Electronic Supplementary Information

Structure Directed Self-Assembly of Alkyl-Aryl-Ethylene oxide Amphiphiles

Gao Liu[1] and Gregory L. Baker[2]

[1]*Lawrence Berkeley National Laboratory, 1 Cyclotron Road, MS 70R0108B, Berkeley, CA 94720, USA. Fax: 510 486-7303; Tel: 510 486-7207; E-mail: gliu@lbl.gov*

[2]*Department of Chemistry, Michigan State University, East Lansing, MI 48824, USA. Fax: 517-353-1793; Tel: 517-355-9715, ext. 160; E-mail: bakerg@msu.edu*

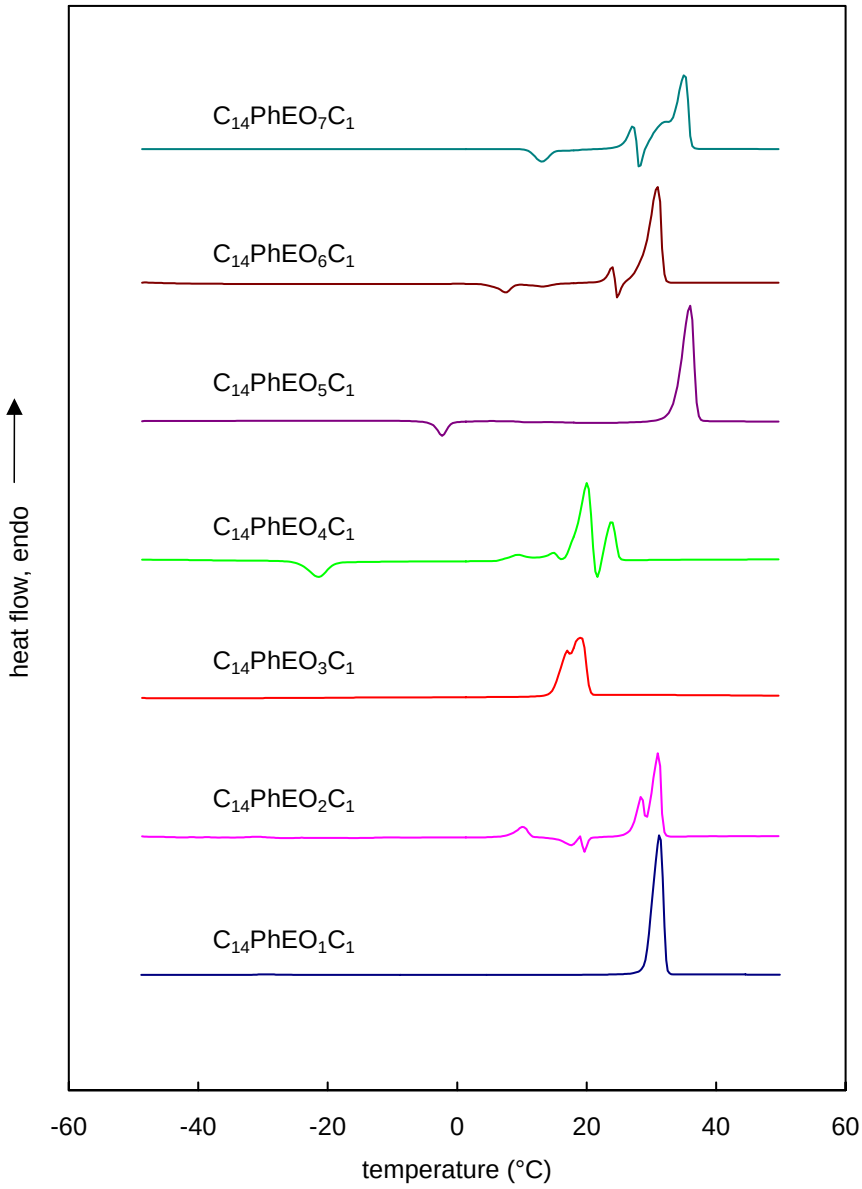

Figure S-1. DSC second heating scans of **$C_{14}PhEO_yC_1$** run at 10 °/min under a He atmosphere. Samples were held at 100 °C for 5 minutes, quenched to -100 °C at rate of 200°/min and held at -100 °C before initiating the heating scan.

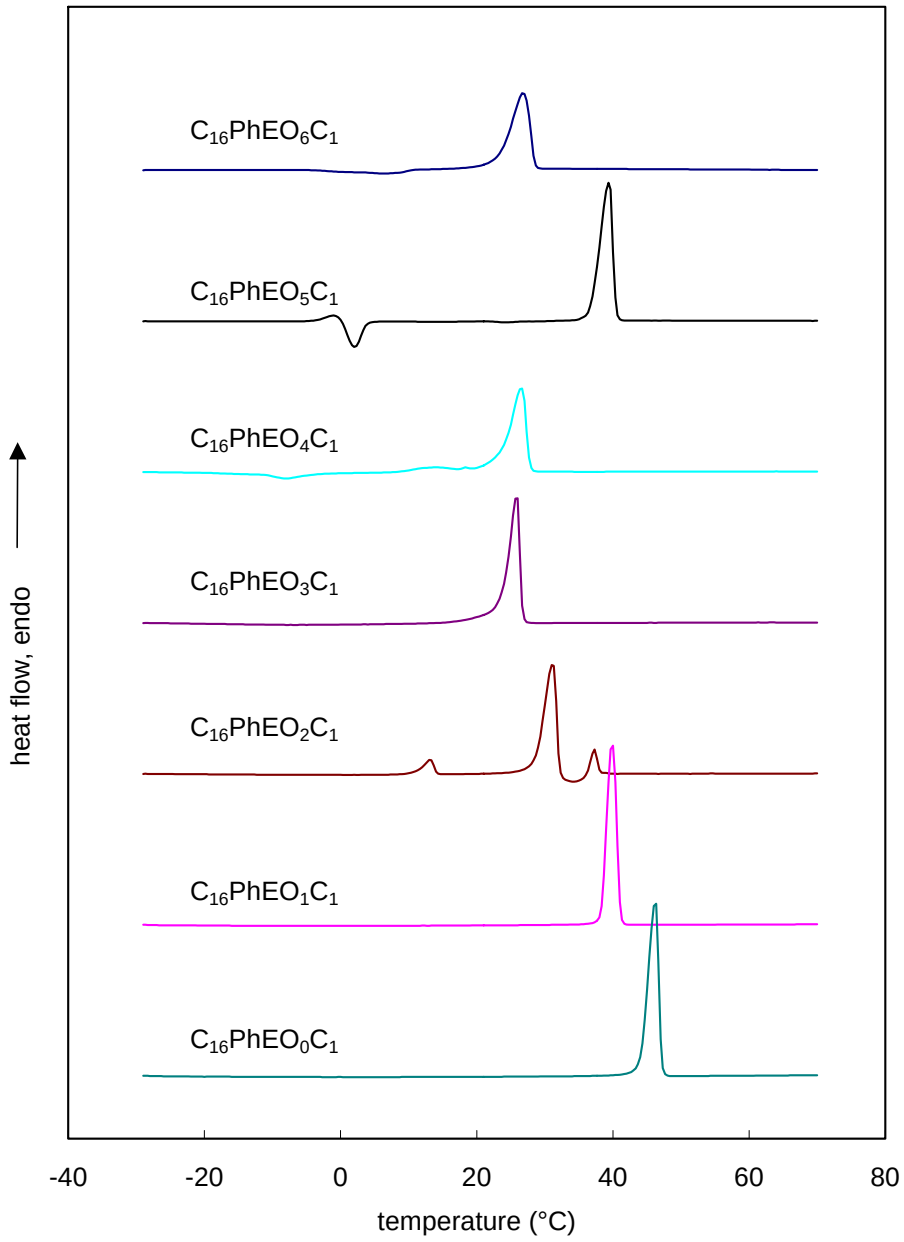

Figure S-2. DSC second heating scans of **$C_{16}PhEO_yC_1$** run at 10 °/min under a He atmosphere. Samples were held at 100 °C for 5 minutes, quenched to -100 °C at rate of 200°/min and held at -100 °C before initiating the heating scan.

