Synthesis of Model Compounds for the Structure Elucidation of a Ladder Polymer from Benzo[1,2-*c*:4,5-*c*′]difuran and a Diquinone Derivative

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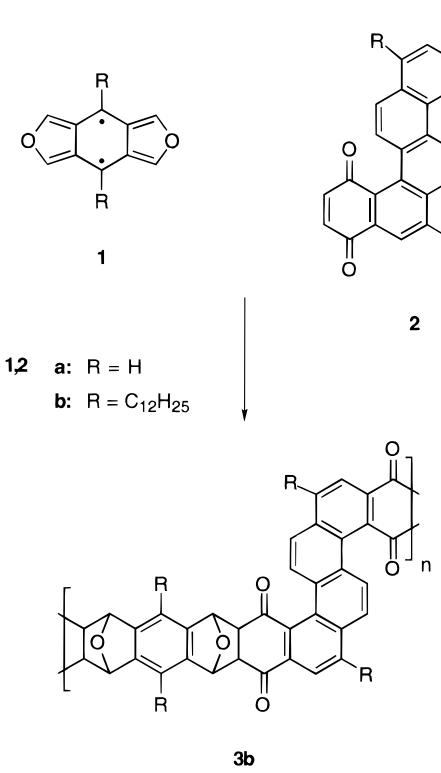
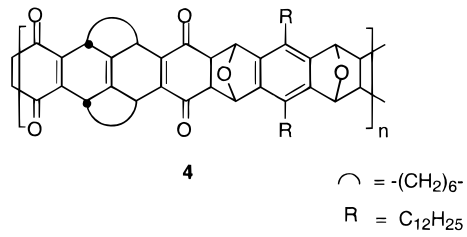
The high yield synthesis of the fully soluble acenequinone model compounds **6** and **7** as well as of the corresponding poly(acene quinone) derivative **4** is reported. Some of the diastereomers of **6** and **7** have been isolated and their structures clarified by NMR spectroscopy. This information was used as a basis for assigning the polymer's structure.

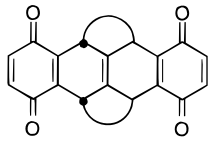
Within the general theme of ladder polymer synthesis,[1] a couple of years ago we reported the synthesis of the angularly annulated polymer **3b** from in situ prepared alkyl derivatives (**1b**) of Hart's benzo[1,2-*c*:4,5-*c*′]difuran (**1a**)[2] and the diquinone **2b** as monomers in a Diels−Alder polyaddition reaction (Scheme 1).[3] At the same time, we

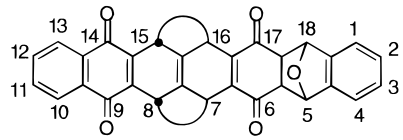
Scheme 1

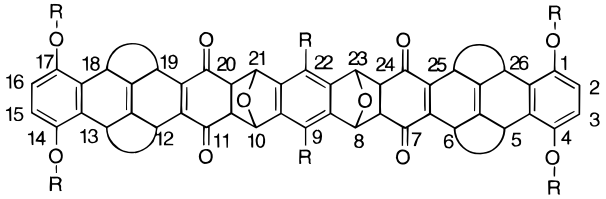


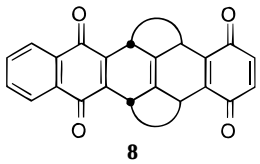
also synthesized the structurally similar, linearly annulated polymer **4** using the same bisdiene **1b** ($R = C_{12}H_{25}$) and the diquinone **5**.[4] Like polymer **3b** and other DA ladder polymers, the backbone of polymer **4** consists of a complex sequence of diastereomeric sites that render structure elucidation by NMR spectroscopy a rather difficult enterprise. Due to the lack of appropriate model compounds the structure of **4** could not be clarified with certainty. Here we report on the synthesis and isolation of various diastereomers of the two multicyclic compounds **6** and **7** and on how they were used to determine the structure of **4**. Polymer **4** is a potential precursor of polyacenequinones.[5]











[⊗] Abstract published in *Advance ACS Abstracts,* September 1, 1996.

