmol). Eluent: $CHCl_3$; yield 5.7 g (99 %); yellow solid; m.p. = 41 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.48-7.34 (m, 7 H, Ar-H), 7.24 (d, ^3J(H,H) = 8.3, 1 H, Ar-H), 7.19 (d, ^4J(H,H) = 1.9, 1 H, Ar-H), 7.10 (dd, ^3J(H,H) = 8.3, ^4J(H,H) = 1.9, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 5.21 (s, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.4, 2 H, OCH_2), 1.79-1.74 (m, 2 H, CH_2), 1.53-1.24 (m, 26 H, CH_2), 0.86 (t, ^3J(H,H) = 6.5, 3 H, CH_3). ^{19}F NMR ($CDCl_3$, 188 MHz) δ = -74.44 (s, 3 F, CF_3).

2.3.4. 2'-Benzyloxy-4-hexadecyloxy-4''-hexyloxy-p-terphenyl (**23**/16.6) *and 2'-benzyloxy- 4''-hexadecyloxy-4-hexyloxy-p-terphenyl* (**23**/6.16):

General procedure: In a two-necked flask equipped with a reflux condenser and a magnetic stirring bar, $Pd(PPh_3)_4$ (0.37 g, 0.32 mmol) was added under an argon atmosphere to a mixture consisting the appropriate trifluoromethanesulfonate **22**/*m* (6.40 mmol), the boronic acid **4**/*m* (12.8 mmol), dry toluene (45 mL), and Na_2CO_3 (dry solid, 1.33 g, 12.5 mmol). The mixture was stirred at reflux temperature for 8 h. After cooling, the solvent was evaporated and the residue was dissolved in diethyl ether (300 mL) and water (50 mL). The organic phase was separated, and was washed with water (3 × 50 mL). After drying over Na_2SO_4 and filtration, the solvent was removed, the crude product was purified by column chromatography over silica gel.

2'-Benzyloxy-4-hexyloxy-4''-hexadecyloxy-p-terphenyl (**23**/6.16): Synthesized from **22**/16 (1.08 g, 1.67 mmol), dry toluene (10 mL), **4**/6 (0.74 g, 3.33 mmol), $Pd(PPh_3)_4$ (96 mg, 0.08 mmol) and Na_2CO_3 (0.35 g, 3.30 mmol). Eluent ethyl acetate/n-hexane = 1/20 V/V; yield 0.74 g (65.5 %); colorless solid; m.p. = 88 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.55 (d, ^3J(H,H) = 8.5, 2 H, Ar-H), 7.50 (d, ^3J(H,H) = 8.5, 2 H, Ar-H), 7.40-7.18 (m, 8 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.3, 2 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.5, 2 H, Ar-H), 5.12 (s, 2 H, CH_2), 3.99 (t, ^3J(H,H) = 6.9, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.9, 2 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.55-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.92-0.82 (m, 6 H, CH_3)

2'-Benzyloxy 4-hexadecyloxy-4''-hexyloxy-p-terphenyl (**23**/16.6): Synthesized from **22**/6 (3.25 g, 6.40 mmol), dry toluene (45 mL), **4**/16 (4.63 g, 12.8 mmol), $Pd(PPh_3)_4$ (0.37 g, 0.32 mmol) and Na_2CO_3 (1.33 g, 12.5 mmol). Eluent ethyl acetate/n-hexane = 1/20 V/V; yield 2.56 g (59 %); colorless solid; m.p. = 90 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.54 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.40-7.18 (m, 8 H, Ar-H), 6.95 (d, ^3J(H,H) = 9.0, 2 H, Ar-H), 6.93 (d, ^3J(H,H) = 9.0, 2 H, Ar-H), 5.12 (s, 2 H, CH_2), 3.99 (t, ^3J(H,H) = 6.7, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.7, 2 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.87 (t, ^3J(H,H) = 6.8, 6 H, CH_3).

2.3.5. 4''-Hexadecyloxy-4-hexyloxy-p-terphenyl-2'-ol (**24**/6.16) *and 4-hexadecyloxy-4''-hexyloxy-p-terphenyl-2'-ol* (**24**/16.6):

General procedure: The benzyl ether **23**/6.16 or **23**/16.6 (3.79 mmol) dissolved in methanol (60 mL) was treated with cyclohexene (60 mL) and *Pearlman's* catalyst (0.5 g, 20 %) under reflux until the starting benzyl ether had been completely consumed as judged by TLC analysis. The

catalyst was filtered off and all volatiles were removed in vacuum, the crude product was purified by column chromatography over silica gel 60 with $CHCl_3$ as eluent.

4''-Hexadecyloxy-4-hexyloxy-p-terphenyl-2'-ol (**24**/6.16): Synthesized from **23**/6.16 (2.99 g, 4.42 mmol), *Pearlman's* catalyst (500 mg), methanol (50 mL) and cyclohexene (50 mL). Eluent: $CHCl_3$; yield 1.8 g (68 %); colorless solid; transitions/°C: Cr 89 SmA 159 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 500 MHz) δ = 7.53 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.40 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.24 (d, ^3J(H,H) = 8.5, 1 H, Ar-H), 7.18-7.15 (m, 2 H, Ar-H), 7.01 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 5.24 (bs, 1 H, OH), 4.00 (t, ^3J(H,H) = 6.3, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.2, 2 H, OCH_2), 1.84-1.75 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.92-0.82 (m, 6 H, CH_3).

4-Hexadecyloxy-4''-hexyloxy-p-terphenyl-2'-ol (**24**/16.6): Synthesized from **23**/16.6 (2.56 g, 3.79 mmol), *Pearlman's* catalyst (500 mg), dry methanol (60 mL) and cyclohexene (60 mL). Eluent: $CHCl_3$; yield 1.0 g (45 %); colorless solid; transitions/°C: 100 SmA 150 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.53 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.39 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.24 (d, ^3J(H,H) = 8.3, 1 H, Ar-H), 7.17-7.13 (m, 2 H, Ar-H), 7.00 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 5.22 (s, 1 H, OH), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.92-0.82 (m, 6 H, CH_3).

2.3.6. Methyl ω-(4-hexadecyloxy-4''-hexyloxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-1-yl]acetates (**25**/16.6/*n*) *and methyl ω-(4-hexyloxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)[oligo(oxyethylene)-yl]acetates* (**25**/6.16/*n*)

General procedure: Under an Argon atmosphere, the **24**/6.16 or **24**/16.6 (1 mmol) was dissolved in dry acetonitrile (30 mL), K_2CO_3 (0.35 g, 2.5 mmol), the tosylate **2**/***n*** (1.5 mmol), and Bu_4NI (10 mg) were added, and the mixture was stirred under reflux for 4-8 h. Afterwards, the solvent was evaporated in vacuum, and the residue was dissolved in water and diethyl ether, the aqueous phase was extracted by diethyl ether (3 × 50 mL). The combined organic phase was washed with water and brine. After drying over Na_2SO_4 and filtration, the solvent was removed, the crude product was purified by column chromatography on silica gel.

Methyl 11-(4-hexyloxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**25**/6.16/3): Synthesized from **24**/6.16 (0.86 g, 1.47 mmol), **2**/3 (1.11 g, 2.94 mmol), dry acetonitrile (50 mL), K_2CO_3 (0.51 g, 3.68 mmol) and $(n\text{-}Bu)_4NI$ (10 mg). Eluent: ethyl acetate/n-hexane = 1/2 (V/V); yield 0.48 g (41 %); colorless solid; m.p. = 56 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.9, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.11 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.71-3.54 (m, 11 H, OCH_2, OCH_3), 1.83-1.75 (m, 4 H, CH_2), 1.51-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.92-0.82 (m, 6 H, CH_3).

Methyl 14-(4-hexyloxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetra-decanoate (**25**/6.16/4): Synthesized from **24**/6.16 (0.89 g, 1.52 mmol), **2**/4 (1.28 g, 3.04 mmol), dry acetonitrile (50 mL), K_2CO_3 (0.53 g, 3.80 mmol) and $(n\text{-}Bu)_4NI$ (20 mg); Eluent: ethyl acetate/n-hexane = 1/2 (V/V); yield 1.10 g (87 %); colorless solid; m.p. = 51 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz,

400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.5, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.71-3.58 (m, 15 H, OCH_2, OCH_3), 1.83-1.75 (m, 4 H, CH_2), 1.51-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.92-0.84 (m, 6 H, CH_3).

Methyl 20-(4-hexyloxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxa-eicosanoate (**25**/6.16/6): Synthesized from **24**/6.16 (0.80 g, 1.36 mmol), **2**/6 (1.39 g, 2.73 mmol), dry acetonitrile (50 mL), K_2CO_3 (0.47 g, 3.40 mmol) and $(n\text{-}Bu)_4NI$ (20 mg). Eluent: ethyl acetate; yield 1.10 g (88 %); colorless solid; m.p. = 41 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.13 (s, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.72-3.58 (m, 23 H, OCH_2, OCH_3), 1.81-1.75 (m, 4 H, CH_2), 1.51-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.92-0.84 (m, 6 H, CH_3).

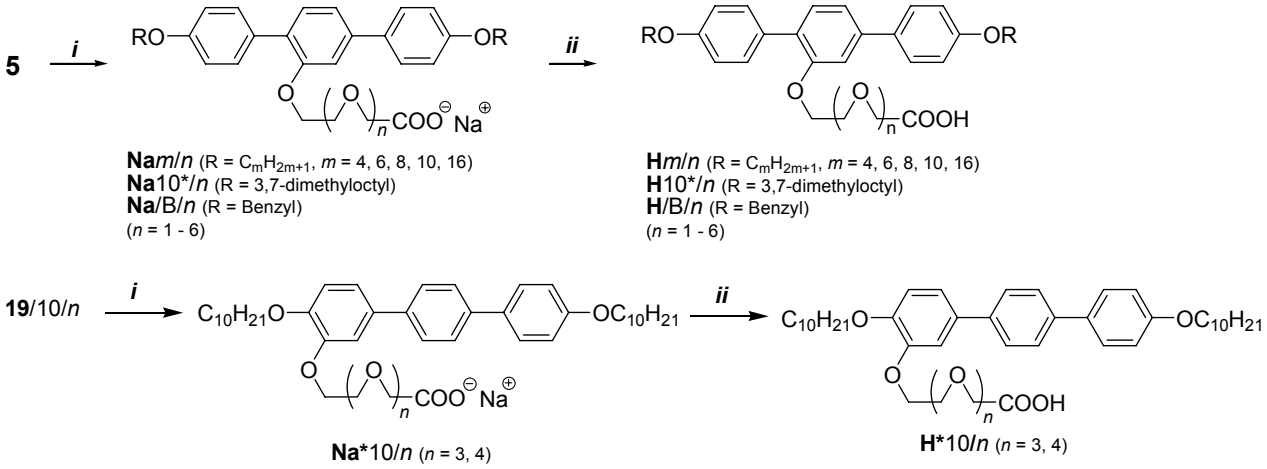
Methyl 11-(4-hexadecyloxy-4''-hexyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**25**/16.6/3): Synthesized from **24**/16.6 (0.71 g, 1.21 mmol), **2**/3 (0.91 g, 2.42 mmol), dry acetonitrile (50 mL), K_2CO_3 (0.42 g, 3.03 mmol) and $(n\text{-}Bu)_4NI$ (20 mg). Eluent ethyl acetate/n-hexane = 1/2 (V/V); yield 0.75 g (78.5 %); colorless solid; transitions/°C: Cr 35 SmA 42 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.3, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.3, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.75-3.58 (m, 11 H, OCH_2, OCH_3), 1.83-1.75 (m, 4 H, CH_2), 1.53-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.92-0.84 (m, 6 H, CH_3).

*Methyl 14-(4-hexadecyloxy-4''-hexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetra-decan**oate*** (**25**/16.6/4): Synthesized from **24**/16.6 (0.86 g, 1.47 mmol), **2**/4 (1.23 g, 2.94 mmol), acetonitrile (50 mL), K_2CO_3 (0.51 g, 3.68 mmol) and $(n\text{-}Bu)_4NI$ (20 mg). Eluent: ethyl acetate/n-hexane = 1/2 (V/V); yield 1.00 g (82 %); colorless solid; transitions/°C: Cr 30 SmA 35 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.12 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.3, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.2, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.71-3.55 (m, 15 H, OCH_2, OCH_3), 1.83-1.75 (m, 4 H, CH_2), 1.51-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.92-0.84 (m, 6 H, CH_3).

Methyl 20-(4-hexadecyloxy-4''-hexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxa-eicosanoate (**25**/16.6/6): Synthesized from **24**/16.6 (0.69 g, 1.18 mmol), **2**/6 (1.20 g, 2.36 mmol), acetonitrile (50 mL), K_2CO_3 (0.41 g, 2.95 mmol) and $(n\text{-}Bu)_4NI$ (20 mg). Eluent: ethyl acetate/n-hexane = 2/1 (V/V); yield 1.05 g (96.5 %); colorless solid; transitions/°C: Cr 25 SmA 29 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.0, 2 H,

OCH_2), 4.13 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.3, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.2, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.72-3.55 (m, 23 H, OCH_2, OCH_3), 1.83-1.75 (m, 4 H, CH_2), 1.51-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.92-0.84 (m, 6 H, CH_3).

2.4. Synthesis of the sodium carboxylates and acids



Scheme S5. Synthesis of the sodium carboxylates and acids. Compounds **Na*m.m'/n*** and **H*m.m'/n*** with different alkyl chains at both ends were synthesized in an analogous way. *Reagents and conditions*: ***i***) NaOH (1 mol/L aq.), reflux 16 h; ***ii***) HCl (10% aq.), diethyl ether.

2.4.1. Synthesis of the sodium carboxylates (Na*m*/*n*, Na10*/*n*, Na6.16/*n* and Na16.6/*n*)

General procedure: The appropriate methyl ester **5** (or **19**, **25)** (1 mmol) and NaOH (aq. 1 M, 50 mL) were heated 16 h under reflux. After cooling to r.t., the precipitated solid was filtered out and dried in air, the crude product was purified by column chromatography on silica gel and repeated crystallization from appropriate solvent.

Sodium 8-(4,4''-dibutyloxy-p-terphenyl-2'-yloxy)-3,6-dioxaoctanoate (**Na**4/2): Synthesized from **5**/4/2 (2.6 g, 4.7 mmol) and 1 M NaOH aq. (100 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate/n-hexane; yield: 1.7 g (65 %), colorless solid; m.p. = 83 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.45 (d, ^3J(H,H) = 7.3, 2 H, Ar-H), 7.40 (d, ^3J(H,H) = 7.9, 2 H, Ar-H), 7.26-7.22 (m, 1 H, Ar-H), 7.17-7.07 (m, 1 H, Ar-H), 7.06-7.00 (m, 1 H, Ar-H), 6.88 (d, ^3J(H,H) = 8.1, 2 H, Ar-H), 6.82 (d, ^3J(H,H) = 8.1, 2 H, Ar-H), 4.10-3.36 (m, 14 H, OCH_2), 1.81-1.67 (m, 4 H, CH_2), 1.46-1.38 (m, 4 H, CH_2), 1.00-0.84 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 218.4, 158.7, 158.0, 155.7, 140.8, 132.9, 130.7, 130.5 (2C), 130.3, 128.9, 127.9 (2C), 119.5, 114.7 (2C), 113.9 (2C), 111.4, 70.4 (2C), 70.4, 70.0 (2C), 67.8, 67.7, 31.5, 31.4, 19.4, 19.4, 14.0, 14.0. EA: $C_{32}H_{39}O_7Na \cdot 0.5H_2O$ (Cal.) C: 67.71 %, H: 7.10 %, (Found) C: 67.79 %, H: 7.01 %.