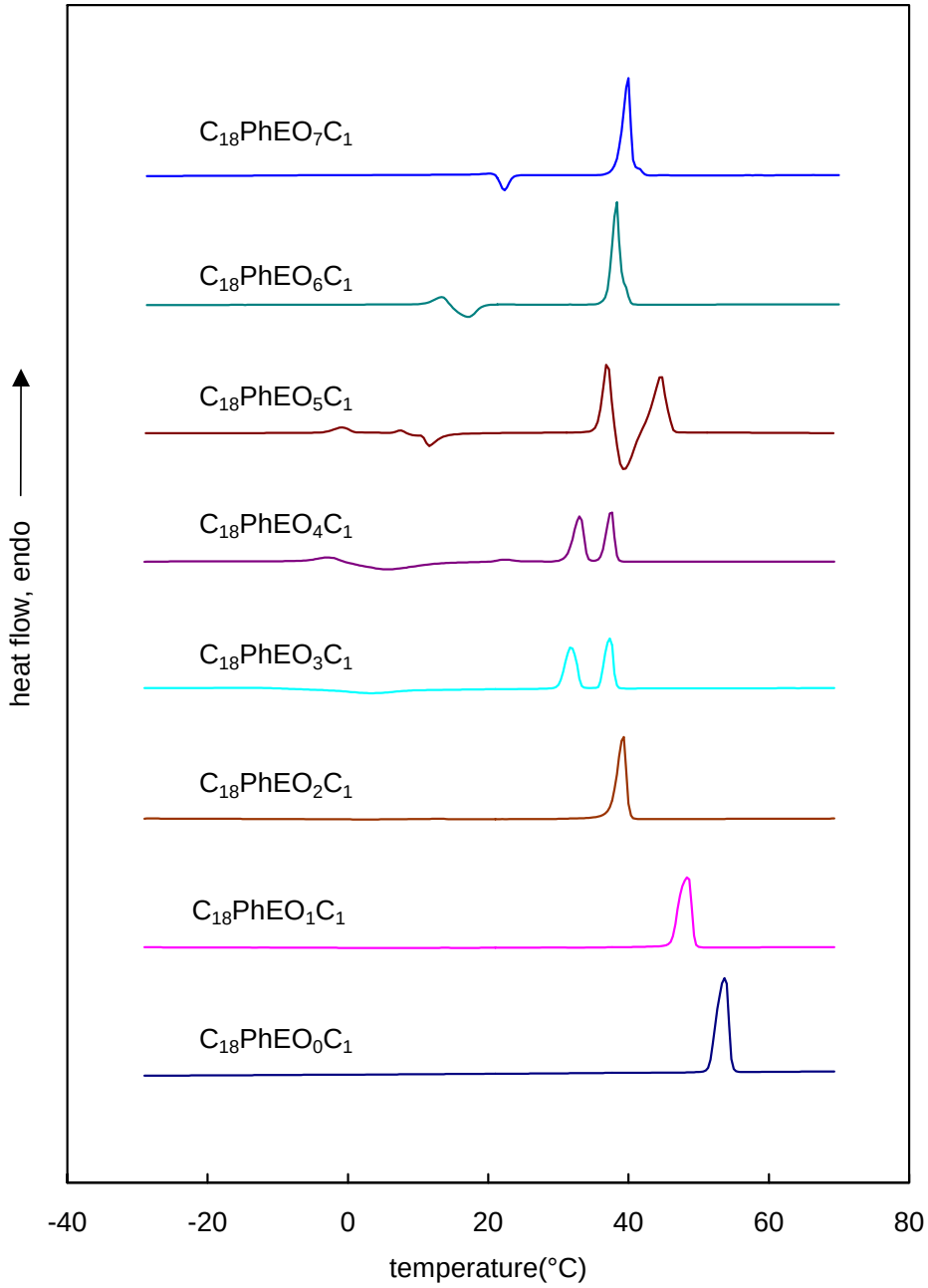

Figure S-3. DSC second heating scans of **$C_{18}PhEO_yC_1$** run at 10 °/min under a He atmosphere. Samples were held at 100 °C for 5 minutes, quenched to -100 °C at rate of 200°/min and held at -100 °C before initiating the heating scan.

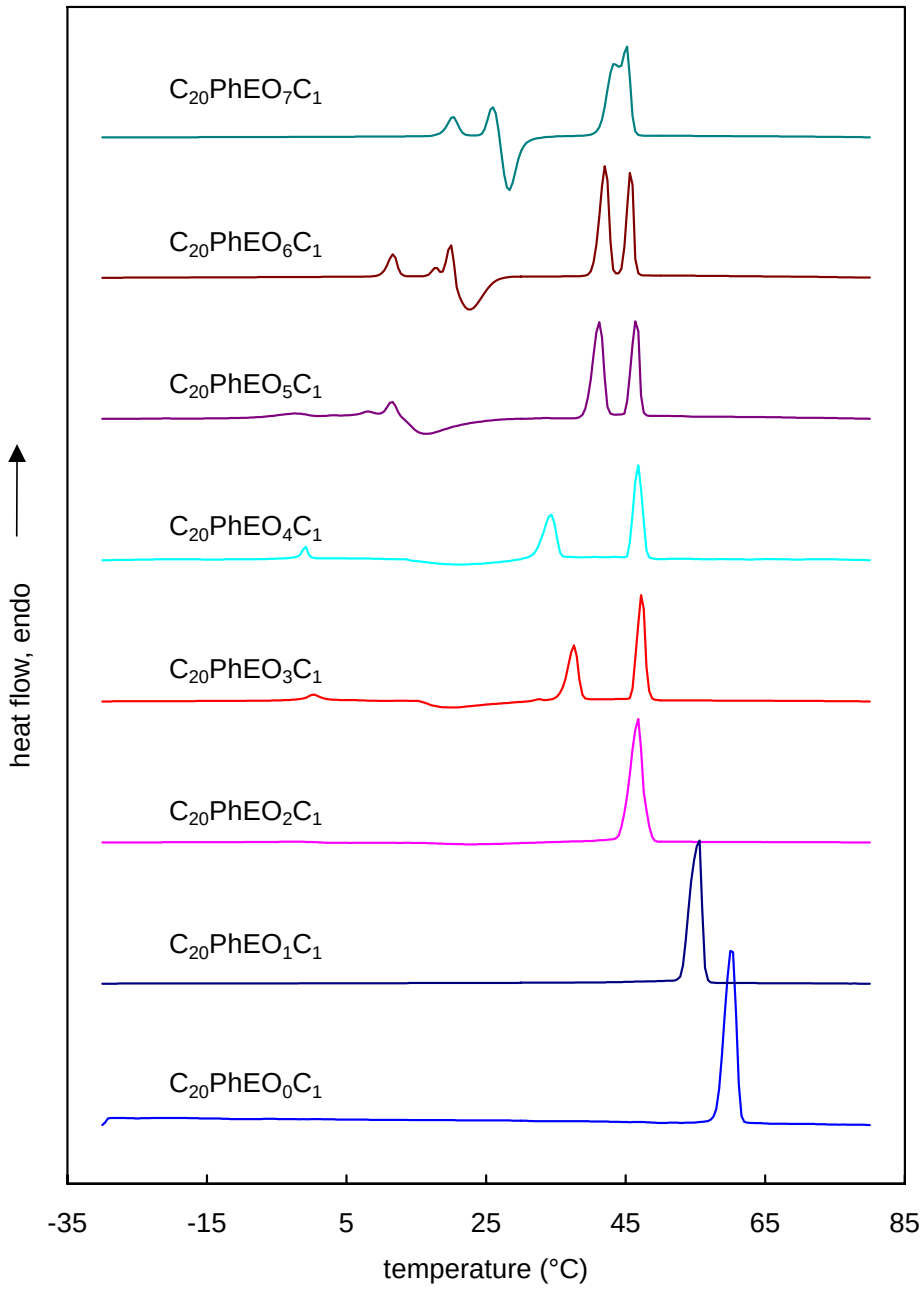

Figure S-4. DSC second heating scans of $\mathbf{C_{20}PhEO_yC_1}$ run at 10 °/min under a He atmosphere. Samples were held at 100 °C for 5 minutes, quenched to -100 °C at rate of 200°/min and held at -100 °C before initiating the heating scan.