(1) Schlüter, A.-D. *Adv. Mater.* **1991**, *3*, 282.
(2) Luo, J.; Hart, H. *J. Org. Chem.* **1988**, *53*, 1341.
(3) Blatter, K.; Schlüter, A.-D. *Macromolecules* **1989**, *22*, 3506.
(4) Godt, A. Dissertation, University of Mainz, Germany, 1991.

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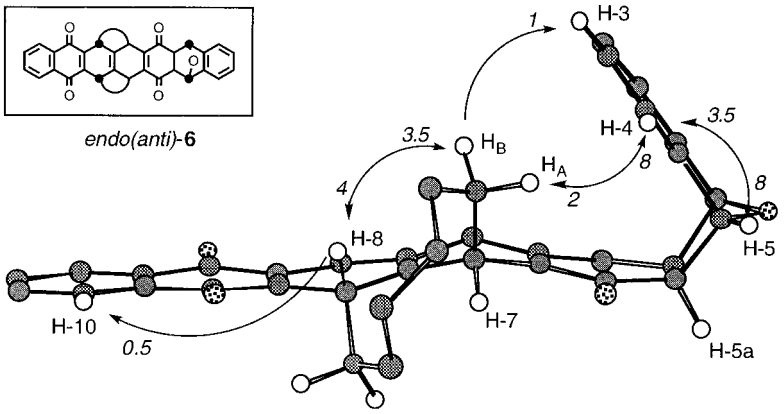


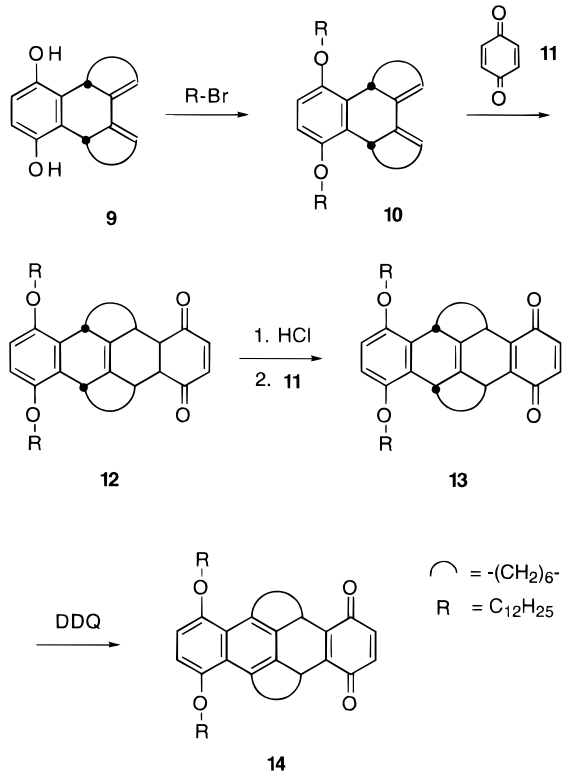
Figure 1. Calculated structure of *endo(anti)*-**6** (Chem 3D) with nuclear Overhauser enhancements (NOE's) between some hydrogens given as percentages.