Sodium 11-(4,4''-dibutyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**Na**4/3): Synthesized from **5**/4/3 (3.1 g, 5.2 mmol) and 1 M NaOH aq. (100 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate/n-hexane; yield: 2.2 g (70 %), colorless solid; m.p. = 58 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.46 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.28 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.14 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.5, 1 H, Ar-H), 7.09 (d, ^4J(H,H) =

1.5, 1 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.86 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.09 (t, ^3J(H,H) = 4.6, 2 H, OCH_2), 3.98-3.92 (m, 6 H, OCH_2), 3.83-3.77 (m, 2 H, OCH_2), 3.59-3.41 (m, 8 H, OCH_2), 1.81-1.70 (m, 4 H, CH_2), 1.53-1.43 (m, 4 H, CH_2), 0.96 (t, ^3J(H,H) = 7.5, 3 H, OCH_3), 0.95 (t, ^3J(H,H) = 7.5, 3 H, OCH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 174.4, 158.7, 158.0, 155.8, 140.8, 133.0, 130.7, 130.5 (2C), 130.4, 128.9, 127.9 (2C), 119.5, 114.7 (2C), 113.9 (2C), 111.4, 70.6, 70.4, 70.0, 69.9, 69.7 (2C), 68.5, 67.8, 67.7, 31.5, 31.4, 19.4, 19.4, 14.0, 14.0. EA: $C_{34}H_{43}O_8Na \cdot 0.2H_2O$ (Cal.) C: 67.35 %, H: 7.22 %, (Found) C: 67.29 %, H: 7.05 %.

Sodium 14-(4,4''-dibutyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**Na**/4/4): Synthesized from **5**/4/4 (2.62 g, 4.11 mmol) and 1 M NaOH (100 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate/n-hexane; yield: 1.78 g (67 %), colorless solid; m.p. = 15 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.50 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.30 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.16 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.11 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 6.88 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.13 (t, ^3J(H,H) = 5.0, 2 H, CH_2^5), 3.97 (t, ^3J(H,H) = 6.4, 2 H, OCH_2), 3.96 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.92 (s, 2 H, OCH_2), 3.74 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.62-3.50 (m, 12 H, OCH_2), 1.81-1.70 (m, 4 H, CH_2), 1.53-1.43 (m, 4 H, CH_2), 0.97 (t, ^3J(H,H) = 7.4, 3 H, OCH_3), 0.96 (t, ^3J(H,H) = 7.4, 3 H, OCH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 174.3, 158.7, 158.0, 155.8, 140.8, 133.1, 130.7, 130.5 (2C), 130.4, 129.0, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.6, 70.8, 70.4, 70.2 (2C), 70.2, 69.9, 69.7 (2C), 68.4, 67.8, 67.7, 31.5, 31.5 ($C^{2, 2'}$), 19.4, 19.4 ($C^{3, 3'}$), 14.0, 14.0 ($C^{4, 4'}$). EA: $C_{36}H_{47}O_9Na \cdot 0.5H_2O$ (Cal.) C: 65.94 %, H: 7.38 %, (Found) C: 66.13 %, H: 7.23 %.

Sodium 11-(4,4''-dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**Na**6/3): Synthesized from **5**/6/3 (3.2 g, 4.9 mmol) and 1 M NaOH aq. (100 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate/n-hexane; yield: 2.7 g (84 %), colorless solid; transitions/°C: Cr 39 LC 60 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.48 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.45 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.28 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.14 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.08 (s, 1 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.86 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.10-4.05 (m, 2 H, OCH_2), 3.94-3.82 (m, 6 H, OCH_2), 3.77-3.70 (m, 2 H, OCH_2), 3.56-3.45 (m, 8 H, OCH_2), 1.78-1.64 (m, 4 H, CH_2), 1.45-1.40 (m, 4 H, CH_2), 1.39-1.22 (m, 8 H, CH_2), 0.89 (t, ^3J(H,H) = 7.1, 3 H, CH_3), 0.88 (t, ^3J(H,H) = 7.1, 3 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 174.5, 158.7, 158.0, 155.8, 140.8, 133.0, 130.7, 130.5 (2C), 130.4, 128.9, 127.9 (2C), 119.5, 114.7 (2C), 113.9 (2C), 111.4, 70.7, 70.4 (2C), 69.9 (2C), 69.7, 68.5, 68.1, 68.0, 31.7 (2C), 29.4, 29.4, 25.9, 25.8, 22.7 (2C), 14.1 (2C). EA: $C_{38}H_{51}O_8Na \cdot 0.5H_2O$ (Cal.) C: 68.34 %, H: 7.85 %, (Found) C: 68.38 %, H: 8.00 %.

Sodium 14-(4,4"-dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**Na**6/4): See ref. [3]

Sodium 17-(4,4''-dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15-pentaoxaheptadecanoate (**Na**6/5): Synthesized from **5**/6/5 (1.0 g, 1.3 mmol). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate/n-hexane; yield: 0.8g (72 %), colorless solid; transitions/°C: Cr < 20 LC 54 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.32 (d,

^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.11 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.94 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 4.13 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.97 (t, ^{3}J(H,H) = 6.5, 2 H, OCH_2), 3.96 (t, ^{3}J(H,H) = 6.5, 2 H, OCH_2), 3.89 (bs, 2 H, OCH_2, H_2O), 3.75 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.61-3.56 (m, 16 H, OCH_2), 1.82-1.74 (m, 4 H, CH_2), 1.48-1.42 (m, 4 H, CH_2), 1.38-1.31 (m, 8 H, CH_2), 0.90 (t, ^{3}J(H,H) = 7.1, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 175.2, 158.8, 158.1, 155.9, 140.8, 133.1, 130.7, 130.5 (2C), 130.4, 129.0, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.7, 70.6, 70.4, 70.3, 70.1, 70.0, 69.9 (2C), 69.8, 69.6, 69.5, 68.4, 68.1, 68.0, 31.6, 31.6, 29.3, 29.3, 25.8, 25.7, 22.6 (2C), 14.1 (2C). EA: $C_{42}H_{59}O_{10}Na \cdot H_2O$ (Cal.) C: 65.95 %, H: 8.04 %, (Found) C: 65.93 %, H: 8.07 %.

Sodium 20-(4,4''-dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoate (**Na**6/6): Synthesized from **5**/6/6 (1.1 g, 1.4 mmol) and 1 M NaOH aq. (40 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate/n-hexane; yield: 0.8g (71 %), colorless solid; transitions/°C: Cr < 20 LC 33 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.50 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 7.49 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.32 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.11 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.94 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 4.14 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.97 (t, ^{3}J(H,H) = 6.5, 2 H, OCH_2), 3.96 (t, ^{3}J(H,H) = 6.5, 2 H, OCH_2), 3.89 (s, 2 H, OCH_2), 3.77 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.61-3.56 (m, 20 H, OCH_2), 1.82-1.74 (m, 4 H, CH_2), 1.48-1.42 (m, 4 H, CH_2), 1.38-1.31 (m, 8 H, CH_2), 0.89 (t, ^{3}J(H,H) = 7.1, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 174.6, 158.7, 158.1, 155.8, 140.8, 133.1, 130.8, 130.5 (2C), 130.3, 129.1, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.6, 70.8, 70.6 (2C), 70.1, 70.0 (3C), 69.7, 69.7, 69.6 (2C), 69.2, 68.4, 68.2, 68.1, 31.7, 31.7, 29.4, 29.4, 25.9, 25.8, 22.7 (2C), 14.1, 14.1. EA: $C_{44}H_{63}O_{11}Na \cdot 1.8H_2O$ (Cal.) C: 64.18 %, H: 8.15 %, (Found) C: 64.22 %, H: 7.92 %.

Sodium 11-(4,4''-dioctyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**Na**8/3): Synthesized from **5**/8/3 (0.71 g, 1.0 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate; yield: 0.32 g (44.5 %), colorless solid; transitions/°C: Cr <20 LC 112 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.48 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.44 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.26 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.13 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.5, 1 H, Ar-H), 7.07 (d, ^{4}J(H,H) = 1.5, 1 H, Ar-H), 6.90 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.85 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 4.08 (t, ^{3}J(H,H) = 4.5, 2 H, OCH_2), 3.92 (t, ^{3}J(H,H) = 6.4, 2 H, OCH_2), 3.91 (t, ^{3}J(H,H) = 6.4, 2 H, OCH_2), 3.84 (s, 2 H, OCH_2), 3.77-3.70 (m, 2 H, OCH_2), 3.58-3.43 (m, 8 H, OCH_2), 1.81-1.70 (m, 4 H, CH_2), 1.49-1.40 (m, 4 H, CH_2), 1.39-1.20 (m, 16 H, CH_2), 0.89-0.84 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 176.6, 158.8, 158.0, 155.8, 140.8, 133.0, 130.7, 130.5 (2C), 130.4, 128.9, 127.9 (2C), 119.4, 114.7 (2C), 113.8 (2C), 111.2, 70.7 (2C), 70.2, 69.7 (2C), 69.2, 68.5, 68.1, 68.0, 31.9, 31.9, 29.6, 29.4, 29.4, 29.4, 29.3, 29.3, 26.1, 26.1, 22.7 (2C), 14.1 (2C). EA: $C_{42}H_{59}O_8Na \cdot 0.5H_2O$ (Cal.) C: 69.68 %, H: 8.35 %, (Found) C: 69.69 %, H: 8.36 %.

Sodium 14-(4,4''-dioctyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**Na**8/4): See ref. [3].

Sodium 5-(4,4''-didecyloxy-p-terphenyl-2'-yloxy)-3-oxapentanoate (**Na**10/1): Synthesized from **5**/10/1 (0.45 g, 0.64 mmol) and 1 M NaOH aq. (30 mL). Purified by columns chromatography,

eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate; yield: 0.32 g (73 %); colorless waxy solid; transitions/°C: Cr < 20 LC 118 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.45-7.27 (m, 5 H, Ar-H), 7.18-7.11 (m, 1 H, Ar-H), 7.10-7.00 (m, 1 H, Ar-H), 6.93-6.70 (m, 4 H, Ar-H), 4.00-3.40 (m, 10 H, OCH_2), 1.78-1.50 (m, 4 H, CH_2), 1.45-1.22 (m, 28 H, CH_2), 0.89-0.83 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 158.6, 158.0, 156.0, 140.4, 133.1, 130.6, 130.3 (2C), 128.8, 127.8 (2C), 119.3, 114.6 (2C), 113.9 (2C), 111.1, 70.5, 69.2 (2C), 68.0, 68.0, 32.0 (2C), 29.7 (4C), 29.6 (2C), 29.4 (4C), 26.2 (2C), 22.8 (2C), 14.2 (2C). EA: $C_{42}H_{59}O_6Na \cdot 0.3H_2O$ (Cal.) C: 73.29 %, H: 8.73 %, (Found) C: 73.36 %, H: 9.06 %.

Sodium 8-(4,4''-didecyloxy-p-terphenyl-2'-yloxy)-3,6-dioxaoctanoate (**Na**10/2): Synthesized from **5**/10/2 (1.9 g, 2.6 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate; yield: 1.3 g (69 %), colorless waxy solid; transitions/°C: Cr 53 LC 119 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.45-7.27 (m, 4 H, Ar-H), 7.24-7.20 (m, 1 H, Ar-H), 7.18-7.08 (m, 1 H, Ar-H), 7.07-6.98 (m, 1 H, Ar-H), 6.86 (d, ^3J(H,H) = 8.5, 2 H, Ar-H), 6.81 (d, ^3J(H,H) = 8.1, 2 H, Ar-H), 4.10-3.95 (m, 2 H, OCH_2), 3.94-3.82 (m, 6 H, OCH_2), 3.80-3.64 (m, 2 H, OCH_2), 3.56-3.33 (m, 4 H, OCH_2), 1.78-1.64 (m, 4 H, CH_2), 1.45-1.22 (m, 28 H, CH_2), 0.89-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 158.9, 158.2, 156.0, 140.9, 133.1, 130.9, 130.6 (3C), 129.0, 128.0 (2C), 119.5, 114.9 (2C), 114.0 (2C), 111.3, 70.5 (2C), 68.2 (3C), 68.1 (2C), 32.1 (2C), 29.8 (4C), 29.7 (2C), 29.6, 29.6, 29.5 (2C) , 26.3, 26.3, 22.9 (2C), 14.3 (2C). EA: $C_{44}H_{63}O_7Na \cdot 0.5H_2O$ (Cal.) C: 71.80 %, H: 8.76 %, (Found) C: 71.90 %, H: 8.66 %.

Sodium 11-(4,4"-didecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**Na**10/3): The methyl ester **5**/10/3 (0.45 g, 0.59 mmol) and aqueous NaOH (1 M, 50 mL) were heated for 16 h under reflux. After cooling to r.t., the precipitated solid was filtered off and dried in air, the crude product was purified by column chromatography on silica gel with $CHCl_3/CH_3OH$ = 10:1 (V/V) as eluent, and repeated crystallized from ethyl acetate. Yield 0.25 g (55 %); colorless waxy solid; Transitions/°C: Cr 69 LC122 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.46 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 7.44 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 7.26 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.12 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.06 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.84 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.06 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 3.93-3.89 (m, 4 H, OCH_2), 3.76 (s, 2 H, CH_2), 3.69 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 3.53-3.43 (m, 8 H, OCH_2), 1.78-1.71 (m, 4 H, CH_2), 1.44-1.25 (m, 28 H, CH_2), 0.88-0.84 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 176.5, 159.3, 158.6, 156.4, 141.3, 133.6, 131.2, 131.0 (2C), 130.9, 129.4, 128.4 (2C), 119.9, 115.2 (2C), 114.4 (2C), 111.8, 71.5, 71.0, 70.6, 70.2, 70.0, 69.4, 69.0, 68.6, 68.5, 32.4 (2C), 30.1 (2C), 30.0 (2C), 30.0 (2C), 29.9 (2C), 29.9 (2C), 26.7, 26.6, 23.2 (2C), 14.6 (2C). EA: $C_{46}H_{67}O_8Na \cdot 0.5H_2O$ (Cal.) C: 70.83 %, H: 8.79 %, (Found) C: 70.70 %, H: 8.54 %.

Sodium 14-(4,4"-didecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**Na**10/4): See ref. [4].

Sodium 20-(4,4''-didecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoate (**Na**10/6): Synthesized from **5**/10/6 (0.97 g, 1.1 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate; yield: 0.16 g (16 %), colorless waxy solid; transitions/°C: Cr < 20 LC 71 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 200 MHz) δ = 7.53-7.46 (m, 4 H, Ar-H), 7.32 (d, ^3J(H,H) = 7.5, 1 H, Ar-H), 7.20-7.14 (m, 1 H, Ar-H), 7.12 (s, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 9.1, 2 H, Ar-H), 6.90 (d,

^{3}J(H,H) = 9.1, 2 H, Ar-H), 4.15 (t, ^{3}J(H,H) = 4.8, 2 H, OCH_2), 4.02-3.82 (m, 6 H, OCH_2), 3.77 (t, ^{3}J(H,H) = 4.8, 2 H, OCH_2), 3.67-3.46 (m, 20 H, OCH_2), 1.83-1.69 (m, 4 H, CH_2), 1.54-1.18 (m, 28 H, CH_2), 0.87 (t, ^{3}J(H,H) = 6.2, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 50 MHz) δ = 158.8, 158.2, 155.9, 140.9, 133.1, 130.8, 130.5 (2C), 130.3, 129.1, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.6, 70.5 (2C), 70.1 (2C), 69.9 (6C), 69.7 (2C), 68.3, 68.0 (2C), 31.9 (2C), 29.5 (4C), 29.4 (4C), 29.3 (2C), 26.0 (2C), 22.6 (2C), 14.1 (2C). EA: $C_{52}H_{79}O_{11}Na$ (Cal.) C: 69.15 %, H: 8.82 %, (Found) C: 69.03 %, H: 8.97 %.