Table S-1. Melting points and ΔH_{fus} derived from DSC measurements of $C_{18}PhEO_yC_1$						
	1st melting peak		2nd melting peak		Crystallization peak	
y	mp (°C)	ΔH (J/g)	mp (°C)	ΔH (J/g)	Crystallization point (°C)	ΔH (J/g)
0	51.5	176.3			46.6	180.2
1	46.2	144.0			43.6	148.2
2	37.4	133.1			35.0	132.6
3	30.0	75.5	35.9	69.3	35.2	72.0
4	31.2	74.9	36.1	62.9	35.3	66.7
5	35.6	138			35.2	62.6
6	36.9	143.2			34.4	55.4
7	38.3	139.0			33.6	52.8

Table S-2. Melting points and ΔH_{fus} derived from DSC measurements of $C_{20}PhEO_yC_1$						
	1st melting peak		2nd melting peak		Crystallization peak	
y	mp (°C)	-ΔH (J/g)	mp (°C)	-ΔH (J/g)	Crystallization point (°C)	ΔH (J/g)
0	58.1	180.3			52.3	179.6
1	53.1	147.6			49.8	152.5
2	44.5	137.7			44.4	82.5
3	35.8	50.3	45.9	73.7	45.0	75.5
4	32.0	48.8	45.4	69.3	44.6	72.9
5	39.3	87.7	45.0	67.6	44.6	69.2
6	40.4	87.2	44.3	60.8	44.8	66.5
7	41.4	91.7	43.6	62.0	43.5	62.0

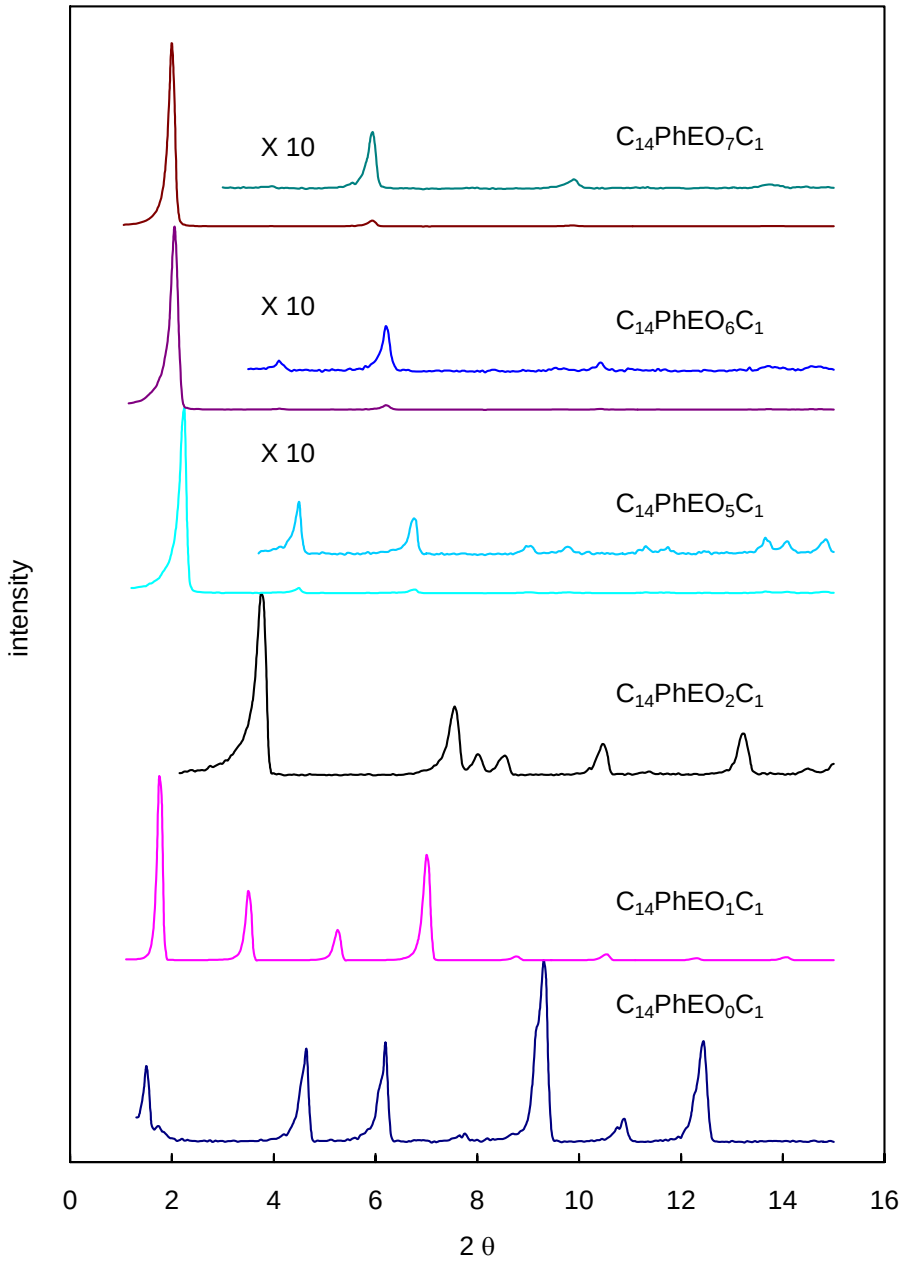

Figure S-5. Low angle XRD data for $\mathbf{C_{14}PhEO_yC_1}$. The samples were cooled from the melt and held at room temperature for 20 h prior to analysis.

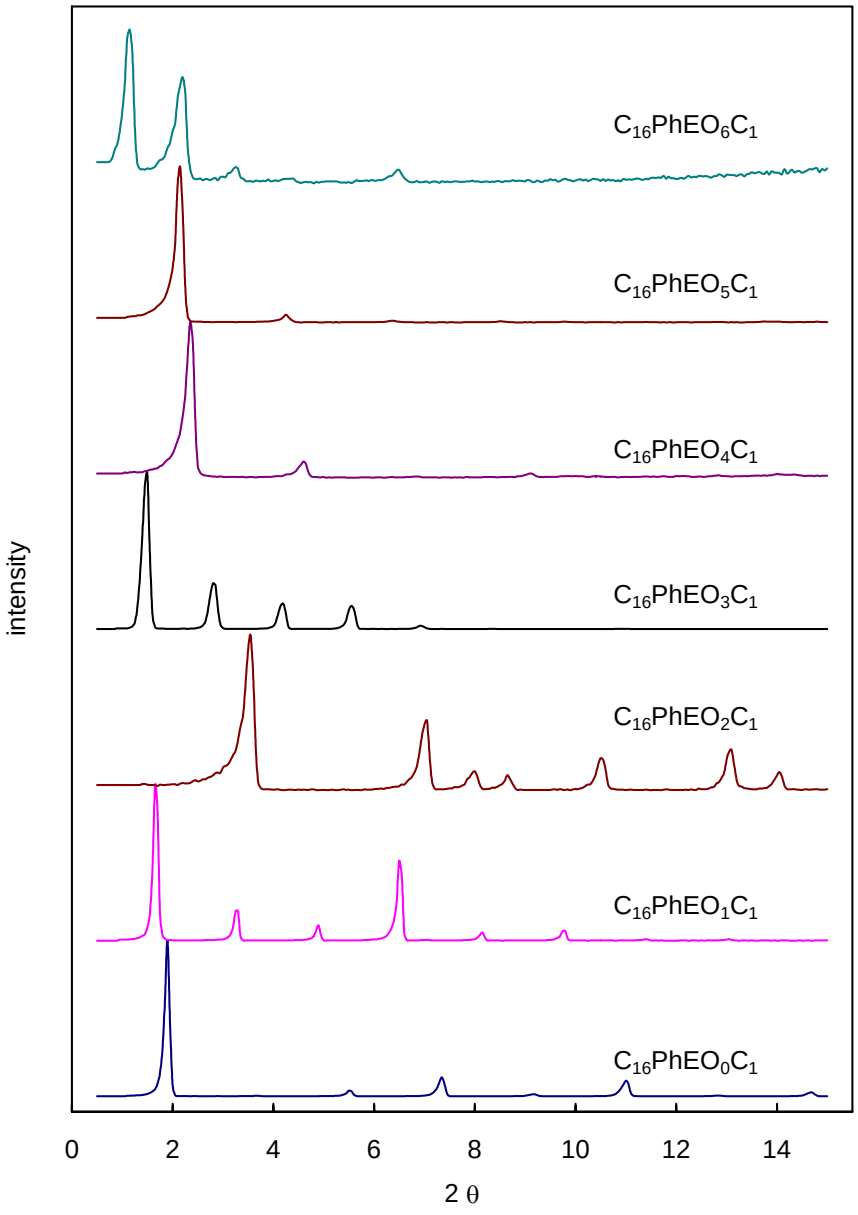

Figure S-6. Low angle XRD data for **$C_{16}PhEO_yC_1$**. The samples were cooled from the melt and held at room temperature for 20 h prior to analysis.