Results and Discussion

The stereochemistry of the part of **4**'s backbone that contains the oxygen bridges has the greatest influence on the NMR spectra. The smallest model compounds having exactly this structural unit are 1:2 adducts of bisdiene **1b** and quinones. The quinones should both resemble the polymer as closely as possible and be monofunctional in order to prevent oligomerization. Quinone **8** and its adduct with **1b** (**1b**:**8** = 1:2) are known.[4] Unfortunately, this adduct (not shown) could not be used to obtain a complete set of NMR data because some of its diastereomers are practically insoluble despite their substitution with alkyl chains and rings. It was therefore necessary to synthesize a similar quinone with more flexible chains and its 1:2 adduct. Compound **13** (Scheme 2) seemed a good candidate. Its synthesis starts from the hydroquinone **9**, whose phenolic groups are etherified with straight dodecyl chains to give **10**. This diene readily undergoes DA cyclization with benzoquinone **11** to furnish **12**. Conversion into **13** was achieved when **12** was first enolized under acid catalysis followed by reacting the intermediately formed hydroquinone with **11**. Compound **13** was obtained on a 5 g scale in an overall yield of 48% based on **9**. This conversion cannot be achieved by dehydrogenation of **12** with dichlorodicyanobenzoquinone (DDQ), as was initially tried. This automatically leads to an "overdehydrogenation", and mixtures of **13** and **14** are obtained for which separation is difficult. By employing 2 equiv of DDQ, this was actually used to synthesize **14** directly from **12** in 76% yield. The structure of **14** rests upon the observation that neither **5** nor **14** can be oxidized with DDQ, indicating that the alternative aromatization of the ring adjacent to the quinoid moiety in **13** does not take place.

Synthesis of Model Compounds 6 and 7. Model compounds **6** and **7** were synthesized by reacting **8** with 1 equiv of isobenzofuran and **13** with 0.5 equiv of **1b**, respectively. Both reactions were carried out in refluxing toluene and gave the products in islolated yields of 94 and 98%. These high yields indicate a virtually complete conversion, which is an important prerequisite for compounds **5** and **1b** to be first choice monomers for a step-growth polymerization. In the case of **6** all possible four

Scheme 2



stereoisomers are formed in the approximate ratio *exo(syn)*:*exo(anti)*:*endo(syn)*:*endo(anti)* = 8:1:5:5.[6] The two *exo*-isomers were separated from the two *endo*-isomers by column chromatography. The assignment whether an isomer is *exo* or *endo* was easily accomplished using the Karplus equation. The dihedral angle between the protons H-5a(17a) and H-5(18) in *exo*-**6** is approximately 90°. Consequently, the signals appear as singlets, whereas the corresponding protons in the *endo*-isomers of **6** appear as doublet of doublets with coupling constants of approximately 4 and 2 Hz. The two pairs of stereoisomers of **6** were further separated by preparative HPLC. Except for *exo(anti)*-**6** the configuration of the isomers could be proven by NOE experiments. This is shown with *endo(anti)*-**6** (Figure 1). Figure 1 depicts the calculated

(5) (a) Miller, L. L.; Thomas, A. D.; Wilkens, C. L.; Weil, D. A. *J. Chem. Soc., Chem. Commun.* **1986**, 661. (b) Jozefiak, T. H.; Miller, L. L. *J. Am. Chem. Soc.* **1987**, *109*, 6560. (3) Rak, S. F.; Jozefiak, T. H.; Miller, L. L. *J. Org. Chem.* **1990**, *55*, 4794.

(6) The prefix *syn*/*anti* of **6** and **7** refers to the relative orientation of the oxygen bridge to the adjacent hexamethylen loop; the prefix *cis*/*trans* of **7** describes the relative orientation of two adjacent oxygen bridges. Since the hexamethylene loops are always arranged *trans* to one another[4] a descriptor referring to this is omitted for clarity.

Table 1. ^{1}H-NMR ($CDCl_3$) Chemical Shift Data of the Four Diastereomeric Isomers of Model Compound 7[a]