Sodium 11-[4,4''-bis(3,7-dimethyloctyloxy)-p-terphenyl-2'-yloxy]-3,6,9-trioxaundecanoate (**Na**10*/3): Synthesized from **5**/10*/3 (0.7 g, 0.92 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate/n-hexane; yield: 0.35 g (49 %), colorless solid; transitions/°C: Cr 17 LC 73 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.48 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 7.44 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.27 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.13 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.07 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.91 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.85 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 4.09 (t, ^{3}J(H,H) = 4.8, 2 H, OCH_2), 3.99-3.91 (m, 4 H, OCH_2), 3.85 (s, 2 H, OCH_2), 3.79-3.71 (m, 2 H, OCH_2), 3.58-3.42 (m, 8 H, OCH_2), 1.84-1.76 (m, 2 H, CH), 1.66-1.60 (m, 2 H, CH), 1.59-1.42 (m, 4 H, CH_2), 1.37-1.07 (m, 12 H, CH_2), 0.93-0.90 (m, 6 H, CH_3), 0.89-0.70 (m, 12 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 158.7, 158.0, 155.8, 140.8, 133.0, 130.7, 130.5 (2C), 130.4, 128.9, 127.9 (2C), 119.4, 114.7 (2C), 113.9 (2C), 111.2, 70.8 (2C), 70.6, 70.2, 69.8, 69.4, 68.6, 66.5, 66.4, 39.3 (2C), 37.4 (2C), 36.4, 36.4, 30.0 (2C), 28.1 (2C), 24.8 (2C), 22.8 (2C), 22.7 (2C), 19.8 (2C). EA: $C_{46}H_{67}O_8Na \cdot 0.5H_2O$ (Cal.) C: 70.83 %, H: 8.79 %, (Found) C: 70.80 %, H: 8.63 %.

Sodium 14-[4,4''-bis(3,7-dimethyloctyloxy)-p-terphenyl-2'-yloxy]-3,6,9,12-tetraoxatetradecanoate (**Na**10*/4): Synthesized from **5**/10*/4 (1.15 g, 1.43 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from n-hexane; yield: 0.55 g (47 %), colorless solid; transitions/°C: Cr < 20 LC 56 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.50 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 7.30 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.16 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.11 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.93 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.88 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 4.12 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 4.04-3.93 (m, 4 H, OCH_2), 3.92 (s, 2 H, OCH_2), 3.73 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.62-3.54 (m, 12 H, OCH_2), 1.85-1.77 (m, 2 H, CH), 1.67-1.61 (m, 2 H, CH), 1.60-1.43 (m, 4 H, CH_2), 1.37-1.07 (m, 12 H, CH_2), 0.93-0.90 (m, 6 H, CH_3), 0.89-0.70 (m, 12 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 175.0, 158.7, 158.0, 155.8, 140.8, 133.1, 130.7, 130.4 (2C), 130.3, 128.9, 127.9 (2C), 119.5, 114.8 (2C), 113.9 (2C), 111.5, 70.8, 70.5, 70.4, 70.3, 70.2, 69.9, 69.7, 69.7, 68.3, 66.5, 66.4, 39.3 (2C), 37.4, 37.4, 36.4, 36.3, 30.0, 30.0, 28.0 (2C), 24.7 (2C), 22.8 (2C), 22.7 (2C), 19.8 (2C). EA: $C_{48}H_{71}O_9Na \cdot 0.5H_2O$ (Cal.) C: 69.96 %, H: 8.81 %, (Found) C: 69.80 %, H: 8.69 %.

Sodium 11-(4,4''-dihexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**Na**16/3): Synthesized from **5**/16/3 (3.9 g, 4.2 mmol) and 1 M NaOH aq. (100 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate; yield: 3.3 g (84 %), colorless solid; transitions/°C: Cr LC 116 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 500 MHz) δ = 7.51 (d, ^{3}J(H,H) = 8.5, 2 H, Ar-H), 7.48 (d, ^{3}J(H,H) = 8.5, 2 H, Ar-H), 7.29 (d, ^{3}J(H,H) = 7.8, 1 H, Ar-H), 7.16 (d, ^{3}J(H,H) = 7.8, 1 H, Ar-H), 7.11 (s, 1 H, Ar-H), 6.93 (d, ^{3}J(H,H) = 8.5, 2 H, Ar-H), 6.88 (d, ^{3}J(H,H) = 8.5, 2 H, Ar-H), 4.14-4.07 (m, 2 H, OCH_2),

3.97-3.92 (m, 6 H, OCH_2), 3.79-3.73 (m, 2 H, OCH_2), 3.59-3.54 (m, 8 H, OCH_2), 1.83-1.76 (m, 4 H, CH_2), 1.50-1.42 (m, 4 H, CH_2), 1.41-1.22 (m, 48 H, CH_2), 0.90 (t, ^3J(H,H) = 6.8, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 125 MHz) δ = 175.7, 158.8, 158.1, 155.8, 140.8, 133.0, 130.7, 130.5 (2C), 130.3, 128.8, 127.9 (2C), 119.4, 114.7 (2C), 113.8 (2C), 111.2, 70.6, 70.2, 70.1, 69.8, 69.6 (2C), 68.3, 68.0, 67.9, 31.9 (2C), 29.7 (8C), 29.6 (4C), 29.6 (2C), 29.6 (2C), 29.4 (2C), 29.4 (2C), 29.3 (2C), 26.1, 26.0, 22.6 (2C), 14.0 (2C). EA: $C_{58}H_{91}O_8Na$ (Cal.) C: 74.16 %, H: 9.76 %, (Found) C: 74.39 %, H: 9.72 %.

Sodium 14-(4,4''-dihexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxaundecanoate (**Na**16/4): See ref. [4].

Sodium 11-(4,4''-didecyloxy-p-terphenyl-3-yloxy)-3,6,9-trioxaundecanoate (**Na***10/3): Synthesized from **19**/10/3 (0.70 g, 0.93 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from chloroform/n-hexane; yield: 178 mg (25 %), colorless solid; transitions/°C: Cr 77 LC 128 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.53 (s, 4 H, Ar-H), 7.48 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.9, 1 H, Ar-H), 7.10 (dd, ^3J(H,H) = 7.5, ^4J(H,H) = 1.9, 1 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.85 (d, ^3J(H,H) = 7.5, 1 H, Ar-H), 4.16 (t, ^3J(H,H) = 4.5, 2 H, OCH_2), 3.95-3.86 (m, 6 H, OCH_2), 3.85 (s, 2 H, OCH_2), 3.80-3.45 (m, 8 H, OCH_2), 1.90-1.75 (m, 4 H, CH_2), 1.52-1.18 (m, 28 H, CH_2), 0.92-0.84 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 175.8, 158.6, 148.7 (2C), 139.1, 138.9, 133.6, 132.8, 127.8 (2C), 126.9 (2C), 126.8 (2C), 119.8, 114.7 (2C), 113.8, 113.3, 70.8 (2C), 70.3, 69.9 (2C), 69.3, 69.1, 68.1 (2C), 32.0 (2C), 29.7, 29.7 (2C), 29.7, 29.5, 29.5, 29.4 (4C), 26.2, 26.1, 22.8 (2C), 14.2 (2C). EA: $C_{46}H_{67}O_8Na$ (Cal.) C: 71.66 %, H: 8.76 %, (Found) C: 71.56 %, H: 8.69 %.

Sodium 14-(4,4''-didecyloxy-p-terphenyl-3-yloxy)-3,6,9,12-tetraoxatetradecanoate (**Na***10/4): Synthesized from **19**/10/4 (1.0 g, 1.2 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from chloroform/n-hexane; yield: 252 mg (25 %), colorless solid; transitions/°C: Cr 90 LC 113 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400M Hz) δ = 7.55 (s, 4 H, Ar-H), 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.17 (d, ^4J(H,H) = 2.1, 1 H, Ar-H), 7.14 (dd, ^3J(H,H) = 8.3, ^4J(H,H) = 2.1, 1 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.3, 1 H, Ar-H), 4.19 (t, ^3J(H,H) = 4.8, 2 H, OCH_2), 4.00-3.94 (m, 4 H, OCH_2), 3.86-3.82 (m, 4 H, OCH_2), 3.72-3.55 (m, 12 H, OCH_2), 1.81-1.75 (m, 4 H, CH_2), 1.46-1.18 (m, 28 H, CH_2), 0.87 (t, ^3J(H,H) = 6.7, 3 H, CH_3), 0.86 (t, ^3J(H,H) = 6.8, 3 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 174.7, 158.6, 148.8, 148.7, 139.1, 138.9, 133.7, 132.9, 127.8 (2C), 126.9 (2C), 126.8 (2C), 120.0, 114.8 (2C), 114.0 (2C), 70.7 (2C), 70.4, 70.2, 70.0, 69.8, 69.7, 69.2 (2C), 68.1 (2C), 32.0 (2C), 29.7, 29.7, 29.6 (2C), 29.5 (2C), 29.4 (3C), 29.4, 26.2, 26.1, 22.8 (2C), 14.2 (2C). EA: $C_{48}H_{71}O_9Na$ (Cal.) C: 70.73 %, H: 8.78 %, (Found) C: 70.60 %, H: 8.71 %.

Sodium 11-(4-hexyloxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**Na**6.16/3): Synthesized from **25**/6.16/3 (0.48 g, 0.61 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate; yield: 124 mg (25.5 %), colorless solid; transitions/°C: Cr 40 LC 89 Iso. ^{1}H-NMR ($CDCl_3$, J/Hz, 400 MHz) δ = 7.51 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.47 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.29 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.15 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.11 (s, 1 H, Ar-H), 6.92 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.88 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 4.10

(s, 2 H, OCH_2), 3.94 (t, $^3J(H,H)$ = 6.6, 4 H, OCH_2), 3.92 (s, 2 H, OCH_2), 3.74 (bs, 2 H, OCH_2), 3.60-3.42 (m, 8 H, OCH_2), 1.82-1.74 (m, 4 H, CH_2), 1.48-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.94-0.88 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 176.0, 158.7, 158.0, 155.8, 140.7, 132.9, 130.7, 130.4 (2C), 130.3, 128.8, 127.8 (2C), 119.4, 114.7 (2C), 113.8 (2C), 111.1, 70.6, 70.3, 70.1, 69.7, 69.6, 69.4, 68.4, 68.0, 67.9, 31.9, 31.6, 29.7 (5C), 29.6 (2C), 29.6 (2C), 29.4, 29.3, 29.3, 26.1, 25.7, 22.6, 22.6, 14.1, 14.0. EA: $C_{48}H_{71}O_8Na \cdot 0.5H_2O$ (Cal.) C: 71.34 %, H: 8.98 %, (Found) C: 71.63 %, H: 8.87 %.

Sodium 14-(4-hexyloxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**Na**6.16/4): Synthesized from **25**/6.16/4 (1.1 g, 1.3 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate; yield: 1.0 g (90 %), colorless solid; transitions/°C: Cr 54 LC 74 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.50 (d, $^3J(H,H)$ = 8.7, 2 H, Ar-H), 7.49 (d, $^3J(H,H)$ = 8.7, 2 H, Ar-H), 7.30 (d, $^3J(H,H)$ = 7.9, 1 H, Ar-H), 7.16 (dd, $^3J(H,H)$ = 7.9, $^4J(H,H)$ = 1.7, 1 H, Ar-H), 7.11 (d, $^4J(H,H)$ = 1.7, 1 H, Ar-H), 6.93 (d, $^3J(H,H)$ = 8.7, 2 H, Ar-H), 6.88 (d, $^3J(H,H)$ = 8.9, 2 H, Ar-H), 4.12 (t, $^3J(H,H)$ = 5.0, 2 H, OCH_2), 3.95 (t, $^3J(H,H)$ = 6.5, 2 H, OCH_2), 3.95 (t, $^3J(H,H)$ = 6.6, 2 H, OCH_2), 3.92 (s, 2 H, OCH_2), 3.73 (t, $^3J(H,H)$ = 5.0, 2 H, OCH_2), 3.75-3.50 (m, 12 H, OCH_2), 1.81-1.73 (m, 4 H, CH_2), 1.47-1.41 (m, 4 H, CH_2), 1.39-1.21 (m, 28 H, CH_2), 0.91-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 175.2, 158.8, 158.1, 155.9, 140.8, 133.1, 130.7, 130.5 (2C), 130.4, 129.0, 127.9 (2C), 119.5, 114.8 (2C), 113.9 (2C), 111.5, 70.8, 70.4, 70.3, 70.2, 70.2, 69.9, 69.7, 69.6, 68.3, 68.1, 68.0, 31.9, 31.6, 29.7 (4C), 29.7 (2C), 29.7, 29.7, 29.6, 29.5, 29.4, 29.4, 26.1, 25.8, 22.7, 22.6, 14.1, 14.1. EA: $C_{50}H_{75}O_9Na$ (Cal.) C: 71.23 %, H: 8.97 %, (Found) C: 71.06 %, H: 8.80 %.

Sodium 20-(4-hexyloxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoate (**Na**6.16/6): Synthesized from **25**/6.16/6 (1.1 g, 1.2 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed repeated crystallization from ethyl acetate; yield: 0.75 g (67.5 %), colorless solid; transitions/°C: Cr 28 LC 70 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.50 (d, $^3J(H,H)$ = 8.7, 2 H, Ar-H), 7.50 (d, $^3J(H,H)$ = 8.7, 2 H, Ar-H), 7.32 (d, $^3J(H,H)$ = 7.9, 1 H, Ar-H), 7.17 (dd, $^3J(H,H)$ = 7.9, $^4J(H,H)$ = 1.7, 1 H, Ar-H), 7.11 (d, $^4J(H,H)$ = 1.7, 1 H, Ar-H), 6.94 (d, $^3J(H,H)$ = 8.9, 2 H, Ar-H), 6.89 (d, $^3J(H,H)$ = 8.9, 2 H, Ar-H), 4.14 (t, $^3J(H,H)$ = 5.0, 2 H, OCH_2), 3.97 (t, $^3J(H,H)$ = 6.6, 2 H, OCH_2), 3.96 (t, $^3J(H,H)$ = 6.5, 2 H, OCH_2), 3.88 (s, 2 H, OCH_2), 3.77 (t, $^3J(H,H)$ = 5.0, 2 H, OCH_2), 3.64-3.55 (m, 20 H, OCH_2), 1.81-1.74 (m, 4 H, CH_2), 1.47-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.91-0.84 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 175.1, 158.7, 158.1, 155.8, 140.8, 133.1, 130.7, 130.4 (2C), 130.3, 129.0, 127.9 (2C), 119.6, 114.7 (2C), 113.9 (2C), 111.6, 70.8, 70.8, 70.6, 70.3, 70.1, 70.1, 70.1, 69.9, 69.8, 69.7, 69.6, 69.3, 68.4, 68.2, 68.0, 32.0, 31.7, 29.8 (4C), 29.7 (2C), 29.7 (2C), 29.7, 29.5, 29.4, 29.4, 26.2, 25.9, 22.8, 22.7, 14.2, 14.1. EA: $C_{54}H_{83}O_{11}Na \cdot 1.5H_2O$ (Cal.) C: 67.68 %, H: 9.05 %, (Found) C: 67.52 %, H: 8.85 %.

Sodium 11-(4-hexadecyloxy-4''-hexyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoate (**Na**16.6/3): Synthesized from **25**/16.6/3 (0.75 g, 0.95 mmol) and 1 M NaOH (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V), followed by repeated crystallization from ethyl acetate; yield: 0.75 g (71 %), colorless solid; transitions/°C: Cr < 20 LC 100 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.48 (d, $^3J(H,H)$ = 8.7, 2 H, Ar-H), 7.44 (d, $^3J(H,H)$ = 8.7, 2 H, Ar-H), 7.26 (d, $^3J(H,H)$ = 7.9, 1 H, Ar-H), 7.12 (dd, $^3J(H,H)$ = 7.9, $^4J(H,H)$ = 1.5, 1 H, Ar-H), 7.12 (d, $^4J(H,H)$ = 1.5, 1 H, Ar-H), 6.90 (d, $^3J(H,H)$ = 8.9, 2 H, Ar-H), 6.85 (d,

^{3}J(H,H) = 8.7, 2 H, Ar-H), 4.08 (t, ^{3}J(H,H) = 4.6, 2 H, OCH_2), 3.94-3.89 (m, 4 H, OCH_2), 3.85 (s, 2 H, OCH_2), 3.75-3.71 (m, 2 H, OCH_2), 3.56-3.43 (m, 8 H, OCH_2), 1.80-1.72 (m, 4 H, CH_2), 1.46-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 28 H, CH_2), 0.91-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 176.7, 158.7, 158.1, 155.8, 140.8, 133.0, 130.7, 130.5 (2C), 130.4, 128.9, 127.9 (2C), 119.4, 114.7 (2C), 113.8 (2C), 111.2, 70.7 (2C), 70.2, 69.7 (2C), 69.2, 68.5, 68.1, 68.0, 31.9, 31.6, 29.7 (4C), 29.7 (2C), 29.7 (2C), 29.5, 29.4, 29.4, 29.3, 26.1, 25.8, 22.7, 22.6, 14.1, 14.1. EA: $C_{48}H_{71}O_8Na \cdot 0.5H_2O$ (Cal.) C: 71.34 %, H: 8.98 %, (Found) C: 71.36 %, H: 8.84 %.