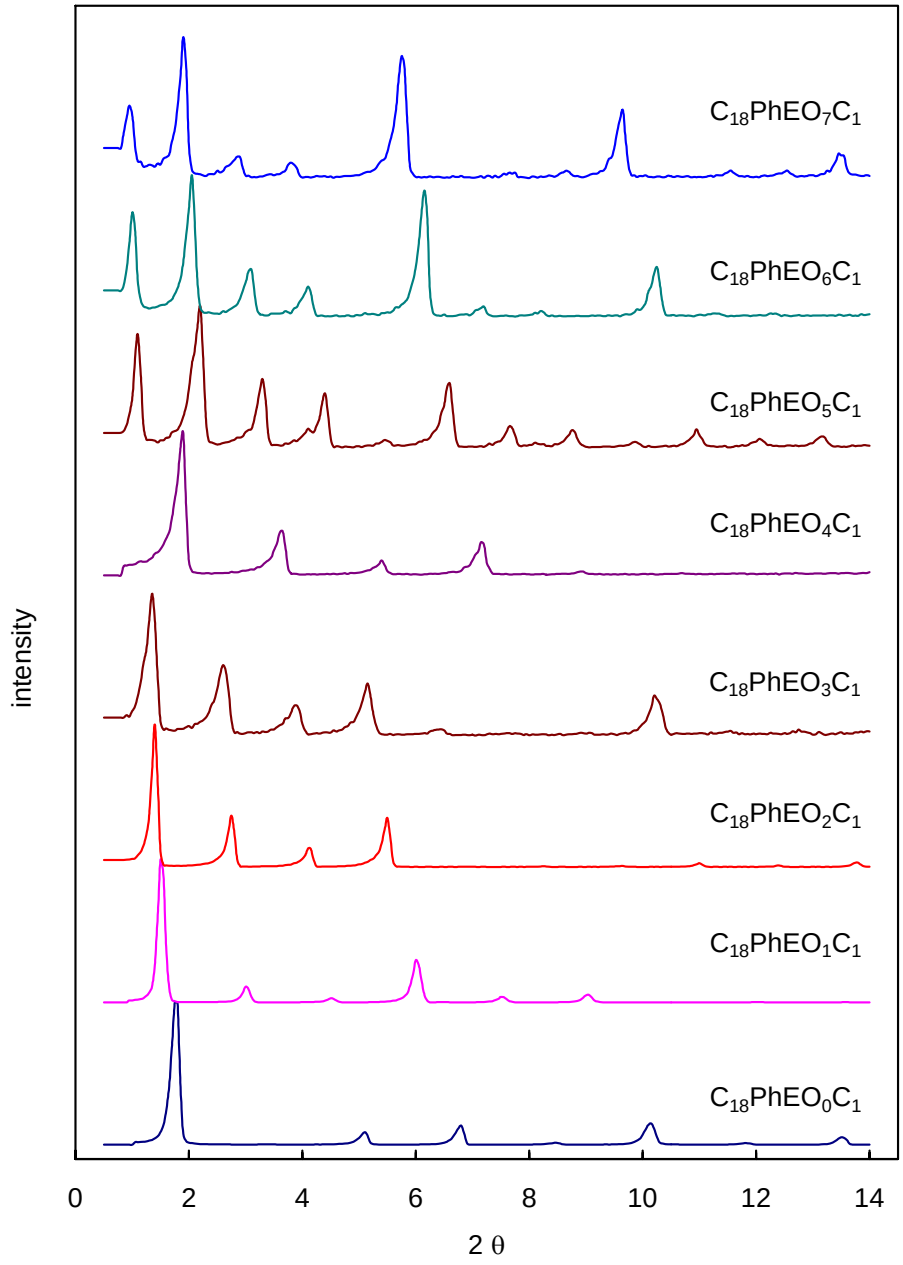

Figure S-7. Low angle XRD data for **$C_{18}PhEO_yC_1$**. The samples were cooled from the melt and held at room temperature for 20 h prior to analysis.

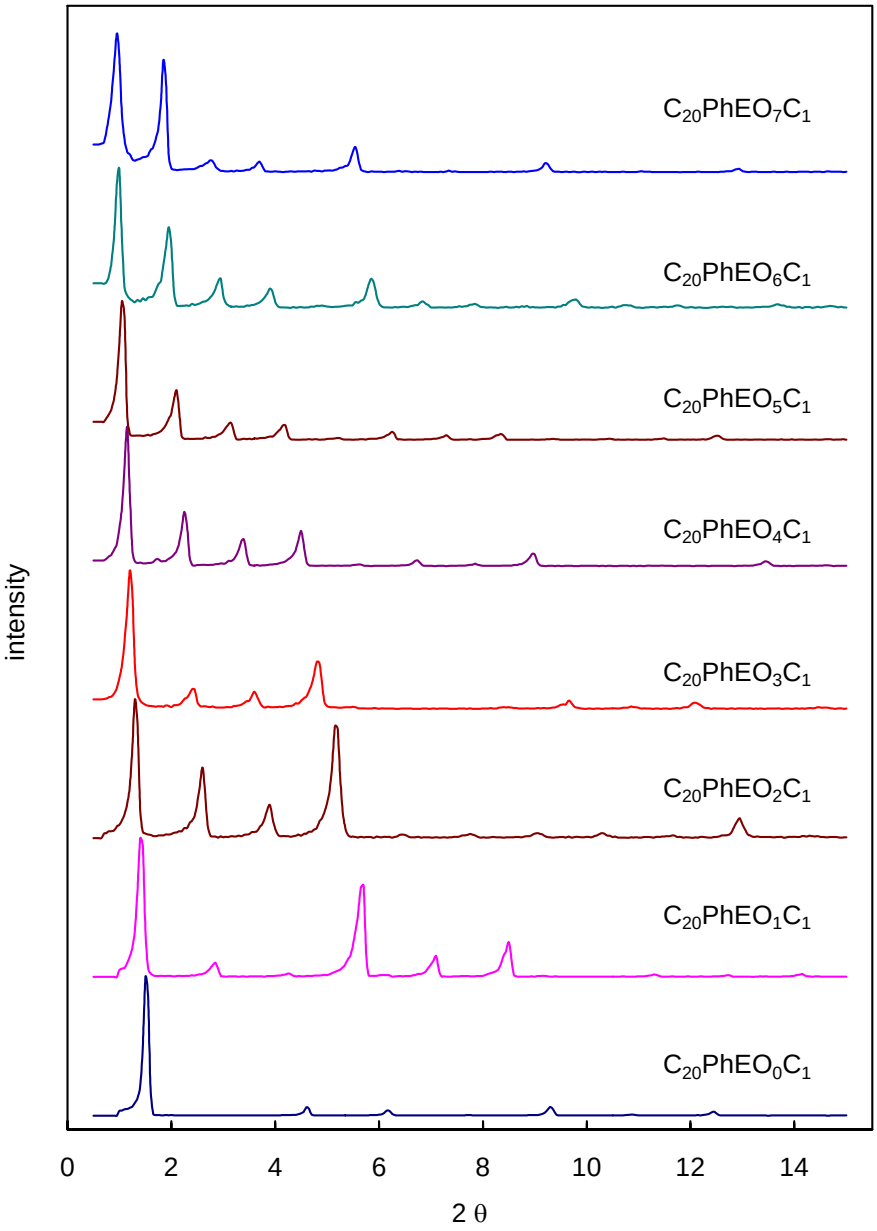

Figure S-8. Low angle XRD data for **$C_{20}PhEO_yC_1$**. The samples were cooled from the melt and held at room temperature for 20 h prior to analysis.

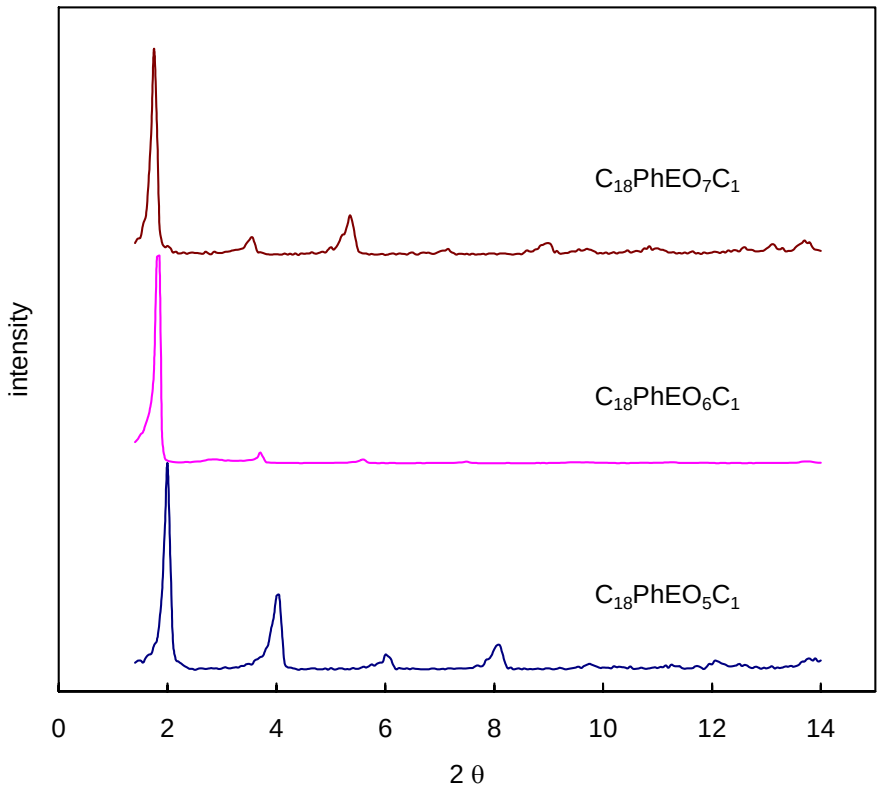

Figure S-9. Low angle XRD data for annealed **$C_{18}PhEO_yC_1$**. Prior to analysis, the samples were annealed 2° below their melting point until they reached their ultimate stable structure (~2-10 h).