H-atom	A	B	C	D
H-2, -3, -15, -16	6.61 (s, 4H)	6.61 (s, 4H)	6.59 (s, 2H)$^{\alpha}$	6.59 (s, 2H)$^{\alpha}$
			6.52 (s, 2H)$^{\beta}$	6.53 (s, 2H)$^{\beta}$
H-5, -13, -18, -26	4.14 (d, 4H)	4.14 (d, 4H)	4.09 (d, 2H)$^{\alpha}$	4.12 (d, 2H)$^{\alpha}$
			3.88 $^{\beta,\gamma}$	4.00 (d, 2H)$^{\beta}$
H-6, -12, -19, -25	4.12 (m, 4H)	4.07 (m, 4H)	3.95 (m, 2H)$^{\alpha}$	3.90 $^{\alpha,\delta}$
			3.74 (m, 2H)$^{\beta}$	3.50 (d, 2H)$^{\beta}$
H-7a, -10a, -20a, -23a	2.82 (s, 4H)	2.77 (s, 4H)	2.43 (s, 2H)$^{\alpha}$	2.72 (s, 2H)$^{\alpha}$
			3.60 (m, 2H)$^{\beta}$	3.63 (m, 2H)$^{\beta}$
H-8, -10, -21, -23	5.40 (s, 4H)	5.43 (s, 4H)	5.29 (s, 2H)$^{\alpha}$	5.27 (2, 2H)$^{\alpha}$
			5.80 (dd, 2H)$^{\beta}$	5.81(m, 2H)$^{\beta}$
O-CH_2	3.92 (m, 8H)	3.92 (m, 8H)	3.88$^{\gamma}$	3.90$^{\delta}$
Bn	2.77 (m, 4H)	2.77 (m, 4H)	2.43 (m, 4H)	2.72 (m, 4H)
alkyl	1.0−2.2 (m, 168H)	1.0−2.2 (m, 168H)	1.0−2.2 (m, 168H)	1.0−2.2 (m, 168H)
CH_3	0.85 (t, 18H)	0.87 (t, 18H)	0.88 (t, 18H)	0.86 (t, 18H)

[a] Key: (A) *exo(syn)/exo(syn)-cis;* (B) *exo(syn)/exo(syn)-trans;* (C) *exo(syn)/endo(anti)-*(*cis* or *trans*); (D) *exo(syn)/endo(syn)-*(*trans* or *cis*); (α) *exo*; (β) *endo*; (γ) superimposed with O-CH_2, respectively, H-5, −13, −18, −26; (δ) superimposed with O-CH_2, respectively, H-6, −12, −19, −25$^{\alpha}$.

Table 2. Selected ^{13}C-NMR ($CDCl_3$) Chemical Shift Data of Four Diastereomers of Model Compound 7[a]

C-atom	A	B	C	D
C-5a$^{\gamma}$	131.39$^{\gamma}$	131.40$^{\gamma}$	131.31$^{\gamma}$	131.36$^{\gamma}$
C-6a	150.96	150.97	150.75$^{\alpha}$	150.73$^{\alpha}$
			149.57$^{\beta}$	149.76$^{\beta}$
C-7a	51.29	51.39	50.70$^{\alpha}$	51.36$^{\alpha}$
			48.37$^{\beta}$	49.52$^{\beta}$
C-7	196.79	196.85	196.30$^{\alpha}$	196.83$^{\alpha}$
			195.20$^{\beta}$	195.78$^{\beta}$
C-8	82.72	82.67	82.87$^{\alpha}$	82.40$^{\alpha}$
			81.50$^{\beta}$	80.58$^{\beta}$
C-8a	142.96	142.84	143.29$^{\alpha}$	142.83$^{\alpha}$
			141.39$^{\beta}$	141.67$^{\beta}$
C-9	124.97	124.89	125.46	126.47

[a] Key: (A) *Exo(syn)/exo(syn)/cis*; (B) *exo(syn)/exo(syn)/trans*; (C) *exo(syn)/endo(anti)-(cis* or *trans),* (D) *exo(syn)/endo(syn)-(trans* or *cis)*; (α) *exo*; (β) *endo*; (γ) there is some uncertainty in the assignment because of signal overlap with C-4a, 26a.

structure (Chem 3D) and the observed enhancements in percent. If, for example, the protons H_A of the hexamethylene loops are irradiated, the intensity of the signal of H-1(4) is enhanced by 8%; saturation of the H_B's results in a smaller enhancement of H-2(3) (1%), as expected. Both findings as well as all other observed NOE effects clearly establish the stereochemistry proposed.