Sodium 14-(4-hexadecyloxy-4''-hexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoate (**Na**16.6/4): Synthesized from **25**/16.6/4 (1.0 g, 1.2 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V),followed by repeated crystallization from ethyl acetate; yield: 0.65 g (64 %), colorless solid; transitions/°C: Cr < 20 LC 79 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.50 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.30 (d, ^{3}J(H,H) = 7.7, 1 H, Ar-H), 7.16 (dd, ^{3}J(H,H) = 7.7, ^{4}J(H,H) = 1.5, 1 H, Ar-H), 7.11 (d, ^{4}J(H,H) = 1.5, 1 H, Ar-H), 6.93 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.88 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 4.11 (t, ^{3}J(H,H) = 4.6, 2 H, OCH_2), 3.96 (t, ^{3}J(H,H) = 5.9, 2 H, OCH_2), 3.94 ((t, ^{3}J(H,H) = 5.9, 2 H, OCH_2), 3.91 (s, 2 H, OCH_2), 3.73 (t, ^{3}J(H,H) = 4.6, 2 H, OCH_2), 3.63-3.50 (m, 12 H, OCH_2), 1.81-1.75 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 28 H, CH_2), 0.89-0.86 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 175.2, 158.7, 158.0, 155.8, 140.7, 133.0, 130.7, 130.4 (2C), 130.3, 128.9, 127.8 (2C), 119.5, 114.7 (2C), 113.8 (2C), 111.5, 70.8, 70.4 (2C), 70.2, 70.2, 69.9, 69.6 (2C), 68.3, 68.1, 68.0, 32.0, 31.7, 29.8 (4C), 29.7 (2C), 29.7 (2C), 29.5, 29.4, 29.4, 29.3, 26.2, 25.8, 22.7, 22.7, 14.2, 14.1. EA: $C_{50}H_{75}O_9Na \cdot 0.5H_2O$ (Cal.) C: 70.47 %, H: 8.99 %, (Found) C: 70.74 %, H: 8.84 %.

Sodium 20-(4-hexadecyloxy-4''-hexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoate (**Na**16.6/6): Synthesized from **25**/16.6/6 (1.05 g, 1.14 mmol) and 1 M NaOH aq. (50 mL). Purified by columns chromatography, eluent: $CHCl_3$/MeOH = 10/1 (V/V) followed by repeated crystallization from ethyl acetate; yield: 0.64 g (60 %), colorless solid; transitions/°C: Cr 55 LC 80 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.50 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.50 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.32 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.17 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.12 (d, ^{4}J(H,H) = 1.6, 1 H, Ar-H), 6.94 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 6.89 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 4.14 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.97 (t, ^{3}J(H,H) = 5.9, 2 H, OCH_2), 3.96 (t, ^{3}J(H,H) = 5.8, 2 H, OCH_2), 3.88 (s, 2 H, OCH_2), 3.76 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.61-3.43 (m, 20 H, OCH_2), 1.80-1.74 (m, 4 H, CH_2), 1.48-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 28 H, CH_2), 0.91-0.84 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 175.4, 158.7, 158.1, 155.9, 140.8, 133.1, 130.8, 130.5 (2C), 130.3, 129.0, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.6, 70.6 (2C), 70.3, 70.2, 70.1 (2C), 70.1, 69.9, 69.8, 69.7, 69.6, 69.3, 68.3, 68.1, 68.0, 31.9, 31.6, 29.7 (3C), 29.7 (2C), 29.6 (2C), 29.6, 29.5, 29.4, 29.4, 29.3, 26.1, 25.8, 22.7, 22.6, 14.1, 14.0. EA: $C_{54}H_{83}O_{11}Na \cdot H_2O$ (Cal.) C: 68.33 %, H: 9.03 %, (Found) C: 68.42 %, H: 8.85 %.

2.4.2. Carboxylic acids (H*m*/*n*, H*10/*n*, H6.16/*n* and H16.6/*n*)

General procedure: The appropriate sodium carboxylate **Na**/*m*/*n* (or **Na***/10/*n*, **Na**/6.16/*n* and **Na**/16.6/*n*) (1 mmol) was dissolved in the mixture of diethyl ether (50 mL) and aqueous HCl (10 %, 2 mL) with stirring. Stirring was continued ca. 4 h until the reaction mixture was clear and then the mixture was poured into a separatory funnel, the organic layer was separated and washed with distilled water until the water phase become neutral. The solvent was evaporated under

vacuum and the crude product was purified by repeated crystallization from ethyl acetate/n-hexane.

8-(4,4''-Dibutyloxy-p-terphenyl-2'-yloxy)-3,6-dioxaoctanoic acid (**H**4/2): Synthesized from **Na**4/2 (1.8 g, 3.2 mmol). The crude product was purified by repeated crystallization from ethyl acetate/n-hexane; yield 1.0 g (56 %); colorless solid; m.p. = 76 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 7.33 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.20 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.12 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 4.16 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 4.08 (s, 2 H, OCH_2), 4.00 (t, ^{3}J(H,H) = 5.9, 2 H, OCH_2), 3.99 (t, ^{3}J(H,H) = 5.9, 2 H, OCH_2), 3.82 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.66-3.60 (m, 4 H, OCH_2), 1.82-1.74 (m, 4 H, CH_2), 1.55-1.45 (m, 4 H, CH_2), 0.98 (t, ^{3}J(H,H) = 7.5, 3 H, CH_3), 0.98 (t, ^{3}J(H,H) = 7.5, 3 H, OCH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.7, 158.8, 158.1, 155.7, 141.0, 133.1, 130.8, 130.5 (2C), 130.3, 129.2, 127.9 (2C), 119.8, 114.8 (2C), 113.9 (2C), 111.7, 71.5, 70.4, 69.9, 68.7, 68.4, 67.9, 67.8, 31.5, 31.4, 19.4, 19.4, 14.0, 14.0. EA: $C_{32}H_{40}O_7$ (Cal.) C: 71.62 %, H: 7.51 %, (Found) C: 71.99 %, H: 7.34 %.

11-(4,4''-Dibutyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoic acid (**H**4/3): Synthesized from **Na**4/3 (1.7 g, 2.8 mmol). The crude product was purified by repeated crystallization from ethyl acetate/n-hexane; yield 1.5 g (92 %); colorless solid; m.p. = 53 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.51 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 7.33 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.14 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.40 (bs, 1 H, OH), 4.16 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 4.09 (s, 2 H, OCH_2), 4.00 (t, ^{3}J(H,H) = 6.5, 2 H, OCH_2), 3.99 (t, ^{3}J(H,H) = 6.5, 2 H, OCH_2), 3.78 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.67-3.56 (m, 8 H, OCH_2), 1.81-1.70 (m, 4 H, CH_2), 1.56-1.44 (m, 4 H, CH_2), 0.98 (t, ^{3}J(H,H) = 7.5, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 172.4, 158.7, 158.1, 155.8, 140.9, 133.1, 130.8, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.7, 71.2, 70.6 (2C), 70.1, 69.7, 68.7, 68.5, 67.8, 67.7, 31.4, 31.4, 19.3, 19.3, 13.9, 13.9. EA: $C_{34}H_{44}O_8 \cdot 0.5H_2O$ (Cal.) C: 69.25 %, H: 7.69 %, (Found) C: 69.32 %, H: 7.51 %.

14-(4,4''-Dibutyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoic acid (**H**4/4): Synthesized from **Na**4/4 (1.56 g, 2.4 mmol). The crude product was purified by repeated crystallization from ethyl acetate/n-hexane; yield 1.06 g (92 %); colorless solid; m.p. = 46 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.51 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.33 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 6.91 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 5.93 (bs, 1 H, OH), 4.17 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 4.08 (s, 2 H, OCH_2), 4.00 (t, ^{3}J(H,H) = 6.4, 2 H, OCH_2), 3.99 (t, ^{3}J(H,H) = 6.5, 2 H, OCH_2), 3.78 (t, ^{3}J(H,H) = 5.0, 2 H, OCH_2), 3.68-3.56 (m, 12 H, OCH_2), 1.81-1.75 (m, 4 H, CH_2), 1.53-1.43 (m, 4 H, CH_2), 0.98 (t, ^{3}J(H,H) = 7.4, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.8, 158.7, 158.0, 155.8, 140.8, 133.1, 130.7, 130.5 (2C), 130.4, 129.0, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.6, 71.3, 70.9, 70.6, 70.5, 70.3, 70.2, 69.7, 69.0, 68.4, 67.8, 67.7, 31.5, 31.4, 19.4, 19.3, 14.0, 13.9. EA: $C_{36}H_{48}O_9 \cdot 0.5H_2O$ (Cal.) C: 68.22 %, H: 7.79 %, (Found) C: 68.34 %, H: 7.63 %.

11-(4,4''-Dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoic acid (**H**6/3): Synthesized from **Na**6/3 (2.9 g, 4.4 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane;

yield 2.5 g (89 %); colorless solid; m.p. = 32 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.50 (d, ^{3}J(H,H) = 8.5, 2 H, Ar-H), 7.33 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 6.91 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 4.16 (t, ^{3}J(H,H) = 4.6, 2 H, OCH_2), 4.08 (s, 2 H, OCH_2), 3.99 (t, ^{3}J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^{3}J(H,H) = 6.5, 2 H, OCH_2), 3.78 (t, ^{3}J(H,H) = 4.6, 2 H, OCH_2), 3.69-3.59 (m, 8 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.50-1.41 (m, 4 H, CH_2), 1.38-1.25 (m, 8 H, CH_2), 0.90 (t, ^{3}J(H,H) = 7.1, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 125 MHz) δ = 171.3, 158.7, 158.1, 155.8, 140.9, 133.1, 130.8, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.8, 71.6, 70.8, 70.7, 70.1, 69.8, 68.9, 68.6, 68.2, 68.1, 31.7, 31.7, 29.4 (2C), 25.9, 25.8, 22.7 (2C), 14.1 (2C). EA: $C_{38}H_{52}O_8 \cdot 0.7H_2O$ (Cal.) C: 70.27 %, H: 8.29 %, (Found) C: 70.10 %, H: 8.05 %.

14-(4,4"-Dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoic acid (**H**6/4): See ref. [3].

17-(4,4''-Dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15-pentaoxaheptadecanoic acid (**H**6/5): Synthesized from **Na**6/5 (0.61 g, 0.82 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield 0.38 g (64 %); colorless solid; m.p. = 21 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.53 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 7.51 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.33 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.90 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 4.17 (t, ^{3}J(H,H) = 4.8, 2 H, OCH_2), 4.09 (s, 2 H, OCH_2), 3.99 (t, ^{3}J(H,H) = 6.6, 2 H, OCH_2), 3.98 (t, ^{3}J(H,H) = 6.6, 2 H, OCH_2), 3.79 (t, ^{3}J(H,H) = 4.8, 2 H, OCH_2), 3.70-3.57 (m, 16 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.50-1.42 (m, 4 H, CH_2), 1.38-1.31 (m, 8 H, CH_2), 0.92-0.88 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.5, 158.7, 158.0, 155.9, 140.8, 133.2, 130.7, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.5, 114.8 (2C), 113.9 (2C), 111.6, 71.2, 70.7, 70.6, 70.6 (2C), 70.4, 70.3(2C), 69.8, 69.1, 68.4, 68.2, 68.1, 31.7, 31.7, 29.4, 29.3, 25.8, 25.8, 22.7, 22.7, 14.1 (2C). EA: $C_{42}H_{60}O_{10} \cdot 0.5\ H_2O$ (Cal.) C: 68.73 %, H: 8.38 %, (Found) C: 68.67 %, H: 8.08 %.

20-(4,4''-Dihexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoic acid (**H**6/6): Synthesized from **Na**6/6 (0.65 g, 0.82 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield 0.33 g (52 %); colorless solid; m.p. = Cr 15 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.51 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 7.51 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 7.33 (d, ^{3}J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^{3}J(H,H) = 7.9, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^{4}J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^{3}J(H,H) = 8.7, 2 H, Ar-H), 6.90 (d, ^{3}J(H,H) = 8.9, 2 H, Ar-H), 4.16 (t, ^{3}J(H,H) = 4.8, 2 H, OCH_2), 4.10 (s, 2 H, OCH_2), 3.99 (t, ^{3}J(H,H) = 6.6, 2 H, OCH_2), 3.98 (t, ^{3}J(H,H) = 6.6, 2 H, OCH_2), 3.79 (t, ^{3}J(H,H) = 4.8, 2 H, OCH_2), 3.70-3.57 (m, 20 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.50-1.42 (m, 4 H, CH_2), 1.38-1.31 (m, 8 H, CH_2), 0.92-0.88 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.1, 158.7, 158.1, 155.9, 140.8, 133.2, 130.7, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.7, 71.3, 70.9, 70.6 (3C), 70.5 (2C), 70.4, 70.3, 70.3, 69.7, 69.2, 68.4, 68.2, 68.1, 31.7, 31.7, 29.4, 29.4, 25.9, 25.8, 22.7 (2C), 14.1 (2C). EA: $C_{44}H_{64}O_{11} \cdot H_2O$ (Cal.) C: 67.15 %, H: 8.45 %, (Found) C: 67.14 %, H: 8.14 %.

11-(4,4''-Dioctyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoic acid (**H**8/3): Synthesized from **Na**8/3 (0.45 g, 0.63 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield 0.30 g (59 %); colorless solid; transitions/°C: Cr 22 LC 43 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^{3}J(H,H) = 8.8, 2 H, Ar-H), 7.51 (d, ^{3}J(H,H) = 8.8, 2 H, Ar-H), 7.33 (d, ^{3}J(H,H)

= 7.8, 1 H, Ar-H), 7.13 (dd, ^3J(H,H) = 7.8, ^4J(H,H) = 1.5, 1 H, Ar-H), 7.14 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.10 (bs, 1 H, OH), 4.16 (t, ^3J(H,H) = 4.9, 2 H, OCH_2), 4.09 (s, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.4, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.77-3.70 (t, ^3J(H,H) = 4.9, 2 H, OCH_2), 3.67-3.60 (m, 8 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.50-1.40 (m, 4 H, CH_2), 1.39-1.20 (m, 16 H, CH_2), 0.88 (t, ^3J(H,H) = 6.9, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 172.2, 158.7, 158.0, 155.7, 140.8, 133.0, 130.7, 130.4 (2C), 130.3, 129.1, 127.9 (2C), 119.6, 114.7 (2C), 113.9 (2C), 111.7, 71.3, 70.6 (2C), 70.1, 69.7, 68.7, 68.5, 68.1, 68.0, 31.9 (2C), 29.6, 29.6, 29.5, 29.5, 29.4, 29.3, 26.1, 26.1, 22.7 (2C), 14.2 (2C). EA: $C_{42}H_{60}O_8$ (Cal.) C: 72.80 %, H: 8.73 %, (Found) C: 72.58 %, H: 8.65 %.

14-(4,4''-Dioctyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoic acid (**H**8/4): See ref. [3]

5-(4,4''-Didecyloxy-p-terphenyl-2'-yloxy)-3-oxapentanoic acid (**H**10/1): Synthesized form **Na**10/1 (0.34 g, 0.52 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield: 0.23 g (0.348 mmol, 67 %), colorless solid; transitions/°C: Cr < 20 LC 147 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.48 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.34 (d, ^3J(H,H) = 7.7, 1 H, Ar-H), 7.21 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.19-4.16 (m, 2 H, OCH_2), 4.11 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.87 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.50-1.27 (m, 28 H, CH_2), 0.90-0.86 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 173.1, 158.8, 158.2, 155.6, 141.0, 133.0, 130.9, 130.4 (2C), 130.1, 129.5, 127.9 (2C), 120.1, 114.8 (2C), 113.9 (2C), 112.0, 70.4, 68.5, 68.4, 68.2, 68.1, 32.0 (2C), 29.7, 29.7 (2C), 29.5, 29.5, 29.4, 29.4 (4C), 26.2, 26.2, 22.8 (2C), 14.2 (2C). EA: $C_{42}H_{60}O_6$ (Cal.) C: 76.32 %, H: 9.15 %, (Found) C: 76.54 %, H: 9.06 %.