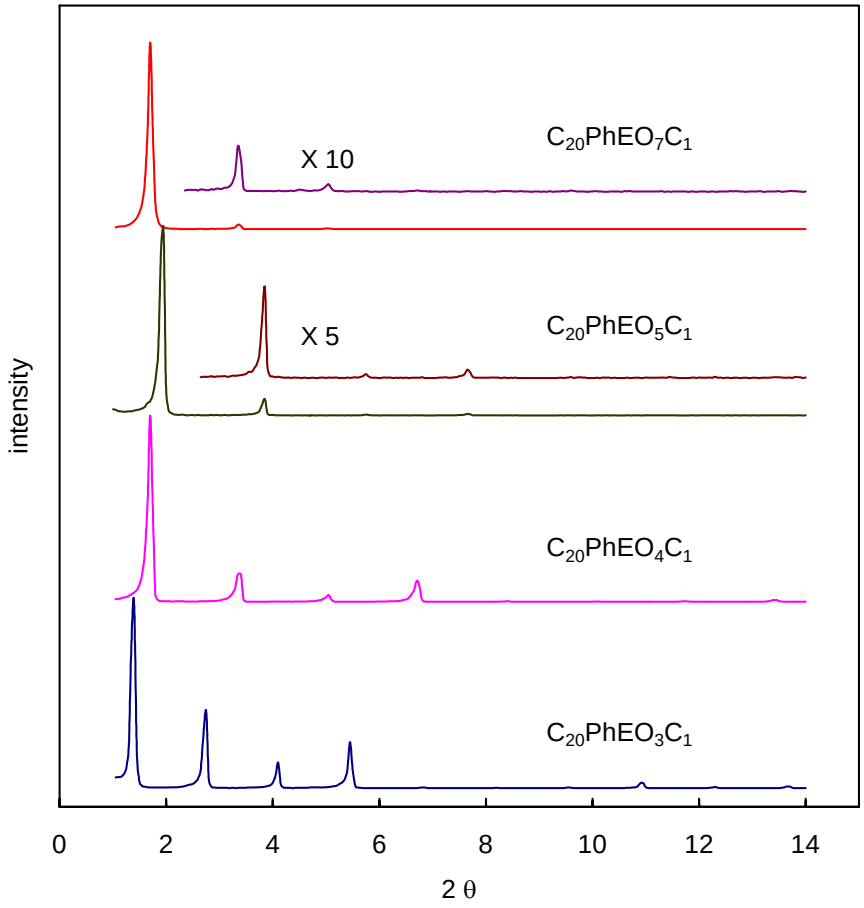

Figure S-10. Low angle XRD data for annealed $\mathbf{C_{20}PhEO_yC_1}$. Prior to analysis, the samples were annealed 2° below their melting point until they reached their ultimate stable structure (~2-10 h).

Synthesis of $C_xPhEO_yC_1$ amphiphiles

General experimental information

Unless otherwise specified, ACS reagent grade starting materials were used as received from the Aldrich. THF was dried by refluxing over CaH_2 overnight, and was then distilled from Na/benzophenone. House nitrogen was used in air and moisture sensitive reactions. The reported melting points points are uncorrected.

^{1}H NMR analyses were carried out at room temperature in $CDCl_3$ on a Varian Gemini-300 spectrometer operating at 300 MHz. The chemical shifts were calibrated using solvent peaks from residual $CHCl_3$ and are reported relative to tetramethylsilane. Infrared spectra (IR) were measured in transmission mode on a Nicolet IR/42 FT-IR spectrometer under nitrogen. The samples were prepared by melting on a NaCl disc. The spectrum for each pure sample was obtained by subtracting the NaCl spectrum from that of the sample plus substrate. Raman spectra were obtained at room temperature with a HoloProbe Raman Spectrograph excited at 633 nm. High resolution mass spectra were measured in the Mass Spectrometry Lab at University of South Carolina. All samples were purified by column chromatography followed by recrystallization in ether solution and dried under vacuum at 60 ºC for 4 days.

1 Synthesis of monotosylated polyethylene glycols

1-Tosyloxy-3-oxapentan-5-ol [$Ts(OCH_2CH_2)_2OH$] (1a) Diethylene glycol (100 mL, 0.84 mol) was dissolved in THF (50 mL) and cooled t o 0 °C in an ice bath. A solution of KOH (23 g, 0.41 mol) in 40 mL water was slowly added to the mixture, and then a solution of TsCl (40 g, 0.21 mol) in 150 mL THF was added drop-wise over one hour with vigorous stirring. After stirring overnight in an ice bath, the mixture was poured into distilled water (500 mL) and extracted with CH_2Cl_2 (2 × 250 mL). The combined organic solutions were washed with saturated $NaHCO_3$ solution (2 × 200 mL), distilled water (2 × 200 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude oil was dissolved in methanol (300 mL), and stored in a freezer over night. The

ditosylate byproduct crystallized and was removed by filtration. The filtrate was concentrated under reduced pressure to give 41.6 g (76%) of $Ts(OCH_2CH_2)_2OH$ as a colorless oil[1]. ^1H NMR (300 MHz, $CDCl_3$) δ 1.92 (s, 1H), 2.41 (s, 3H), 3.50 (t, 2H), 3.65 (m, 4H), 4.18 (t, 2H), 7.35 (d, 2H), 7.78 (d, 2H).

1-Tosyloxy-3,6-dioxaoctan-8-ol [$Ts(OCH_2CH_2)_3OH$] (1b) A clear colorless oil[1] obtained as described for 1-tosyloxy-3-oxapentan-5-ol [$Ts(OCH_2CH_2)_2OH$] (1a) in 89% yield. ^1H NMR (300 MHz, $CDCl_3$) δ 1.92 (s, 1H), 2.41 (s, 3H), 3.50 -3.65 (m, 10H), 4.18 (t, 2H), 7.35 (d, 2H), 7.78 (d, 2H).

1-Tosyloxy-3,6,9-trioxaundecan-11-ol [$Ts(OCH_2CH_2)_4OH$] (1c) A clear colorless oil[2] obtained as described for 1-tosyloxy-3-oxapentan-5-ol [$Ts(OCH_2CH_2)_2OH$] (1a) in 92% yield. ^1H NMR (300 MHz, $CDCl_3$) δ 1.92 (s, 1H), 2.41 (s, 3H), 3.50 -3.65 (m, 14H), 4.18 (t, 2H), 7.35 (d, 2H), 7.78 (d, 2H).

2 THP protection of monotosylated polyethylene glycols

2-(1-Tosyloxy-3-oxapentan-5-oxy)tetrahydropyran [$Ts(OCH_2CH_2)_2OTHP$] (2a) Over a period of 5 minutes, dihydro-4*H*-pyran (50 g, 0.59 mol) was added drop-wise to a stirred solution of **1a** (48 g, 0.18 mol) and *p*-toluenesulfonic acid monohydrate (2.50 g, 13.1 mmol) in anhydrous dioxane (500 mL) at 20 °C. After stirring for 15 minutes, half-saturated methanolic ammonia was added until the solution was slightly basic. The mixture was concentrated under reduced pressure, and redissolved in $CHCl_3$ (300 mL). The solution was washed with 5% NaCl (3 × 200 mL), dried over $MgSO_4$, and concentrated under reduced pressure to give 61.4 g (97%) of $Ts(OCH_2CH_2)_2OTHP$ as a clear light yellow oil[3]. The product was used in the next step without further purification. ^1H NMR (300 MHz, $CDCl_3$) δ 1.40-1.90 (m, 6H), 2.41 (s, 3H), 3.40-3.80 (m, 8H), 4.18 (t, 3H), 4.60 (t, 1H), 7.35 (d, 2H), 7.78 (d, 2H).

2-(1-Tosyloxy-3,6-dioxaoctan-8-oxy)tetrahydropyran [$Ts(OCH_2CH_2)_3OTHP$] (2b) Obtained as described above as a clear light yellow oil[4] in 98% yield. ^1H NMR (300 MHz, $CDCl_3$) δ 1.40-1.90 (m, 6H), 2.41 (s, 3H), 3.40-3.80 (m, 12H), 4.18 (t, 3H), 4.60 (t, 1H), 7.35 (d, 2H), 7.78 (d, 2H).