In the case of model compound **7** 20 diastereomers are possible. From an inspection of the ^{1}H NMR spectrum of the raw reaction mixture it was concluded that the *exo*/*endo* ratio is approximately 3:1 and that *endo*/*endo* isomers are formed only in the subpercent range if they are formed at all.[7] This reduces the number of isomers to 14, 12 of which could be differentiated by ^{1}H NMR spectroscopy. Four of these 12, *exo(syn)/exo(syn)/cis*-**7**, *exo(syn)/exo(syn)/trans*-**7**, *exo(syn)/endo(anti)/(trans or cis)*-**7**, and *exo(syn)/endo(syn)/(cis or trans)*-**7**, were isolated by preparative HPLC. Tables 1 and 2 contain important ^{1}H and ^{13}C NMR chemical shifts. The assignment given was easily done on the basis of the respective data of **13** and the four diastereomers of **6**. The ^{1}H NMR data of **7** differ only slightly from those of **6** except for the hydrogens adjacent to oxygen bridges (H-8, -10, -21, -23). For example, these protons in *exo*(*syn*)/*endo*(*syn*)-**7**[8] (Tables 1 and 2; columns C, D) are shifted upfield as compared with *exo(syn)*-**6**. The same effect is observed for the *exo(anti)-endo*-isomers, the data of which are not included in the tables. They are minor components, and due to signal overlap not all of their signals were assigned. With all these data at hand it was possible to proof the structure of polymer **4** (see below).

Synthesis and Structure Elucidation of Polymer 4. Polymer **4** was synthesized in analogy to polymer **3b**[3] by generating bisdiene **1b** at 180 °C in tetralin or decalin from a multicyclic precursor[2] in the presence of diquinone **5**. The stoichiometry of the two components was exactly 1:1. After approximately 10 min at this temperature the color of the solution turned from an intense into a faint yellow and the reaction was finished [by size exclusion chromatography (SEC)]. Tetraphenylbenzene, a side product from the generation of **1b**, was removed by hot extraction with ethanol to give the fully soluble product in yields ranging from 75 to 85%. SEC shows two main peaks that are not monomodal. The one with long retention times (26−30 min) is due to oligomeric material (P_n = 3−4), whereas the one with short retention times (15−25 min) reflects the polymer.[9] The broadness of the latter peak may indicate aggregation on the column. These main peaks were separated by preparative SEC. The low molecular mass fraction seems to consist of cyclic oligomers, because neither the ^{1}H- nor the ^{13}C-NMR spectrum indicates end groups. This issue is presently under investigation. The ^{1}H NMR spectrum of the polymer shows very broad and unstructured signals that are not in disagreement with the proposed structure. Figure 2 compares the ^{13}C NMR spectra of polymer **4** and model compound **7**. The match between the relatively broad signals of **4** at δ = 195, 150, 142, 132−126, 83, 81, and 50 ppm and the corresponding signals of **7** is excellent and establishes the proposed structure of **4**.

Experimental Section

General Procedures. All experiments were performed under N_2. Solvents were dried using standard procedures. For column chromatography Kieselgel 60 was used as the stationary phase. ^{1}H-NMR spectra were determined at 270 MHz and ^{13}C-NMR spectra at 68 MHz. All quinoid compounds were handled in the dark.

***rel*-(1*R*,4*S*)-5,8-Bis(dodecyloxy)-1,2:4,3-di(1-heptanyl-7-ylidene)-1,4-dihydronaphthalene (10).** Hydroquinone **9** (2.96 g, 8.40 mmol) was dissolved in 100 mL of degassed acetone. Then, KO-*t*-Bu (2.07 g, 18.48 mmol) was added and the dark solution was stirred for 15 min before 1-bromodo-

(7) This follows from ^{1}H-NMR integration of *endo*- and *exo*-H-8, -10, -21, and -23.