8-(4,4''-Didecyloxy-p-terphenyl-2'-yloxy)-3,6-dioxaoctanoic acid (**H**10/2): Synthesized from **Na**10/2 (1.0 g, 1.4 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield: 0.75 g (1.06 mmol, 77 %), colorless solid; transitions/°C: Cr 77 LC 90 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.48 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.20 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.11 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 4.07 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.2, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.2, 2 H, OCH_2), 3.83 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 3.66-3.61 (m, 4 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.50-1.27 (m, 28 H, CH_2), 0.87 (t, ^3J(H,H) = 6.8, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.6, 158.9, 158.2, 155.7, 141.1, 133.1, 130.9, 130.6 (2C), 130.4, 129.3, 128.0 (2C), 119.9, 114.8 (2C), 113.9 (2C), 111.7, 71.5, 70.4, 69.9, 68.7, 68.3, 68.1, 68.1, 31.9 (2C), 29.6 (2C), 29.6, 29.6, 29.6, 29.4, 29.4, 29.3, 29.3, 29.3, 26.1, 26.1, 22.7 (2C), 14.1 (2C). EA: $C_{44}H_{64}O_7$ (Cal.) C: 74.96 %, H: 9.15 %, (Found) C: 75.04 %, H: 9.04 %.

11-(4,4''-Didecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoic acid (**H**10/3): Synthesized from **Na**10/3 (3.4 g, 4.4 mmol). Purified by repeated crystallized from ethyl acetate/n-hexane. Yield 2.5 g (76 %); colorless solid; m.p. = 56 °C. ^{1}H NMR (acetone-D_6, *J*/Hz, 400 MHz) δ = 7.56 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 7.50 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 7.27 (d, ^3J(H,H) = 7.8, 1 H, Ar-H), 7.22 (d, ^4J(H,H) = 1.6, 1 H, Ar-H), 7.16 (dd, ^3J(H,H) = 7.8, ^4J(H,H) = 1.6, 1 H, Ar-H), 6.93

(d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.88 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 4.01 (s, 2 H, OCH_2), 3.98-3.94 (m, 4 H, OCH_2), 3.73 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 3.60-3.51 (m, 8 H, OCH_2), 1.75-1.68 (m, 4 H, CH_2), 1.44-1.39 (m, 4 H, CH_2), 1.28-1.21 (m, 24 H, CH_2), 0.80 (t, ^3J(H,H) = 6.8, 6 H, CH_3). EA: $C_{46}H_{68}NO_8$ (Cal.) C: 73.76 %, H: 9.15 %, (Found) C: 70.56 %, H: 8.99 %.

14-(4,4"-Didecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoic acid (**H**10/4): Synthesized from **Na**10/4 (3.0 g, 3.7 mmol). Purified by repeated crystallized from ethyl acetate/n-hexane. Yield 2.3 g (79 %); transitions/°C: Cr 45 M 51 Col 52 Iso. ^{1}H-NMR (acetone-d_6, *J*/Hz, 400 MHz) δ = 11.0 (bs, 1 H, OH), 7.63 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.57 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 7.35 (d, ^3J(H,H) = 8.0, 1 H, Ar-H), 7.29 (d, ^4J(H,H) = 1.8, 1 H, Ar-H), 7.23 (dd, ^3J(H,H) = 8.0, ^4J(H,H) = 1.8, 1 H, Ar-H), 7.00 (d, ^3J(H,H) = 9.0, 2 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.24 (t, ^3J(H,H) = 4.5, 2 H, OCH_2), 4.08 (s, 2 H, OCH_2), 4.04-4.00 (m, 4 H, OCH_2), 3.81-3.78 (m, 2 H, OCH_2), 3.67-3.56 (m, 12 H, OCH_2), 1.81-1.75 (m, 4 H, CH_2), 1.51-1.29 (m, 28 H, CH_2), 0.90-0.85 (m, 6 H, CH_3). (acetone-d_6, 100 MHz) δ = 171.5, 159.8, 159.1, 157.1, 141.5, 133.7, 131.3, 131.3 (2C), 129.6, 128.7 (2C), 119.8, 115.6 (2C), 114.7 (2C), 114.7, 111.9, 71.4, 71.4, 71.3, 71.2, 71.2, 71.1, 70.3, 69.0, 68.7, 68.6, 68.5, 32.6 (2C), 26.8, 26.8, 23.3 (2C), 14.4 (2C), other signals overlapped by acetone. $C_{48}H_{72}O_9 \cdot 0.5H_2O$ (Cal.) C: 71.87 %, H: 9.17 %, (Found) C: 71.88 %, H: 9.46 %.

11-[4,4''-Bis(3,7-dimethyloctyloxy)-p-terphenyl-2'-yloxy]-3,6,9-trioxaundecanoic acid (**H**10*/3): Synthesized from **Na**10*/3 (0.45 g, 0.58 mmol). Purified by repeated crystallization from n-hexane; yield 0.25 g (64 %); colorless liquid. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.53 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.52 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.20 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.15 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.97 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.10 (s, 2 H, OCH_2), 4.05-3.99 (m, 4 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.68-3.60 (m, 8 H, OCH_2), 1.87-1.80 (m, 2 H, CH), 1.72-1.64 (m, 2 H, CH), 1.61-1.45 (m, 4 H, CH_2), 1.39-1.07 (m, 12 H, CH_2), 0.96 (d, ^3J(H,H) = 6.4, 6 H, CH_3), 0.88 (d, ^3J(H,H) = 6.6, 12 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 172.3, 158.7, 158.1, 155.8, 140.9, 133.1, 130.8, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.8, 71.2, 70.6 (2C), 70.1, 69.7, 68.7, 68.5, 66.5, 66.4, 39.3 (2C), 37.3, 37.3, 36.3, 36.3, 29.9 (2C), 28.0 (2C) , 24.7 (2C), 22.7 (2C), 22.6 (2C), 19.7 (2C).

14-[4,4''-Bis(3,7-dimethyloctyloxy)-p-terphenyl-2'-yloxy]-3,6,9,12-tetraoxa-tetra-decanoic acid (**H**10*/4): Synthesized from **Na**10*/4 (0.60 g, 0.74 mmol). Purified by repeated crystallization from n-hexane; yield 0.30 g (51 %); colorless liquid. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.53 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 7.52 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.14 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 9.0, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 4.9, 2 H, OCH_2), 4.08 (s, 2 H, OCH_2), 4.04-4.01 (m, 4 H, OCH_2), 3.80 (t, ^3J(H,H) = 4.9, 2 H, OCH_2), 3.67-3.56 (m, 12 H, OCH_2), 1.90-1.79 (m, 2 H, CH), 1.74-1.63 (m, 2 H, CH), 1.62-1.47 (m, 4 H, CH_2), 1.37-1.12 (m, 12 H, CH_2), 0.95 (d, ^3J(H,H) = 6.6, 6 H, CH_3), 0.87 (d, ^3J(H,H) = 6.6, 12 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.8, 158.7, 158.0, 155.8, 140.8, 133.1, 130.7, 130.4 (2C), 130.3, 129.0, 127.9 (2C), 119.6, 114.7 (2C), 113.9 (2C), 111.6, 71.1, 70.9, 70.5, 70.4, 70.2, 70.2, 69.7, 69.0, 68.3, 66.5, 66.4, 39.3 (2C), 37.4, 37.3, 36.3, 36.3, 30.0, 29.9, 28.0 (2C) , 24.7 (2C), 22.8 (2C), 22.7 (2C), 19.7 (2C).

11-(4,4''-Dihexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoic acid (**H**16/3): Synthesized from **Na**16/3 (1.1 g, 1.2 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield: 0.75 g (70 %), colorless solid; transitions/°C: Cr 79 LC 100 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.50 (d, ^3J(H,H) = 9.0, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 9.0, 2 H, Ar-H), 4.16 (t, 2 H, ^3J(H,H) = 5.0, OCH_2), 4.08 (s, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.78 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.68-3.59 (m, 8 H, OCH_2), 1.80-1.76 (m, 4 H, CH_2), 1.47-1.42 (m, 4 H, CH_2), 1.41-1.20 (m, 48 H, CH_2), 0.88-0.84 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.1, 158.8, 158.1, 155.8, 140.9, 133.1, 130.8, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.8, 71.6, 70.8, 70.7, 70.0, 69.8, 69.8, 68.6, 68.2, 68.1, 32.0 (2C), 29.8 (4C), 29.7 (4C), 29.7 (4C), 29.7 (4C), 29.5, 29.5, 29.4 (3C), 29.4, 26.2, 26.2, 22.8 (2C), 14.2 (2C). EA: $C_{58}H_{92}O_8 \cdot 0.3H_2O$ (Cal.) C: 75.49 %, H: 10.16 %, (Found) C: 75.47 %, H: 10.19 %.

14-(4,4''-Dihexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoic acid (**H**16/4): Synthesized from **Na**16/4 (1.2 g, 1.2 mmol). Yield 1.11 g (95 %); transition temperatures: Cr 74 SmA 89 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.17 (t, 2 H, ^3J(H,H) = 5.0, OCH_2), 4.08 (s, 2 H, OCH_2COO), 4.00-3.96 (m, 4 H, OCH_2), 3.80 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.70-3.57 (m, 12 H, OCH_2), 1.81-1.76 (m, 4 H, CH_2), 1.48-1.41 (m, 4 H, CH_2), 1.40-1.21 (m, 48 H, CH_2), 0.86 (t, ^3J(H,H) = 6.8, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.1, 158.8, 158.2, 155.9, 140.9, 133.2, 130.8, 130.6 (2C), 130.5, 129.1, 128.0 (2C), 119.7, 114.8 (2C), 114.0 (2C), 111.7, 71.6, 71.0, 70.7, 70.6, 70.3, 70.2, 69.7, 69.2, 68.4, 68.2, 68.1, 32.0 (2C), 29.7 (8C), 29.7 (4C), 29.7 (2C), 29.5 (2C), 29.5 (2C), 29.4 (2C), 29.4 (2C), 26.2, 26.1, 22.7 (2C), 14.1 (2C). EA: $C_{60}H_{96}O_9 \cdot 0.5H_2O$ (Cal.) C: 74.26 %, H: 10.07 %, (Found) C: 74.31 %, H: 9.75 %.

11-(4,4''-Didecyloxy-p-terphenyl-3-yloxy)-3,6,9-trioxaundecanoic acid (**H***10/3): Synthesized from **Na***10/3 (155 mg, 0.20 mmol). Purified by epeated crystallization from ethyl acetate/n-hexane; yield 59 mg (39 %); colorless solid; transitions/°C: Cr 76 LC 107 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.58 (s, 4 H, Ar-H), 7.53 (d, ^3J(H,H) = 8.5, 2 H, Ar-H), 7.20-7.15 (m, 2 H, Ar-H), 6.97-6.92 (m, 3 H, Ar-H), 4.25 (t, ^3J(H,H) = 4.9, 2 H, OCH_2), 4.11 (s, 2 H, OCH_2), 4.02 (t, ^3J(H,H) = 7.5, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.7, 2 H, OCH_2), 3.89 (t, ^3J(H,H) = 4.9, 2 H, OCH_2), 3.80-3.63 (m, 8 H, OCH_2), 1.90-1.75 (m, 4 H, CH_2), 1.52-1.41 (m, 4 H, CH_2), 1.39-1.18 (m, 24 H, CH_2), 0.87 (t, ^3J(H,H) = 6.7, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.1, 158.6, 148.8, 148.7, 139.2, 139.0, 133.7, 132.9, 127.8 (2C), 127.0 (2C), 126.9 (2C), 120.1, 114.8 (2C), 113.9, 110.0, 71.6, 70.9, 70.7, 70.2, 70.0, 69.4, 69.3, 69.0, 68.2, 32.0 (2C), 29.7 (2C), 29.7 (3C), 29.6, 29.5, 29.4 (3C), 26.2, 26.1, 22.8 (2C), 14.2 (2C). EA: $C_{46}H_{68}O_8 \cdot 0.4H_2O$ (Cal.) C: 73.06 %, H: 9.17 %, (Found) C: 73.06 %, H: 8.93 %.

14-(4,4''-Didecyloxy-p-terphenyl-3-yloxy)-3,6,9,12-tetraoxatetradecanoic acid (**H***10/4): Synthesized from **Na***10/4 (165 mg, 0.20 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield 82 mg (51 %); colorless solid; transitions/°C: Cr 65 LC 92 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.57 (s, 4 H, Ar-H), 7.53 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.20 (d,

^4J(H,H) = 2.1, 1 H, Ar-H), 7.17 (dd, ^3J(H,H) = 8.4, ^3J(H,H) = 2.1, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.5, 1 H, Ar-H), 4.24 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 4.10 (s, 2 H, OCH_2), 4.01 (t, ^3J(H,H) = 6.3, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.89 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 3.80-3.63 (m, 12 H, OCH_2), 1.90-1.75 (m, 4 H, CH_2), 1.52-1.41 (m, 4 H, CH_2), 1.39-1.18 (m, 24 H, CH_2), 0.87 (t, ^3J(H,H) = 6.9, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.3, 158.6, 148.9, 148.7, 139.2, 139.0, 133.7, 132.9, 127.8 (2C), 126.9 (2C), 126.8 (2C), 120.1, 114.8 (2C), 114.0, 114.0, 71.6, 71.0, 70.7, 70.6, 70.4, 70.2, 69.9, 69.3, 69.3, 69.1, 68.1, 32.0 (2C), 29.7, 29.7 (2C), 29.6, 29.5, 29.5, 29.4 (2C), 29.4 (2C), 26.2, 26.1, 22.8 (2C), 14.2 (2C). EA: $C_{48}H_{72}O_9 \cdot 0.5H_2O$ (Cal.) C: 71.87 %, H: 9.17 %, (Found) C: 71.98 %, H: 9.12 %.

11-(4-Hexoxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoic acid (**H**6.16/3): Synthesized from **Na**6.16/3 (250 mg, 0.31 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield: 89 mg (37 %), colorless solid; m.p. = 68 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.51 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.14 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 4.8, 2 H, OCH_2), 4.10 (s, 2 H, OCH_2), 4.00-3.97 (m, 4 H, OCH_2), 3.79 (t, ^3J(H,H) = 4.8, 2 H, OCH_2), 3.69-3.60 (m, 8 H, OCH_2), 1.83-1.76 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.93-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 172.2, 158.8, 158.1, 155.9, 140.9, 133.1, 130.8, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.7, 71.4, 70.7, 70.7, 70.1, 69.8, 68.7, 68.5, 68.1, 68.1, 31.9, 31.6, 29.7 (3C), 29.7 (2C), 29.7 (2C), 29.6, 29.6, 29.4, 29.4, 29.3, 26.1, 25.8, 22.7, 22.6, 14.0, 14.0. EA: $C_{48}H_{72}O_8$ (Cal.) C: 74.19 %, H: 9.34 %, (Found) C: 74.10 %, H: 9.31 %.

14-(4-Hexoxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoic acid (**H**6.16/4): Synthesized from **Na**6.16/4 (0.45 g, 0.53 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield: 0.25 g (57 %), colorless solid; M.p. = 66 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.52 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.09 (s, 2 H, OCH_2), 4.00-3.96 (m, 4 H, OCH_2), 3.80 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.69-3.56 (m, 12 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H, CH_2), 0.93-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 172.0, 158.8, 158.1, 155.9, 140.9, 133.1, 130.7, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.6, 71.3, 70.9, 70.6, 70.5, 70.3, 70.2, 69.7, 68.9, 68.3, 68.1, 68.0, 31.9, 31.6, 29.7 (3C), 29.7 (2C), 29.6 (2C), 29.6, 29.6, 29.4, 29.3, 29.3, 26.1, 25.8, 22.7, 22.6, 14.1, 14.0. EA: $C_{50}H_{76}O_9$ (Cal.) C: 73.13 %, H: 9.33 %, (Found) C: 73.09 %, H: 9.25 %.