2-(1-Tosyloxy-3,6,9-trioxaundecan-11-oxy)tetrahydropyran [Ts(OCH_2CH_2)$_4$OTHP] (2c) Obtained as described above as a clear light yellow oil[5] in 98% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.40-1.90 (m, 6H), 2.41 (s, 3H), 3.40-3.80 (m, 16H), 4.18 (t, 3H), 4.60 (t, 1H), 7.35 (d, 2H), 7.78 (d, 2H).

3 THP protection of polyethylene glycol monomethyl ethers

2-(2,5,8,11,14-pentaoxahexadecan-16-oxy)tetrahydropyran [CH_3(OCH_2CH_2)$_5$OTHP] (3b) Over a period of 20 minutes, a solution of triethylene glycol monomethyl ether (29 mL, 0.18 mol) in THF (200 mL) was added drop-wise to a mixture of sodium hydride (9.0 g, 0.38 mol) in THF (150 mL) at reflux. Heating was continued for an hour, and then a solution of 2-(5-tosyloxy-3-oxapentanoxy)tetrahydropyran (61.4 g, 0.178 mol) in THF (200 mL) was added to the mixture. After stirring overnight, the precipitates were removed by filtration and the THF solution was concentrated under reduced pressure to give 53.8 g, (89%) of CH_3(OCH_2CH_2)$_5$OTHP as a clear light yellow oil[6]. The product was used in the next step without further purification. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.40-1.90 (m, 6H), 3.36 (s, 3H), 3.50-3.80 (m, 22H), 4.60 (t, 3H).

2-(2,5,8,11-hexaoxanonadecan-13-oxy)tetrahydropyran [CH_3(OCH_2CH_2)$_4$OTHP] (3a) Obtained as described above as a clear light yellow oil in 80% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.40-1.90 (m, 6H), 3.36 (s, 3H), 3.50-3.80 (m, 18H), 4.60 (t, 3H).

2-(2,5,8,11,14,17-hexaoxanonadecan-19-oxy)tetrahydropyran [CH_3(OCH_2CH_2)$_6$OTHP] (3c) Obtained as described above as a clear light yellow oil in 78% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.40-1.90 (m, 6H), 3.36 (s, 3H), 3.50-3.80 (m, 26H), 4.60 (t, 3H).

2-(2,5,8,11,14,17,20-heptaoxadocosan-22-oxy)tetrahydropyran [CH_3(OCH_2CH_2)$_7$OTHP] (3d) Obtained as described above as a clear light yellow oil in 82% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.40-1.90 (m, 6H), 3.36 (s, 3H), 3.50-3.80 (m, 30H), 4.60 (t, 3H).

4 Deprotection of polyethylene glycol monomethyl ethers

2,5,8,11,14-pentaoxahexadecan-16-ol [$CH_3(OCH_2CH_2)_5OH$] (4b) HCl (50 mL, 2M) was added to an ethanol solution (400 mL) of 2-(14-methoxy-3,6,9,12-tetraoxatetradecan-1-oxy)tetrahydropyran (50.0 g, 0.147 mol) and the mixture was refluxed for 5 hours. Concentration under reduced pressure and vacuum distillation gave a 72% yield of $CH_3(OCH_2CH_2)_5OH$. bp 102 °C (40 mTorr) (lit.[7] bp 145 – 147 °C/1 Torr). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.33 (s, 1H), 3.36 (s, 3H), 3.50-3.80 (m, 20H).

2,5,8,11-hexaoxanonadecan-13-ol [$CH_3(OCH_2CH_2)_4OH$] (4a) Obtained as described above as a clear colorless oil in 72% yield after vacuum distillation. bp 100 °C (40 mTorr) (lit.[7] bp 160 – 167 °C (1 Torr)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.33 (s, 1H), 3.36 (s, 3H), 3.50-3.80 (m, 24H).

2,5,8,11,14,17-hexaoxanonadecan-19-ol [$CH_3(OCH_2CH_2)_6OH$] (4c) Obtained as described above as a clear colorless oil[7] in 61% yield after vacuum distillation. bp 135 °C (40 mTorr) (lit.[7] bp 160 – 167 °C (1 Torr)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.33 (s, 1H), 3.36 (s, 3H), 3.50-3.80 (m, 24H).

2,5,8,11,14,17,20-hexaoxadocosan-22-ol [$CH_3(OCH_2CH_2)_7OH$] (4d) Obtained as described above as a clear colorless oil in 45% yield after vacuum distillation. bp 205 °C (40 mTorr) (lit.[8] bp 198 – 205 °C (0.04 Torr)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.33 (s, 1H), 3.36 (s, 3H), 3.50-3.80 (m, 28H).

5 Synthesis of tosylated derivates of polyethylene glycol monomethyl ethers

4-Tosyloxy-2-oxabutane [$TsOCH_2CH_2OCH_3$] (5a) Prepared according to the procedure for 5-tosyloxy-3-oxapentanol except that stoichiometric amounts of starting materials were used. Yield: 98% as a clear colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.43 (s, 3H), 3.29 (s, 3H), 3.56 (t, 2H), 4.15 (t, 2H), 7.31 (d, 2H), 7.77 (d, 2H).

7-Tosyloxy-2,5-dioxaheptane [$Ts(OCH_2CH_2)_2OCH_3$] (5b) Obtained as described above as a clear colorless oil in 99% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.43 (s, 3H), 3.33 (s, 3H), 3.45 (t, 2H), 4.04 (t, 2H), 3.58 (t, 2H), 7.68 (d, 2H) 3.67 (t, 2H), 4.17 (t, 2H), 7.31 (d, 2H), 7.77 (d, 2H).

10-Tosyloxy-2,5,8-trioxadecane [$Ts(OCH_2CH_2)_3OCH_3$] (5c) Obtained as described above as a clear colorless oil[9] in 99% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.3 (s, 3H), 3.22 (s, 3H), 3.28-3.70 (m, 10H), 4.04 (t, 2H), 7.24 (d, 2H), 7.68 (d, 2H).

13-Tosyloxy-2,5,8,11-tetraoxatridecane [$Ts(OCH_2CH_2)_4OCH_3$] (5d) Obtained as described above as a clear colorless oil in 99% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.3 (s, 3H), 3.22 (s, 3H), 3.28-3.70 (m, 14H), 4.04 (t, 2H), 7.24 (d, 2H), 7.68 (d, 2H).

16-Tosyloxy-2,5,8,11,14-pentaoxahexadecane [$Ts(OCH_2CH_2)_5OCH_3$] (5e) Obtained as described above as a clear colorless oil[10] in 99% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.3 (s, 3H), 3.22 (s, 3H), 3.28-3.70 (m, 18H), 4.04 (t, 2H), 7.24 (d, 2H), 7.68 (d, 2H).

19-Tosyloxy-2,5,8,11,14,17-hexaoxanonadecane [$Ts(OCH_2CH_2)_6OCH_3$] (5f) Obtained as described above as a clear colorless oil[11] in 99% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.3 (s, 3H), 3.22 (s, 3H), 3.28-3.70 (m, 24H), 4.04 (t, 2H), 7.24 (d, 2H), 7.68 (d, 2H).

22-Tosyloxy-2,5,8,11,14,17,19-heptaoxadocosane [$Ts(OCH_2CH_2)_7OCH_3$] (5g) Obtained as described above as a clear colorless oil[12] in 99% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.3 (s, 3H), 3.22 (s, 3H), 3.28-3.70 (m, 28H), 4.04 (t, 2H), 7.24 (d, 2H), 7.68 (d, 2H).

6 Coupling of alkyl chains to benzene ring

4-Decylanisole [$CH_3(CH_2)_9PhOCH_3$] (6a) A solution of 1-decylbromide (44.2 mL, 0.2 mol) in THF (200 mL) was added under nitrogen to Mg turnings (5.0 g, 0.22 mol) over a period of 15 minutes. Heating was applied to initiate the reaction. After the reaction was no longer exothermic, the mixture was refluxed for an additional hour. The Grignard solution was transferred while hot to a stirred THF solution (250 mL) of 4-chloroaniline (20 g, 0.14 mol) and $(dppp)Cl_2Ni$ (0.28 g, 7.0 mmol). The mixture was refluxed for 2 days under nitrogen and then cooled to room temperature. The mixture was washed with saturated aqueous NaCl (3 × 200 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The crude brown solid was purified by recrystallization from pentane and ether to give 25 g (72%) of $CH_3(CH_2)_9PhOCH_3$ as a

white crystalline powder. mp 16.2 – 17.1 °C (lit.[13] mp 17.0 – 17.5 °C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 16H), 1.55 (t, 2H), 2.52 (t, 2H), 3.78 (s, 3H), 6.80 (d, 2H), 7.08 (d, 2H).