20-(4-Hexoxy-4''-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoic acid (**H**6.16/6): Synthesized from **Na**6.16/6 (0.55 g, 0.59 mmol). Purified by repeated crystalization from ethyl acetate/n-hexane; yield: 0.30 g (0.33 mmol, 56 %), colorless solid; m.p. = 52 °C. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.10 (bs, 1 H, OH), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.11 (s, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.70- 3.66 (m, 2 H, OCH_2), 3.63-3.57 (m, 18 H, OCH_2), 1.83-1.75 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.22 (m, 28 H,

CH_2), 0.92-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.7, 158.7, 158.1, 155.9, 140.8, 133.1, 130.7, 130.5 (2C), 130.3, 129.1, 127.9 (2C), 119.5, 114.8 (2C), 113.9 (2C), 111.6, 71.1, 70.8, 70.6 (3C), 70.5 (2C), 70.4, 70.3, 70.2, 69.7, 69.0, 68.3, 68.1, 68.0, 31.9, 31.6, 29.7 (3C), 29.7 (2C), 29.6 (2C), 29.6 (2C), 29.4, 29.4, 29.3, 26.1, 25.8, 22.7, 22.6, 14.1, 14.1. EA: $C_{54}H_{84}O_{11}\cdot H_2O$ (Cal.) C: 69.95 %, H: 9.35 %, (Found) C: 70.25 %, H: 9.28 %.

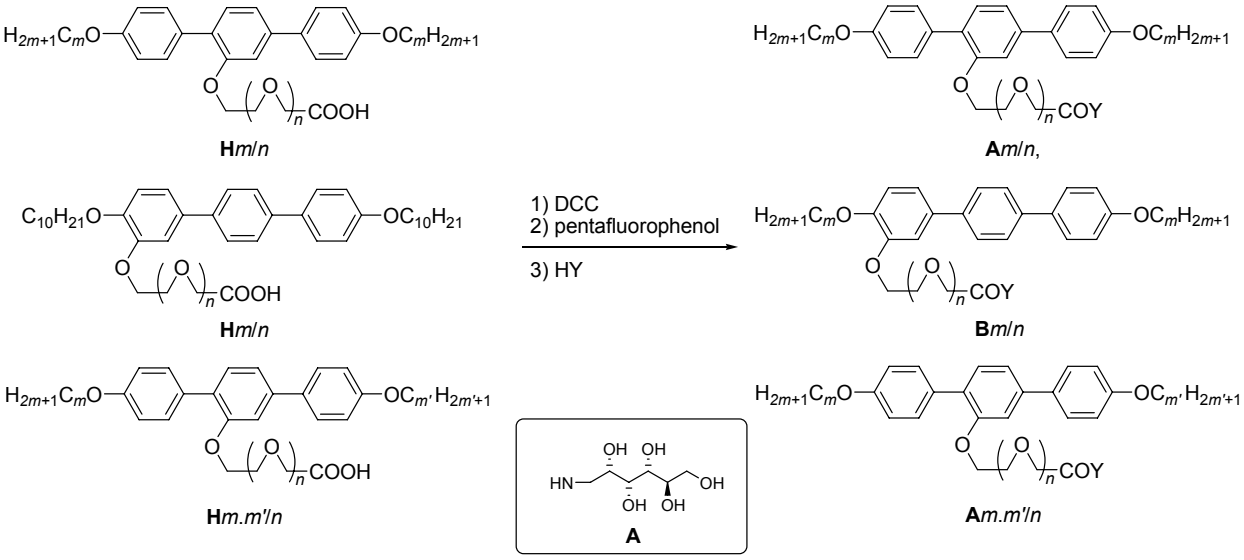
11-(4-Hexadecyloxy-4''-hexoxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoic acid (**H**16.6/3): Synthesized from **Na**16.6/3 (0.50 g, 0.63 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield 0.29 g (60 %); colorless solid; transitions/°C: Cr 48 LC 63 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.52 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.51 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.14 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.0 (bs, 1 H, OH), 4.16 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.10 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.68-3.60 (m, 8 H, OCH_2), 1.82-1.76 (m, 4 H, CH_2), 1.49-1.42 (m, 4 H, CH_2), 1.40-1.20 (m, 28 H, CH_2), 0.96-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.9, 158.8, 158.1, 155.9, 140.9, 133.1, 130.8, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.8, 71.5, 70.7, 70.7, 70.1, 69.8, 68.8, 68.5, 68.2, 68.1, 32.0, 31.6, 29.7 (4C), 29.7 (2C), 29.7, 29.6, 29.5, 29.4, 29.4, 29.3, 26.1, 25.8, 22.7, 22.6, 14.1, 14.1. EA: $C_{48}H_{72}O_8\cdot 0.5H_2O$ (Cal.) C: 73.34 %, H: 9.36 %, (Found) C: 73.64 %, H: 9.31 %.

14-(4-Hexadecyloxy-4''-hexoxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoic acid (**H**16.6/4): Synthesized from **Na**16.6/4 (0.50 g, 0.59 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield 0.33 g (68 %); colorless solid; transitions/°C: Cr_2 47 LC 61 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.53 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.51 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.14 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.96 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.91 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.00 (bs, 1 H, OH), 4.17 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 4.08 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.98 (t, ^3J(H,H) = 6.5, 2 H, OCH_2), 3.80 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.68-3.57 (m, 12 H, OCH_2), 1.83-1.76 (m, 4 H, CH_2), 1.49-1.42 (m, 4 H, CH_2), 1.40-1.20 (m, 28 H, CH_2), 0.96-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.8, 158.8, 158.1, 155.9, 140.9, 133.1, 130.7, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.6, 71.2, 70.9, 70.6, 70.5, 70.3, 70.2, 69.7, 69.0, 68.4, 68.1, 68.0, 31.9, 31.6, 29.7 (4C), 29.7, 29.6, 29.6, 29.5, 29.4, 29.4 (2C), 29.3, 26.1, 25.8, 22.7, 22.6, 14.1, 14.1. EA: $C_{50}H_{76}O_9\cdot H_2O$ (Cal.) C: 71.56 %, H: 9.37 %, (Found) C: 71.81 %, H: 9.22 %.

20-(4-Hexadecyloxy-4''-hexoxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoic acid (**H**16.6/6): Synthesized from **Na**16.6/6 (0.50 g, 0.54 mmol). Purified by repeated crystallization from ethyl acetate/n-hexane; yield 0.36 g (74 %); colorless solid; transitions/°C: Cr 34 LC 46 Iso. ^{1}H-NMR ($CDCl_3$, *J*/Hz, 500 MHz) δ = 7.52 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.51 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.5, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.9, 2 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.16 (t, ^3J(H,H) = 5.2, 2 H, OCH_2), 4.10 (s, 2 H, OCH_2), 3.99 (t, ^3J(H,H) = 6.4, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.4, 2 H, OCH_2), 3.79 (t, ^3J(H,H) = 5.2, 2 H, OCH_2), 3.69-3.57 (m, 20 H, OCH_2), 1.82-1.75 (m, 4 H, CH_2), 1.49-1.42 (m, 4 H, CH_2), 1.40-1.20 (m, 28 H, CH_2), 0.92-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 125 MHz) δ = 171.9, 159.1, 158.4, 156.2, 141.1, 133.5, 131.1, 130.8 (2C), 130.6, 129.3, 128.2 (2C), 119.9, 115.0 (2C), 114.2 (2C), 111.9, 71.5, 71.1,

70.9 (3C), 70.7 (2C), 70.6, 70.6, 70.5, 69.9, 69.4, 68.6, 68.4, 68.3, 32.2, 31.8, 29.9 (4C), 29.9 (2C), 29.9, 29.9, 29.7, 29.6, 29.6, 29.5, 26.4, 26.0, 22.9, 22.9, 14.4, 14.3. EA: $C_{54}H_{84}O_{11}\cdot 0.5H_2O$ (Cal.) C: 70.63 %, H: 9.33 %, (Found) C: 70.87 %, H: 9.32 %.

2.5. Synthesis of the amides (A/*m*/*n*, B/10/*n*, A/6.16/*n* and A/16.6/*n*)



Scheme S5. Synthesis of the amides.

General procedure: The appropriate carboxylic acid **H**/*m*/*n* (or **H***/10/*n*, **H**/6.16/*n*, **H**/16.6/*n*) was dissolved in dry THF (50 mL), to which, DCC was added at 0 °C. After 30 min, the reaction mixture was allowed to warm to r.t., then pentafluorophenol was added, and the reaction mixture was stirred at r.t. for 24h. 1-amino-1-deoxy-D-sorbitol was added and the resulting mixture was stirred for another 24 h. After that the reaction mixture was filtered and the residue was washed with chloroform (5 × 5 mL). The solvent of the combined organic solution was evaporated in reduced pressure, the residue was purified by column chromatography on silica gel and repeated crystallization from appropriate solvent. The transition temperatures are collated in Tables 1-3 of the main text.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-8-(4,4"-dibutyloxy-p-terphenyl-2'-yl-oxy)-3,6-dioxaoctanoylamide (**A**4/2): Synthesized from **H**4/2 (0.40 g, 0.75 mmol), DCC (170 mg, 0.83 mmol), pentafluorophenol (170 mg, 0.91 mmol) and 1-amino-1-deoxy-D-sorbitol (0.69 g, 3.8 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield 0.18 g (34.5 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.49-7.43 (m, 5 H, Ar-H, NH), 7.27 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.14 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.08 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.90 (d, ^3J(H,H) = 9.1, 2 H, Ar-H), 6.86 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.09 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.94 (t, ^3J(H,H) = 6.4, 2 H, OCH_2), 3.93 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.87 (s, 2 H, OCH_2), 3.80-3.0 (m, 19 H, OCH_2, OCH, NCH_2, OH), 1.79-1.69 (m, 4 H, CH_2), 1.50-1.40 (m, 4 H, CH_2), 0.95 (t, ^3J(H,H) = 7.3, 3 H, CH_3), 0.94 (t, ^3J(H,H) = 7.5, 3 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.6, 158.7, 158.1, 155.7, 140.8, 132.9, 130.8, 130.4 (2C), 130.2, 129.0, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.5, 72.8, 72.4, 71.9, 70.9, 70.4, 70.2, 70.2, 69.8, 68.5, 67.8, 67.8, 63.8, 42.2, 31.5, 31.4, 19.4, 19.4,

14.0, 14.0. EA: $C_{38}H_{53}NO_{11}\cdot H_2O$ (Cal.) C: 63.58 %, H: 7.7.72 %, N: 1.95 %, (Found) C: 63.44 %, H: 7.45 %, N: 1.99 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-11-(4,4"-dibutyloxy-p-terphenyl-2'-yl-oxy)-3,6,9-trioxaundecanoylamide (**A**4/3): Synthesized from **H**4/3 (0.45 g, 0.78 mmol), DCC (180 mg, 0.86 mmol), pentafluorophenol (180 mg, 0.95 mmol) and 1-amino-1-deoxy-D-sorbitol (0.71 g, 3.9 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.23 g (40 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.57 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.49 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 7.47 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 7.29 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.15 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.10 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.88 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.11 (t, ^3J(H,H) = 4.9, 2 H, OCH_2), 3.96 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.95 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.92 (s, 2 H, OCH_2), 3.85-3.1 (m, 26 H, OCH_2, OCH, NCH_2, OH, 1,5 H_2O), 1.79-1.70 (m, 4 H, CH_2), 1.52-1.42 (m, 4 H, CH_2), 0.95 (t, ^3J(H,H) = 7.3, 3 H, CH_3), 0.95 (t, ^3J(H,H) = 7.6, 3 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.7, 158.7, 158.1, 155.7, 140.9, 133.0, 130.8, 130.5 (2C), 130.4, 129.0, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.6, 72.9, 72.5, 71.9, 70.9, 70.7, 70.5, 70.2, 70.1, 70.1, 69.7, 68.5, 67.8, 67.8, 63.9, 42.2, 31.5, 31.4, 19.4, 19.4, 14.0, 14.0. EA: $C_{40}H_{57}NO_{12}\cdot 1.5\ H_2O$ (Cal.) C: 62.32 %, H: 7.85 %, N: 1.82 %, (Found) C: 62.59 %, H: 7.64 %, N: 1.91 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-14-(4,4"-dibutyloxy-p-terphenyl-2'-yl-oxy)-3,6,9,12-tetraoxatetradecanoylamide (**A**4/4): Synthesized from **H**4/4 (0.56 g, 0.90 mmol), DCC (200 mg, 0.99 mmol), pentafluorophenol (200 mg, 1.09 mmol) and 1-amino-1-deoxy-D-sorbitol (0.81 g, 4.5 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.29 g (41 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.61 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.50 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.17 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.11 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.13 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.97 (t, ^3J(H,H) = 6.4, 2 H, OCH_2), 3.96 (t, ^3J(H,H) = 6.4, 2 H, OCH_2), 3.93 (s, 2 H, OCH_2), 3.85-3.3 (m, 31 H, OCH_2, OCH, NCH_2, OH, 2 H_2O), 1.80-1.70 (m, 4 H, CH_2), 1.53-1.42 (m, 4 H, CH_2), 0.96 (t, ^3J(H,H) = 7.3, 3 H, CH_3), 0.96 (t, ^3J(H,H) = 7.3, 3 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.6, 158.7, 158.0, 155.8, 140.8, 133.0, 130.8, 130.5 (2C), 130.3, 129.0, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.6, 72.9, 72.5, 71.9, 70.9, 70.8, 70.4, 70.4, 70.3, 70.2, 70.1, 70.1, 69.7, 68.5, 67.8, 67.7, 63.9, 42.2, 31.5, 31.4, 19.4, 19.4, 14.0, 14.0. EA: $C_{42}H_{61}NO_{13}\cdot 2\ H_2O$ (Cal.) C: 61.22 %, H: 7.95 %, N: 1.70 %, (Found) C: 61.16 %, H: 7.89 %, N: 1.70 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-11-(4,4"-dibutyloxy-p-terphenyl-2'-yl-oxy)-3,6,9-trioxaundecanoylamide (**A**6/3): Snythesized from **H**6/3 (0.80 g, 1.26 mmol), DCC (286 mg, 1.39 mmol), pentafluorophenol (278 mg, 1.51 mmol), 1-amino-1-deoxy-D-sorbitol (2.3 g, 12.6 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.43 g (43 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.57 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.50 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.48 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.17 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.5, 1 H, Ar-H), 7.11 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.12 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.96 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.95 (t, ^3J(H,H) = 6.6, 2 H, OCH_2), 3.92 (s, 2 H, OCH_2), 3.85-3.4 (m, 18 H, OCH_2, OCH, NCH_2), 3.11-

(bs, 8 H, OH, 1.5 H_2O), 1.82-1.73 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 8 H, CH_2), 0.89 (t, ^3J(H,H) = 6.8, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.8, 158.8, 158.1, 155.8, 141.0, 133.0, 130.9, 130.5 (2C), 130.4, 128.9, 128.0 (2C), 119.8, 114.8 (2C), 114.0 (2C), 111.7, 73.0, 72.7, 71.9, 70.9, 70.7, 70.5, 70.1, 70.1, 70.0, 69.7, 68.5, 68.2, 68.1, 63.9, 42.2, 31.6 (2C), 29.3, 29.3, 25.8, 25.8, 22.6 (2C), 14.1 (2C). EA: $C_{44}H_{65}NO_{12}$·1.5 H_2O (Cal.) C: 63.90 %, H: 8.29 %, N: 1.69 %, (Found) C: 63.94 %, H: 8.29 %, N: 1.54 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-14-(4,4"-dihexyloxy-p-terphenyl-2'-yl-oxy)-3,6,9,12-tetraoxatetradecanoylamide (**A**6/4): See ref. [3].

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-17-(4,4"-dihexyloxy-p-terphenyl-2'-yl-oxy)-3,6,9,12,15-pentaoxaheptadecanoylamide (**A**6/5): Synthesized from **H**6/5 (0.38 g, 0.52 mmol), DCC (118 mg, 0.57 mmol), pentafluorophenol (115 mg, 0.62 mmol) and 1-amino-1-deoxy-D-sorbitol (1.0 g, 5.5 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.20 g (43 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.72 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.17 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.14 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.99-3.95 (m, 6 H, OCH_2), 3.9-3.1 (m, 34 H, OCH_2, OCH, NCH_2, OH, 1.5 H_2O), 1.82-1.73 (m, 4 H, CH_2), 1.48-1.41 (m, 4 H, CH_2), 1.39-1.30 (m, 8 H, CH_2), 0.88 (t, ^3J(H,H) = 7.1, 6 H, CH_3). EA: $C_{48}H_{73}NO_{14}$·1.5 H_2O (Cal.) C: 63.00 %, H: 8.37 %, N: 1.53 %, (Found) C: 63.10 %, H: 8.08 %, N: 1.59 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-20-(4,4"-dihexyloxy-p-terphenyl-2'-yl-oxy)-3,6,9,12,15,18-hexaoxaeicosanoylamide (**A**6/6): Synthesized from **H**6/6 (0.40 g, 0.52 mmol), DCC (118 mg, 0.57 mmol), pentafluorophenol (115 mg, 0.62 mmol) and 1-amino-1-deoxy-D-sorbitol (1.0 g, 5.5 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.18 g (37 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.70 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.17 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.14 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.99-3.95 (m, 6 H, OCH_2), 3.9-3.2 (m, 39 H, OCH_2, OCH, NCH_2, OH, 2 H_2O), 1.82-1.73 (m, 4 H, CH_2), 1.48-1.41 (m, 4 H, CH_2), 1.39-1.30 (m, 8 H, CH_2), 0.89 (t, ^3J(H,H) = 7.1, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.6, 158.7, 158.0, 155.8, 140.8, 133.1, 130.7, 130.5 (2C), 130.3, 129.0, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.6, 73.0, 72.5, 71.9, 70.9, 70.8, 70.6, 70.5, 70.4 (3C), 70.4, 70.3, 70.2, 70.1, 70.1, 69.7, 68.4, 68.2, 68.1, 63.9, 42.2, 31.7, 31.7, 29.4, 29.4, 25.9, 25.8, 22.7 (2C), 14.1 (2C). EA: $C_{50}H_{77}NO_{15}$·2 H_2O (Cal.) C: 62.03 %, H: 8.43 %, N: 1.45 %, (Found) C: 62.01 %, H: 8.42 %, N: 1.32 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-11-(4,4"-dioctyloxy-p-terphenyl-2'-yl-oxy)-3,6,9-trioxaundecanoylamide (**A**8/3): Synthesized from **H**8/3 (0.24 g, 0.35 mmol), DCC (80 mg, 0.39 mmol), pentafluorophenol (79 mg, 0.43 mmol) and 1-amino-1-deoxy-D-sorbitol (0.33 g, 1.8 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.15 g (50.5 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.58 (bs, 1 H, NH), 7.50 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.48 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.7, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.7, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.12

(d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.14 (t, ^3J(H,H) = 4.5, 2 H, OCH_2), 3.98-3.93 (m, 6 H, OCH_2), 3.9-3.3 (m, 18 H, OCH_2, OCH, NCH_2), 2.89 (bs, 6 H, OH, 0.5 H_2O), 1.79-1.73 (m, 4 H, CH_2), 1.49-1.40 (m, 4 H, CH_2), 1.39-1.20 (m, 16 H, CH_2), 0.87 (t, ^3J(H,H) = 6.8, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.9, 158.8, 158.1, 155.7, 140.9, 133.0, 130.8, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.8, 114.8 (2C), 114.0 (2C), 111.7, 73.1, 72.9, 72.0, 71.0, 70.7, 70.5, 70.1 (2C), 70.0, 69.7, 68.6, 68.2, 68.1, 63.9, 42.4, 32.0, 31.9, 31.0, 29.6, 29.5, 29.4, 29.4, 29.4, 26.2, 26.2, 22.8 (2C), 14.2 (2C). EA: $C_{48}H_{73}NO_{12}$·0.5 H_2O (Cal.) C: 66.64 %, H: 8.62 %, N: 1.62 %, (Found) C: 66.68 %, H: 8.69 %, N: 1.64 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-5-(4,4"-didecyloxy-p-terphenyl-2'-yl-oxy)-3-oxapentanoylamide (**A**10/1): Synthesized from **H**10/1 (0.28 g, 0.43 mmol), DCC (97 mg, 0.47 mmol), pentafluorophenol (95 mg, 0.52 mmol) and 1-amino-1-deoxy-D-sorbitol (0.40 g, 2.2 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystallization from ethyl acetate; yield: 0.19 g (51 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.45 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.42 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.26 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.14 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.10-7.05 (m, 2 1 H, NH, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.86 (d, ^3J(H,H) = 9.1, 2 H, Ar-H), 4.16-4.06 (bt, 2 H, OCH_2), 3.93-3.87 (m, 6 H, OCH_2), 3.93-3.1 (m, 15 H, OCH_2, OCH, NCH_2, OH), 1.79-1.68 (m, 4 H, CH_2), 1.44-1.36 (m, 4 H, CH_2), 1.35-1.20 (m, 24 H, CH_2), 0.86 (t, ^3J(H,H) = 6.8, 3 H, CH_3), 0.85 (t, ^3J(H,H) = 6.8, 3 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.4, 158.8, 158.2, 155.5, 140.9, 132.8, 131.0, 130.3 (2C), 130.2, 129.2, 127.8 (2C), 120.0, 114.8 (2C), 114.2 (2C), 111.9, 73.2, 72.6, 71.9, 71.0, 70.4, 70.3, 70.1, 68.2, 68.0, 63.9, 42.2, 32.0 (2C), 29.7, 29.7 (3C), 29.6, 29.6, 29.5 (2C), 29.4 (2C), 26.2 (2C), 22.8 (2C), 14.2 (2C). EA: $C_{48}H_{73}NO_{10}$ (Cal.) C: 69.96 %, H: 8.93 %, N: 1.70 %, (Found) C: 69.93 %, H: 8.83 %, N: 1.77 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-8-(4,4"-didecyloxy-p-terphenyl-2'-yl-oxy)-3,6-dioxaoctanoylamide (**A**10/2): Synthesized from **H**10/2 (0.30 g, 0.43 mmol), DCC (97 mg, 0.47 mmol), pentafluorophenol (95 mg, 0.52 mmol) and 1-amino-1-deoxy-D-sorbitol (0.40 g, 2.2 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystallization from ethyl acetate; yield: 0.19 g (51 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.50 (d, ^3J(H,H) = 9.3, 2 H, Ar-H), 7.48 (d, ^3J(H,H) = 8.6, 2 H, Ar-H), 7.45 (bt, ^3J(H,H) = 5.3, 1 H, NH), 7.32 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.5, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.15 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 3.97-3.93 (m, 4 H, OCH_2), 3.90 (s, 2 H, OCH_2), 3.77 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 3.75-3.2 (m, 12 H, OCH_2, OCH, NCH_2), 2.74 (bs, 7 H, OH, H_2O), 1.83-1.73 (m, 4 H, CH_2), 1.46-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 24 H, CH_2), 0.87 (t, ^3J(H,H) = 6.8, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 172.0, 158.9, 158.2, 155.7, 141.0, 132.9, 130.9, 130.5 (2C), 130.2, 129.1, 127.9 (2C), 119.9, 114.9 (2C), 114.0 (2C), 111.7, 73.1, 73.0, 72.0, 71.0, 70.5, 70.1, 70.0, 69.9, 68.5, 68.2, 68.1, 63.9, 42.3, 31.9 (2C), 29.6 (2C), 29.6 (2C), 29.5, 29.5, 29.4, 29.4 (3C), 26.1, 26.1, 22.7 (2C), 14.1 (2C). EA: $C_{50}H_{77}NO_{11}$·H_2O (Cal.) C: 67.76 %, H: 8.99 %, N: 1.58 %, (Found) C: 67.58 %, H: 8.81 %, N: 1.47 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-11-(4,4"-didecyloxy-p-terphenyl-2'-yl-oxy)-3,6,9-trioxaundecanoylamide (**A**10/3): See ref. [5].

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-14-(4,4”-didecyloxy-p-terphenyl-2’-yl-oxy)-3,6,9,12-tetraoxatetradecanoylamide (**A**10/4): See ref. [5].

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-20-(4,4”-didecyloxy-p-terphenyl-2’-yl-oxy)-3,6,9,12,15,18-hexaoxaeicosanoylamide (**A**10/6): See ref. [5].

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-11-[4,4”-bis(3,7-dimethyloctyloxy)-p-terphenyl-2’-yloxy]-3,6,9-trioxaundecanoylamide (**A**10*/3): Synthesized from **H**10*/3 (0.31 g, 0.41 mmol), DCC (94 mg, 0.45 mmol), pentafluorophenol (92 mg, 0.50 mmol) and 1-amino-1-deoxy-D-sorbitol (0.40 g, 2.2 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate/n-hexane; yield: 0.10 g (26.5 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.58 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.50 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.5, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.90 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.14 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 4.02-3.97 (m, 4 H, OCH_2), 3.93 (s, 2 H, OCH_2), 3.85-3.3 (m, 18 H, OCH_2, OCH, NCH_2), 3.09 (bs, 8 H, OH, 1.5 H_2O), 1.85-1.78 (m, 2 H, CH), 1.73-1.64 (m, 2 H, CH), 1.62-1.45 (m, 4 H, CH_2), 1.37-1.07 (m, 12 H, CH_2), 0.93 (d, ^3J(H,H) = 6.6, 6 H, CH_3), 0.86 (d, ^3J(H,H) = 6.6, 12 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.9, 158.8, 158.2, 155.8, 141.0, 133.1, 130.9, 130.5 (2C), 130.4, 129.1, 128.0 (2C), 119.8, 114.8 (2C), 114.0 (2C), 111.7, 73.0, 72.8, 72.0, 71.0, 70.7, 70.5, 70.2, 70.1, 70.0, 69.7, 68.5, 66.5, 66.4, 63.9, 42.2, 39.3 (2C), 37.4 (2C), 36.3, 36.3, 29.9 (2C), 28.0 (2C), 24.7 (2C), 22.7 (2C), 22.6 (2C), 19.7 (2C). EA: $C_{52}H_{81}NO_{12}\cdot 1.5\ H_2O$ (Cal.) C: 66.50 %, H: 9.01 %, N: 1.49 %, (Found) C: 66.46 %, H: 8.94 %, N: 1.42 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-14-[4,4”-bis(3,7-dimethyloctyloxy)-p-terphenyl-2’-yloxy]-3,6,9,12-tetraoxatetradecanoylamide (**A**10*/4): Synthesized from **H**10*/4 (0.50 g, 0.63 mmol), DCC (144 mg, 0.70 mmol), pentafluorophenol (142 mg, 0.77 mmol) and 1-amino-1-deoxy-D-sorbitol (0.58 g, 3.2 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate/n-hexane; yield: 0.20 g (33 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.62 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.32 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.18 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.90 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.15 (t, ^3J(H,H) = 5.2, 2 H, OCH_2), 4.02-3.98 (m, 4 H, OCH_2), 3.94 (s, 2 H, OCH_2), 3.84-3.3 (m, 29 H, OCH_2, OCH, NCH_2, OH, H_2O), 1.87-1.78 (m, 2 H, CH), 1.73-1.64 (m, 2 H, CH), 1.62-1.45 (m, 4 H, CH_2), 1.37-1.07 (m, 12 H, CH_2), 0.93 (d, ^3J(H,H) = 6.6, 6 H, CH_3), 0.86 (d, ^3J(H,H) = 6.6, 12 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.7, 158.7, 158.0, 155.8, 140.9, 133.0, 130.8, 130.5 (2C), 130.4, 129.0, 127.9 (2C), 119.7, 114.8 (2C), 113.9 (2C), 111.6, 73.0, 72.7, 72.0, 71.0, 70.8, 70.5, 70.4, 70.3, 70.2, 70.1, 70.0, 69.7, 68.5, 66.5, 66.4, 63.9, 42.2, 39.3 (2C), 37.4, 37.4, 36.4, 36.3, 30.0 (2C), 28.1 (2C), 24.8 (2C), 22.8 (2C), 22.7 (2C), 19.8 (2C). EA: $C_{54}H_{85}NO_{13}\cdot H_2O$ (Cal.) C: 66.57 %, H: 9.00 %, N: 1.44 %, (Found) C: 66.38 %, H: 8.69 %, N: 1.52 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-11-(4,4”-dihexadecyloxy-p-terphenyl-2’-yl-oxy)-3,6,9-trioxaundecanoylamide (**A**16/3): Synthesized from **H**16/3 (0.40 g, 0.44 mmol), DCC (99 mg, 0.48 mmol), pentafluorophenol (97 mg, 0.53 mmol) and 1-amino-1-deoxy-D-sorbitol (0.40 g, 2.2 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.18 g (38 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400

MHz) δ = 7.59 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.20 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.13 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.90 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.17 (t, 2 H, ^3J(H,H) = 4.5, OCH_2), 4.00-3.94 (m, 6 H, OCH_2), 3.82-3.3 (m, 18 H, OCH_2, OCH, NCH_2, OH), 3.0-2.0 (bs, 6 H, OH, 0.5 H_2O), 1.82-1.75 (m, 4 H, CH_2), 1.47-1.42 (m, 4 H, CH_2), 1.41-1.20 (m, 48 H, CH_2), 0.86 (t, ^3J(H,H) = 6.8, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 182.5, 158.8, 158.1, 155.7, 141.0, 133.0, 130.9, 130.5 (2C), 130.4, 129.1, 127.9 (2C), 119.8, 114.8 (2C), 114.0 (2C), 111.7, 73.2, 72.1, 71.1, 71.0, 70.8, 70.7, 70.6, 70.5, 70.1, 70.0, 69.8, 68.6, 68.2, 68.2, 64.0, 42.5, 32.0 (2C), 29.8 (5C), 29.7 (2C), 29.7 (3C), 29.5 (2C), 29.5 (2C), 29.4 (2C), 29.4 (2C), 29.3 (2C), 29.3 (2C), 26.2, 26.2, 22.8 (2C), 14.2 (2C). EA: $C_{64}H_{105}NO_{12}$·0.5 H_2O (Cal.) C: 70.55 %, H: 9.81 %, N: 1.29 %, (Found) C: 70.38 %, H: 9.57 %, N: 1.42 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-14-(4,4"-dihexadecyloxy-p-terphenyl-2'-yl-oxy)-3,6,9,12-tetraoxatetradecanoylamide (**A**16/4): Synthesized from **H**16/4 (0.40 g, 0.42 mmol), DCC (0.10 g, 0.48 mmol), pentafluorophenol (0.10 g, 0.54 mmol) and 1-amino-1-deoxy-D-sorbitol (0.5 g, 2.8 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.13 g (28 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.70 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.50 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.33 (d, ^3J(H,H) = 8.1, 1 H, Ar-H), 7.19 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.95 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.90 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.16 (t, 2 H, ^3J(H,H) = 4.5, OCH_2), 4.00-3.95 (m, 6 H, CH_2), 3.9-3.3 (m, 22 H, OCH_2, OCH, NCH_2), 2.20 (bs, 7 H, OH, H_2O), 1.82-1.75 (m, 4 H, CH_2), 1.47-1.42 (m, 4 H, CH_2), 1.41-1.20 (m, 48 H, CH_2), 0.86 (t, ^3J(H,H) = 6.8, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 172.0, 158.8, 158.1, 155.8, 140.9, 133.0, 130.8, 130.5 (2C), 130.4, 129.0, 127.9 (2C), 119.7, 114.8 (2C), 114.0 (2C), 111.6, 73.1, 73.0, 72.1, 71.0, 70.8, 70.7, 70.5, 70.4, 70.3, 70.2, 70.1, 70.0, 69.8, 68.5, 68.2, 68.2, 42.3, 32.0 (2C), 29.8 (8C), 29,8 (2C), 29.8 (2C), 29.7 (2C), 29.7 (2C), 29.5 (2C), 29.5 (2C), 29.4 (2C), 26.2, 26.2, 22.8 (2C), 14.2 (2C). EA: $C_{66}H_{109}NO_{13}$·H_2O (Cal.) C: 69.38 %, H: 9.79 %, N: 1.23 %, (Found) C: 69.51 %, H: 9.52 %, N: 1.30 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-11-(4,4"-didecyloxy-p-terphenyl-3-yl-oxy)-3,6,9-trioxaundecanoylamide (**B**10/3): Synthesized from **H***10/3 (0.40 g, 0.53 mmol), DCC (132 mg, 0.64 mmol), pentafluorophenol (118 mg, 0.64 mmol) and 1-amino-1-deoxy-D-sorbitol (0.5 g, 2.8 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.11 g (22.5 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.66 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.57 (s, 4 H, Ar-H), 7.53 (d, ^3J(H,H) = 9.1, 2 H, Ar-H), 7.20-7.15 (m, 2 H, Ar-H), 6.97-6.92 (m, 3 H, Ar-H), 4.23 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 4.08-3.95 (m, 6 H, OCH_2), 3.9-3.3 m, 18 H, OCH_2, OCH, NCH_2), 1.83-1.77 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 24 H, CH_2), 0.89-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 172.0, 158.7, 148.9, 148.5, 139.3, 138.9, 133.8, 132.9, 127.8 (2C), 127.0 (2C), 126.9 (2C), 120.4, 114.8 (2C), 114.3, 114.0, 73.1, 73.0, 72.1, 71.1, 70.7, 70.6, 70.2, 70.0, 69.9, 69.5, 69.3, 68.2 (2C), 64.0, 42.4, 32.0 (2C), 29.7, 29.7 (2C), 29.7, 29.5, 29.5, 29.4, 29.4 (3C), 26.2, 26.1, 22.8 (2C), 14.2 (2C). EA: $C_{52}H_{81}NO_{12}$·0.5 H_2O (Cal.) C: 67.79 %, H: 8.97 %, N: 1.52 %, (Found) C: 67.48 %, H: 8.97 %, N: 1.40 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-14-(4,4"-dihexadecyloxy-p-terphenyl-3-yloxy)l]-3,6,9,12-tetraoxatetradecanoylamide (**B**10/4): Synthesized from **H***10/4 (0.45 g, 0.57