4-Tetradecylanisole [$CH_3(CH_2)_{13}PhOCH_3$] (6b) Obtained as described above as a white crystalline powder in 61% yield. mp 36.5 °C (lit.[14] mp 38 °C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 24H), 1.55 (t, 2H), 2.52 (t, 2H), 3.78 (s, 3H), 6.80 (d, 2H), 7.08 (d, 2H). HRMS calc. for $C_{21}H_{36}O_1$ 304.2766, found 304.2767.

4-Hexadecylanisole [$CH_3(CH_2)_{15}PhOCH_3$] (6c) Obtained as described above as a white crystalline powder in 61% yield. mp 44.1 °C (lit.[15] mp 43 – 44 °C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 28H), 1.55 (t, 2H), 2.52 (t, 2H), 3.78 (s, 3H), 6.80 (d, 2H), 7.08 (d, 2H). HRMS calc. for $C_{23}H_{40}O_1$ 332.3079, found 332.3086.

4-Octadecylanisole [$CH_3(CH_2)_{17}PhOCH_3$] (6d) Obtained as described above as a white crystalline powder[13] in 45% yield. mp 51.5 °C (lit.[16] mp 51 – 52 °C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 32H), 1.55 (t, 2H), 2.52 (t, 2H), 3.78 (s, 3H), 6.80 (d, 2H), 7.08 (d, 2H). HRMS calc. for $C_{25}H_{44}O_1$ 360.3392, found 360.3393.

4-Eicosylanisole [$CH_3(CH_2)_{19}PhOCH_3$] (6e) Obtained as described above as a white crystalline powder in 45% yield. mp 58.1 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 36H), 1.55 (t, 2H), 2.52 (t, 2H), 3.78 (s, 3H), 6.80 (d, 2H), 7.08 (d, 2H).

7 Synthesis of 4-alkylphenols

4-Decylphenol [$CH_3(CH_2)_9PhOH$] (7a) An anhydrous solution of 4-decylanisole (7.50 g, 30.2 mmol) in methylene chloride (200 mL) was cooled in a dry ice/acetone bath. After the temperature reached equilibrium, a solution of BBr_3 (2.9 mL, 30 mmol) in methylene chloride (50 mL) was added over a period of 10 minutes. The mixture was allowed to warm to room temperature and stirred over night. The reaction was quenched by the drop-wise addition of water (100 mL) and extracted with ether (200 mL). The organic layer was washed with saturated aqueous NaCl (200 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude white solid was recrystallized from cold methylene chloride to give 7.0 g (99%) of 4-decylphenol as white crystalline powder. mp

57.5 – 58.5 °C (lit.[13] mp 57.5 – 58.5 °C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 16H), 1.55 (t, 2H), 2.50 (t, 2H), 4.45 (s, 1H), 6.72 (d, 2H), 7.02 (d, 2H).

4-Tetradecylphenol [$CH_3(CH_2)_{13}PhOH$] (7b) A white crystalline powder obtained as described for 4-decylphenol [$CH_3(CH_2)_9PhOH$] in 90% yield. mp 72.5 – 73.5 °C (lit.[17] mp 73 – 74 °C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 24H), 1.55 (t, 2H), 2.50 (t, 2H), 4.45 (s, 1H), 6.72 (d, 2H), 7.02 (d, 2H).

4-Hexadecylphenol [$CH_3(CH_2)_{15}PhOH$] (7c) A white crystalline powder obtained as described for 4-decylphenol [$CH_3(CH_2)_9PhOH$] in 90% yield. mp 78 – 79 °C (lit.[18] mp 78 – 79 °C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 28H), 1.55 (t, 2H), 2.50 (t, 2H), 4.45 (s, 1H), 6.72 (d, 2H), 7.02 (d, 2H).

4-Octadecylphenol [$CH_3(CH_2)_{17}PhOH$] (7d) A white crystalline powder obtained as described for 4-decylphenol [$CH_3(CH_2)_9PhOH$] in 91% yield. mp 82.0 – 84.0 °C (lit.[13] mp 83.0 – 84.0 °C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 32H), 1.55 (t, 2H), 2.50 (t, 2H), 4.45 (s, 1H), 6.72 (d, 2H), 7.02 (d, 2H).

4-Eicosylphenol [$CH_3(CH_2)_{19}PhOH$] (7e) A white crystalline powder obtained as described for 4-decylphenol [$CH_3(CH_2)_9PhOH$] in 89% yield. mp 88.0 – 89.0 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 36H), 1.55 (t, 2H), 2.50 (t, 2H), 4.45 (s, 1H), 6.72 (d, 2H), 7.02 (d, 2H).

8 Synthesis of $C_xPhEO_yC_1$

4-Decyl-1-(2,5,8-trioxadecan-10-oxy)benzene [$CH_3(OCH_2CH_2)_3OPh(CH_2)_9CH_3$] A THF solution (100 mL) of **7a** (2.5 g, 11 mmol) was added drop-wise to a mixture of sodium hydride (0.50 g, 21 mmol) in anhydrous THF (50 mL) under nitrogen. After refluxing for an hour, a THF solution (100 mL) of **5c** (4.00 g, 12.6 mmol) was added and heating was continued overnight. The mixture was cooled to room temperature and filtered. The filtrate was washed with saturated aqueous NaCl (3 × 200 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The resulting oil was purified using flash chromatography (60/40 ethyl acetate/hexane) to yield 3.4 g (75%) of product as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 14H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 8H), 3.82 (t,

2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{23}H_{40}O_4$ 380.2927, found 380.2921.

4-Tetradecyl-1-(2-oxabutan-4-oxy)benzene [$CH_3OCH_2CH_2OPh(CH_2)_{13}CH_3$] Obtained as described above as a white crystalline solid in 80% yield after purification by column chromatography (35/65 ethyl acetate/hexane). mp 30.1 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 22H), 1.55 (t, 2H), 2.51 (t, 2H), 3.43 (s, 3H), 3.73 (t, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{23}H_{40}O_2$ 348.3028, found 348.3021.

4-Tetradecyl-1-(2,5-dioxaheptan-7-oxy)benzene [$CH_3(OCH_2CH_2)_2OPh(CH_2)_{13}CH_3$] Obtained as described above as a white crystalline solid obtained in 77% yield after purification by column chromatography (40/60 ethyl acetate/hexane). mp 30.5 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 22H), 1.55 (t, 2H), 2.51 (t, 2H), 3.43 (s, 3H), 3.55 (t, 2H), 2.72 (d, 2H), 3.83 (t, 2H), 4.10 (t, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{25}H_{44}O_3$ 392.3290, found 392.3291.

4-Tetradecyl-1-(2,5,8-trioxadecan-10-oxy)benzene [$CH_3(OCH_2CH_2)_3OPh(CH_2)_{13}CH_3$] Obtained as described above as a white solid in 73% yield after purification by column chromatography (50/50 ethyl acetate/hexane). mp 16.1 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 22H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 8H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{27}H_{48}O_4$ 436.3553, found 436.3549.

4-Tetradecyl-1-(2,5,8,11-tetraoxatridecan-13-oxy)benzene [$CH_3(OCH_2CH_2)_4OPh(CH_2)_{13}CH_3$] Obtained as described above as a white solid in 60% yield after purification by column chromatography (60/40 ethyl acetate/hexane). mp 22.3 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 22H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 20H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{29}H_{52}O_5$ 480.3815, found 480.3806.

4-Tetradecyl-1-(2,5,8,11,14-pentaoxahexadecan-16-oxy)benzene [$CH_3(OCH_2CH_2)_5OPh(CH_2)_{13}CH_3$] Obtained as described above as a white solid in 52% yield after purification by column chromatography (80/40 ethyl acetate/hexane). mp 31.7 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 22H), 1.55 (t, 2H),

2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 16H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{31}H_{56}O_6$ 524.4077, found 524.4081.

4-Tetradecyl-1-(2,5,8,11,14,17-hexaoxanonadecan-19-oxy)benzene [$CH_3(OCH_2CH_2)_6OPh(CH_2)_{13}CH_3$] Obtained as described above as a white solid in 65% yield after purification by column chromatography (80/20 ethyl acetate/hexane). mp 26.9 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 22H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 20H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{33}H_{60}O_7$ 568.4339, found 568.4327.

4-Tetradecyl-1-(2,5,8,11,14,17,19-heptaoxadocosan-22-oxy)benzene [$CH_3(OCH_2CH_2)_7OPh(CH_2)_{13}CH_3$] A white solid obtained in 62% yield after purification by column chromatography (80/20 ethyl acetate/hexane). mp 29.1 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 22H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 24H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{35}H_{64}O_8$ 612.4601, found 612.4607.

4-Hexadecyl-1-(2-oxabutan-4-oxy)benzene [$CH_3OCH_2CH_2OPh(CH_2)_{15}CH_3$] Obtained as described above as a white crystalline solid in 71% yield after purification by column chromatography (35/65 ethyl acetate/hexane). mp 38.3 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 26H), 1.55 (t, 2H), 2.51 (t, 2H), 3.43 (s, 3H), 3.73 (t, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{25}H_{44}O_2$ 376.3341, found 376.3335.

4-Hexadecyl-1-(2,5-dioxaheptan-7-oxy)benzene [$CH_3(OCH_2CH_2)_2OPh(CH_2)_{15}CH_3$] Obtained as described above as a white crystalline solid in 79% yield after purification by column chromatography (40/60 ethyl acetate/hexane). mp 28.6 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 26H), 1.55 (t, 2H), 2.51 (t, 2H), 3.43 (s, 3H), 3.55 (t, 2H), 2.72 (d, 2H), 3.83 (t, 2H), 4.10 (t, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{27}H_{48}O_3$ 420.3603, found 420.3601.

4-Hexadecyl-1-(2,5,8-trioxadecan-10-oxy)benzene [$CH_3(OCH_2CH_2)_3OPh(CH_2)_{15}CH_3$] Obtained as described above as a white solid in 78% yield after purification by column chromatography (50/50 ethyl acetate/hexane). mp 23.6 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 26H), 1.55 (t, 2H),

2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 8H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{29}H_{52}O_4$ 464.3866, found 464.3857.

4-Hexadecyl-1-(2,5,8,11-tetraoxatridecan-13-oxy)benzene [$CH_3(OCH_2CH_2)_4OPh(CH_2)_{15}CH_3$] Obtained as described above as a white solid in 51% yield after purification by column chromatography (60/40 ethyl acetate/hexane). mp 23.6 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 26H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 20H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{31}H_{56}O_5$ 508.4128, found 508.4127.

4-Hexadecyl-1-(2,5,8,11,14-pentaoxahexadecan-16-oxy)benzene [$CH_3(OCH_2CH_2)_5OPh(CH_2)_{15}CH_3$] Obtained as described above as a white solid in 71% yield after purification by column chromatography (70/30 ethyl acetate/hexane). mp 36.8 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 26H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 16H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{33}H_{60}O_6$ 552.4390, found 552.4370.

4-Hexadecyl-1-(2,5,8,11,14,17-hexaoxanonadecan-19-oxy)benzene [$CH_3(OCH_2CH_2)_6OPh(CH_2)_{15}CH_3$] Obtained as described above as a white solid in 65% yield after purification by column chromatography (80/20 ethyl acetate/hexane). mp 23.2°C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 26H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 20H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{35}H_{64}O_7$ 596.4652, found 596.4660.

4-Octadecyl-1-(2-oxabutan-4-oxy)benzene [$CH_3OCH_2CH_2OPh(CH_2)_{17}CH_3$] Obtained as described above as a white crystalline solid in 72% yield after purification by column chromatography (35/65 ethyl acetate/hexane). mp 46.2 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 30H), 1.55 (t, 2H), 2.51 (t, 2H), 3.43 (s, 3H), 3.73 (t, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{27}H_{48}O_2$ 404.3654, found 404.3656.

4-Octadecyl-1-(2,5-dioxaheptan-7-oxy)benzene [$CH_3(OCH_2CH_2)_2OPh(CH_2)_{17}CH_3$] Obtained as described above as a white crystalline solid in 81% yield after purification by column chromatography (40/60 ethyl acetate/hexane). mp 37.4 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b,

30H), 1.55 (t, 2H), 2.51 (t, 2H), 3.43 (s, 3H), 3.55 (t, 2H), 2.72 (d, 2H), 3.83 (t, 2H), 4.10 (t, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{29}H_{52}O_3$ 448.3916, found 448.3918.

4-Octadecyl-1-(2,5,8-trioxadecan-10-oxy)benzene

[$CH_3(OCH_2CH_2)_3OPh(CH_2)_{17}CH_3$] Obtained as described above as a white solid in 71% yield after purification by column chromatography (50/50 ethyl acetate/hexane). mp 35.9 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 30H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 8H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{31}H_{56}O_4$ 492.4179, found 492.4189.

4-Octadecyl-1-(2,5,8,11-tetraoxatridecan-13-oxy)benzene

[$CH_3(OCH_2CH_2)_4OPh(CH_2)_{17}CH_3$] Obtained as described above as a white solid in 65% yield after purification by column chromatography (60/40 ethyl acetate/hexane). mp 36.1 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 30H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 20H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{33}H_{60}O_5$ 536.4441, found 536.4447.

4-Octadecyl-1-(2,5,8,11,14-pentaoxahexadecan-16-oxy)benzene

[$CH_3(OCH_2CH_2)_5OPh(CH_2)_{17}CH_3$] Obtained as described above as a white solid in 74% yield after purification by column chromatography (70/30 ethyl acetate/hexane). mp 42.1 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 30H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 16H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H). HRMS calc. for $C_{35}H_{64}O_6$ 580.4703, found 580.4694.

4-Octadecyl-1-(2,5,8,11,14,17-hexaoxanonadecan-19-oxy)benzene

[$CH_3(OCH_2CH_2)_6OPh(CH_2)_{17}CH_3$] Obtained as described above as a white solid in 71% yield after purification by column chromatography (80/20 ethyl acetate/hexane). mp 36.9 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 30H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 20H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H).

4-Octadecyl-1-(2,5,8,11,14,17,19-heptaoxadocosan-22-oxy)benzene

[$CH_3(OCH_2CH_2)_7OPh(CH_2)_{17}CH_3$] Obtained as described above as a white solid in 51% yield after purification by column chromatography (90/10 ethyl acetate/hexane). mp

38.3 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 30H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 24H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H).

4-Eicosyl-1-(2-oxabutan-4-oxy)benzene [$CH_3OCH_2CH_2OPh(CH_2)_{19}CH_3$] Obtained as described above as a white crystalline solid in 78% yield after purification by column chromatography (35/65 ethyl acetate/hexane). mp 53.1 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 34H), 1.55 (t, 2H), 2.51 (t, 2H), 3.43 (s, 3H), 3.73 (t, 2H), 6.80 (d, 2H), 7.04 (d, 2H).

4-Eicosyl-1-(2,5-dioxaheptan-7-oxy)benzene [$CH_3(OCH_2CH_2)_2OPh(CH_2)_{19}CH_3$] Obtained as described above as a white crystalline solid in 75% yield after purification by column chromatography (40/60 ethyl acetate/hexane). mp 44.5 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 22H), 1.55 (t, 2H), 2.51 (t, 2H), 3.43 (s, 3H), 3.55 (t, 2H), 2.72 (d, 2H), 3.83 (t, 2H), 4.10 (t, 2H), 6.80 (d, 2H), 7.04 (d, 2H).

4-Eicosyl-1-(2,5,8-trioxadecan-10-oxy)benzene [$CH_3(OCH_2CH_2)_3OPh(CH_2)_{19}CH_3$] Obtained as described above as white solid in 82% yield after purification by column chromatography (50/50 ethyl acetate/hexane). mp 45.9 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 34H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 8H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H).

4-Eicosyl-1-(2,5,8,11-tetraoxatridecan-13-oxy)benzene [$CH_3(OCH_2CH_2)_4OPh(CH_2)_{19}CH_3$] Obtained as described above as a white crystalline solid in 72% yield after purification by column chromatography (60/40 ethyl acetate/hexane). mp 45.4 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 34H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 20H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H).

4-Eicosyl-1-(2,5,8,11,14-pentaoxahexadecan-16-oxy)benzene [$CH_3(OCH_2CH_2)_5OPh(CH_2)_{19}CH_3$] Obtained as described above as a white crystalline solid in 66% yield after purification by column chromatography (70/30 ethyl acetate/hexane). mp 45.0 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b,

34H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 16H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H).

4-Eicosyl-1-(2,5,8,11,14,17-hexaoxanonadecan-19-oxy)benzene [$CH_3(OCH_2CH_2)_6OPh(CH_2)_{19}CH_3$] Obtained as described above as a white crystalline solid in 62% yield after purification by column chromatography (80/20 ethyl acetate/hexane). mp 44.3 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 34H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 20H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H).

4-Eicosyl-1-(2,5,8,11,14,17,19-heptaoxadocosan-22-oxy)benzene [$CH_3(OCH_2CH_2)_7OPh(CH_2)_{19}CH_3$] Obtained as described above as a white crystalline solid in 42% yield after purification by column chromatography (90/10 ethyl acetate/hexane). mp 43.6 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.85 (t, 3H), 1.18-1.32 (b, 34H), 1.55 (t, 2H), 2.52 (t, 2H), 3.35 (s, 3H), 3.52-3.75 (m, 24H), 3.82 (t, 2H), 4.08 (s, 2H), 6.80 (d, 2H), 7.04 (d, 2H).

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