mmol), DCC (140 mg, 0.68 mmol), pentafluorophenol (126 mg, 0.68 mmol), 1-amino-1-deoxy-D-sorbitol (0.5 g, 2.8 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, recrystalization from ethyl acetate; yield: 0.11 g (22.5 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.66 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.57 (s, 4 H, Ar-H), 7.53 (d, ^3J(H,H) = 9.1, 2 H, Ar-H), 7.20-7.15 (m, 2 H, Ar-H), 6.97-6.92 (m, 3 H, Ar-H), 4.23 (t, ^3J(H,H) = 4.7, 2 H, OCH_2), 4.02-3.95 (m, 6 H, OCH_2), 3.9-3.3 (m, 22 H, OCH_2, OCH, NCH_2), 1.83-1.77 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 24 H, CH_2), 0.89-0.85 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.9, 158.7, 148.9, 148.6, 139.2, 138.9, 133.8, 132.9, 127.8 (2C), 126.9 (2C), 126.8 (2C), 120.2, 114.8 (2C), 114.2, 114.1, 73.1, 72.9, 72.1, 71.0, 70.8, 70.5, 70.4, 70.5, 70.1, 70.0, 69.9, 69.5, 69.3, 68.2 (2C), 64.0, 42.4, 32.0 (2C), 29.7, 29.7, 29.6, 29.5, 29.5, 29.4 (2C), 29.4 (3C), 26.2, 26.1, 22.8 (2C), 14.2 (2C). EA: $C_{54}H_{85}NO_{13}$ (Cal.) C: 67.82 %, H: 8.96 %, N: 1.46 %, (Found) C: 67.57 %, H: 8.67 %, N: 1.29 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-11-(4-hexyloxy-4"-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoylamide (**A**6.16/3): Synthesized from **H**6.16/3 (0.15 g, 0.19 mmol), DCC (43 mg, 0.21 mmol), pentafluorophenol (42 mg, 0.23 mmol) and 1-amino-1-deoxy-D-sorbitol (0.24 g, 1.3 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.09 g (49.5 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.57 (bs, 1 H, NH), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.48 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.29 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.16 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.5, 1 H, Ar-H), 7.10 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.88 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 5.0-4.0 (bs, 5 H, OH), 4.11 (t, ^3J(H,H) = 4.5, 2 H, OCH_2), 3.96-3.90 (m, 6 H, OCH_2), 3.9-3.3 (m, 18 H, OCH_2, OCH, NCH_2), 1.82-1.76 (m, 4 H, CH_2), 1.49-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 28 H, CH_2), 0.89-0.86 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.7, 158.8, 158.1, 155.8, 140.8, 133.0, 130.8, 130.5 (2C), 130.4, 129.0, 127.9 (2C), 119.6, 114.8 (2C), 113.9 (2C), 111.7, 72.8, 72.3, 71.8, 70.8, 70.6, 70.4, 70.1, 70.0 (2C), 69.6, 68.4, 68.1, 68.0, 63.8, 42.0, 31.9, 31.6, 29.7 (4C), 29.7 (2C), 29.6, 29.6, 29.5, 29.3 (2C), 29.3, 26.1, 25.8, 22.7, 22.6, 14.1, 14.0. EA: $C_{54}H_{85}NO_{12}\cdot 0.7\ H_2O$ (Cal.) C: 68.07 %, H: 9.14 %, N: 1.47 %, (Found) C: 68.06 %, H: 9.22 %, N: 1.54 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-14-(4-hexyloxy-4"-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoylamide (**A**6.16/4): Synthesized from **H**6.16/4 (0.25 g, 0.30 mmol), DCC (68 mg, 0.33 mmol), pentafluorophenol (66 mg, 0.36 mmol) and 1-amino-1-deoxy-D-sorbitol (0.27 g, 1.5 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.15 g (49.5 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.63 (bs, 1 H, NH), 7.52 (d, ^3J(H,H) = 8.7, 4 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.17 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.3, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.3, 1 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.9-4.0 (bs, 5 H, OH), 4.13 (t, ^3J(H,H) = 4.6, 2 H, OCH_2), 3.98-3.90 (m, 6 H, OCH_2), 3.90-3.25 (m, 22 H, OCH_2, OCH, NCH_2), 1.82-1.74 (m, 4 H, CH_2), 1.48-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 28 H, CH_2), 0.93-0.86 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.6, 158.7, 158.0, 155.8, 140.7, 133.0, 130.7, 130.4 (2C), 130.3, 128.9, 127.8 (2C), 119.5, 114.7 (2C), 113.8 (2C), 111.4, 72.7, 72.1, 71.7, 70.8, 70.7, 70.3, 70.2, 70.2, 70.2, 70.1, 70.0, 69.6, 68.3, 68.0, 67.9, 63.7, 42.0, 31.9, 31.6, 29.6 (3C), 29.6 (2C), 29.6, 29.5, 29.4, 29.3 (3C), 29.3, 26.0, 25.7, 22.6, 22.5, 14.0, 14.0. EA: $C_{56}H_{89}NO_{13}\cdot 0.5\ H_2O$ (Cal.) C: 67.71 %, H: 9.13 %, N: 1.41 %, (Found) C: 67.72 %, H: 8.88 %, N: 1.45 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-20-(4-hexyloxy-4"-hexadecyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoylamide (**A**6.16/6): Synthesized from **H**6.16/6 (0.25 g, 0.28 mmol), DCC (64 mg, 0.31 mmol), pentafluorophenol (63 mg, 0.34 mmol) and 1-amino-1-deoxy-D-sorbitol (0.25 g, 1.4 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, recrystalization from ethyl acetate; yield: 0.13 g (44 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.69 (bs, 1 H, NH), 7.51 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.50 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.16 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.3, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.3, 1 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 5.2-4.2 (bs, 5 H, OH), 4.13 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.97-3.87 (m, 6 H, OCH_2), 3.9-3.2 (m, 30 H, OCH_2, OCH, NCH_2), 1.82-1.74 (m, 4 H, CH_2), 1.48-1.41 (m, 4 H, CH_2), 1.39-1.20 (m, 28 H, CH_2), 0.93-0.83 (m, 6 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.4, 158.6, 157.9, 155.7, 140.6, 132.9, 130.6, 130.3 (2C), 130.2, 128.8, 127.7 (2C), 119.4, 114.6 (2C), 113.7 (2C), 111.3, 72.4, 71.8, 71.6, 70.6, 70.6, 70.3, 70.3, 70.2, 70.2, 70.1 (4C), 70.1, 69.9, 69.5, 68.1, 67.9, 67.8, 63.6, 41.9, 31.8, 31.5, 29.5 (4C), 29.5 (2C), 29.5, 29.4, 29.3, 29.2, 29.2 (2C), 25.9, 25.6, 22.5, 22.5, 14.0, 13.9. EA: $C_{60}H_{97}NO_{15}\cdot H_2O$ (Cal.) C: 66.09 %, H: 9.15 %, N: 1.28 %, (Found) C: 66.18 %, H: 8.55 %, N: 1.18 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-11-(4-hexadecyloxy-4"-hexyloxy-p-terphenyl-2'-yloxy)-3,6,9-trioxaundecanoylamide (**A**16.6/3): Synthesized from **H**16.6/3 (0.15 g, 0.19 mmol), DCC (43 mg, 0.21 mmol), pentafluorophenol (42 mg, 0.23 mmol) and 1-amino-1-deoxy-D-sorbitol (0.24 g, 1.3 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 89 mg (49 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.58 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.47 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.30 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.16 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.11 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.92 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.87 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 4.12 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.97-3.92 (m, 6 H, OCH_2), 4.4-3.2 (m, 24 H, OCH_2, OCH, NCH_2, OH, 0.5 H_2O), 1.81-1.73 (m, 4 H, CH_2), 1.50-1.40 (m, 4 H, CH_2), 1.39-1.20 (m, 28 H, CH_2), 0.89 (t, ^3J(H,H) = 7.1, 3 H, CH_3), 0.87 (^3J(H,H) = 7.1, 3 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.6, 158.7, 158.0, 155.7, 140.8, 133.0, 130.7, 130.4 (2C), 130.3, 129.0, 127.8 (2C), 119.6, 114.7 (2C), 113.9 (2C), 111.5, 72.8, 72.3, 71.9, 70.9, 70.7, 70.4, 70.2, 70.1, 70.1, 69.6, 68.4, 68.1, 68.1, 63.8, 42.1, 32.0, 31.7, 29.8 (5C), 29.7 (2C), 29.7, 29.5, 29.4, 29.3 (2C), 26.2, 25.8, 22.7, 22.7, 14.2, 14.1. EA: $C_{54}H_{85}NO_{12}\cdot 0.5\ H_2O$ (Cal.) C: 68.32 %, H: 9.13 %, N: 1.48 %, (Found) C: 68.41 %, H: 9.20 %, N: 1.40 %."

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-14-(4-hexadecyloxy-4"-hexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12-tetraoxatetradecanoylamide (**A**16.6/4): Synthesized from **H**16.6/4 (0.20 g, 0.24 mmol), DCC (54 mg, 0.26 mmol), pentafluorophenol (54 mg, 0.29 mmol) and 1-amino-1-deoxy-D-sorbitol (0.22 g, 1.2 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, then recrystalization from ethyl acetate; yield: 0.10 g (42 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.62 (bt, ^3J(H,H) = 6.0, 1 H, NH), 7.50 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.49 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.9, 1 H, Ar-H), 7.17 (dd, ^3J(H,H) = 7.9, ^4J(H,H) = 1.5, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.5, 1 H, Ar-H), 6.93 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.7, 2 H, Ar-H), 5.0-4.2 (bs, 5 H, OH), 4.13 (t, ^3J(H,H) = 5.0, 2 H, OCH_2), 3.98-3.93 (m, 6 H, OCH_2), 3.9-3.3 (m, 22 H, OCH_2, OCH, NCH_2), 1.82-1.74 (m, 4 H, CH_2), 1.50-1.42 (m, 4 H, CH_2), 1.40-1.20 (m, 28 H, CH_2), 0.90 (t, ^3J(H,H) = 7.1, 3 H, CH_3), 0.87 (^3J(H,H) = 7.1, 3 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.6, 158.7, 158.1, 155.8, 140.8, 133.0, 130.7, 130.5 (2C), 130.3, 128.9, 127.9 (2C), 119.6, 114.7 (2C), 113.9 (2C), 111.5,

72.7, 72.2, 71.8, 70.8, 70.7, 70.4, 70.3 (3C), 70.0, 70.0, 69.6, 68.3, 68.1, 68.0, 63.9, 42.0, 31.9, 31.6, 29.7 (4C), 29.6 (2C), 29.6 (2C), 29.4, 29.3 (2C), 29.3, 26.1, 25.7, 22.7, 22.6, 14.1, 14.0. EA: $C_{56}H_{89}NO_{13}\cdot H_2O$ (Cal.) C: 67.10 %, H: 9.15 %, N:1.40 %, (Found) C: 67.34 %, H: 9.28 %, N:1.36 %.

N-[(2S,3R,4R,5R)-2,3,4,5,6-Pentahydroxyhexyl]-20-(4-hexadecyloxy-4"-hexyloxy-p-terphenyl-2'-yloxy)-3,6,9,12,15,18-hexaoxaeicosanoylamide (**A**16.6/6): Synthesized from **H**16.6/6 (0.30 g, 0.33 mmol), DCC (74 mg, 0.36 mmol), pentafluorophenol (74 mg, 0.40 mmol) and 1-amino-1-deoxy-D-sorbitol (0.31 g, 1.7 mmol). Purified by column chromatography over silica gel 60, eluent: $CHCl_3$/MeOH = 10/1 V/V, recrystalization from ethyl acetate; yield: 0.16 g (45 %); ^{1}H-NMR ($CDCl_3$, *J*/Hz, 400 MHz) δ = 7.62 (t, ^3J(H,H) = 6.0, 1 H, NH), 7.50 (d, ^3J(H,H) = 8.8, 4 H, Ar-H), 7.31 (d, ^3J(H,H) = 7.8, 1 H, Ar-H), 7.17 (dd, ^3J(H,H) = 7.8, ^4J(H,H) = 1.7, 1 H, Ar-H), 7.12 (d, ^4J(H,H) = 1.7, 1 H, Ar-H), 6.94 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 6.89 (d, ^3J(H,H) = 8.8, 2 H, Ar-H), 4.14 (t, ^3J(H,H) = 4.9, 2 H, OCH_2), 4.00-3.94 (m, 6 H, OCH_2), 4.4-3.3 (m, 38 H, OCH_2, OCH, NCH_2, OH, 1.5 H_2O), 1.81-1.73 (m, 4 H, CH_2), 1.48-1.40 (m, 4 H, CH_2), 1.39-1.20 (m, 28 H, CH_2), 0.89 (t, ^3J(H,H) = 7.1, 3 H, CH_3), 0.86 (^3J(H,H) = 6.9, 3 H, CH_3). ^{13}C-NMR ($CDCl_3$, 100 MHz) δ = 171.4, 158.6, 158.0, 155.8, 140.7, 133.0, 130.7, 130.4 (2C), 130.3, 128.9, 127.8 (2C), 119.5, 114.7 (2C), 113.8 (2C), 111.5, 72.8, 72.2, 71.8, 70.9, 70.8, 70.6, 70.5, 70.5, 70.5 (2C), 70.4 (2C), 70.3, 70.2, 70.2, 69.7, 68.4, 68.2, 68.1, 63.8, 42.0, 31.9, 31.6, 29.8 (4C), 29.8, 29.8, 29.8, 29.7, 29.6, 29.5, 29.5, 29.4, 26.1, 25.8, 22.7, 22.6, 14.1, 14.1. EA: $C_{60}H_{97}NO_{15}\cdot 1.5\ H_2O$ (Cal.) C: 65.55 %, H: 9.17 %, N:1.27 %, (Found) C: 65.64 %, H: 8.94 %, N:1.31 %.

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