(8) *Cis* or *trans* orientation of the oxygen bridges not determined.

(9) The relative molecular weight measured by size exclusion chromatography in THF versus polystyrene standard is: M_n = 21 341; M_w = 64 615; D = 3.03.

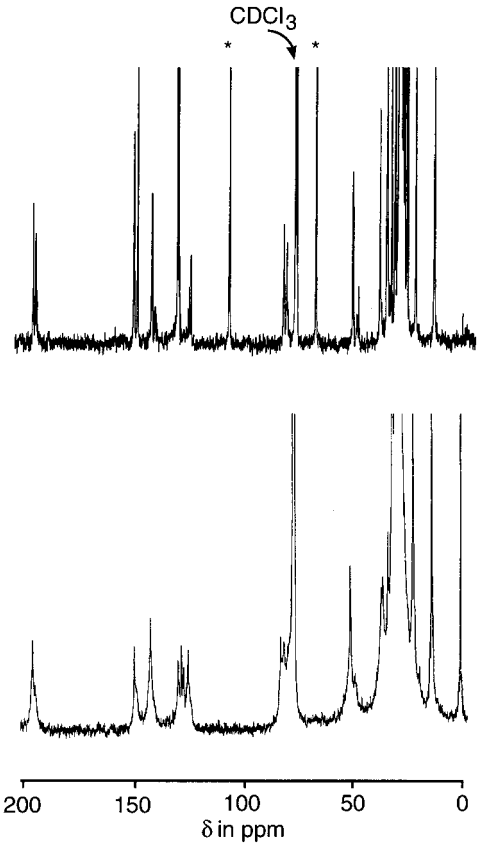


Figure 2. High-resolution ^{13}C NMR spectra of polymer **4** (bottom) and the mixture of model compound **7**'s diastereomers (top) in $CDCl_3$ at room temperature. The signals of model compound **7**, which are marked with an asterisk, cannot appear in the polymer spectrum.

decane (4.24 g, 17.01 mmol) was added in one portion. The mixture was refluxed for 5 h and after cooling neutralized with dilute aqueous HCl. The mixture was diluted with water and extracted with dichloromethane. The organic layer was dried ($MgSO_4$) and evaporated. The brown oil was purified by silica gel column chromatography (hexane) to afford 4.71 g (81%) of a colorless oil: ^1H NMR δ 0.9 (t, 6 H), 1.1−2.0 (m, 60 H), 2.17 (m, 2 H), 2.56 (m, 2 H), 3.89 (m, 4 H), 4.32 (t, 2 H), 6.0 (dd, 2 H), 6.59 (s, 2 H); ^{13}C NMR δ 14.12, 22.69, 24.03, 26.36, 26,84, 27.32, 27.61, 28.10, 29.37, 29.48, 29.70, 31.93, 35.14, 36.90, 68.07, 108.13, 122.62, 132.55, 140.93, 149.26. Anal. Calcd for $C_{48}H_{80}0_2$: C, 83.66; H, 11.70. Found: C, 83.88; H, 11.36.

***rel*-(5*R*,6*R*,11*S*,12*S*)-7,10-Bis(dodecyloxy)-4a,5,6,11,12,-12a-hexahydro-5,6:11,12-dihexano-1,4-naphthacenedione (12).** Compounds **10** (3.22 g, 4.67 mmol) and **11** (505 mg, 4,67 mmol) in 35 mL $CHCl_3$ were stirred for 2 h. The solvent was evaporated, and the remaining red oil was purified by silica gel column chromatography (hexane:AcOEt = 20:1) to afford 3.06 g (82%) of a yellow viscous oil: ^1H NMR δ 0.88 (t, 6 H), 1.1−2.1 (m, 64 H), 2.75 (m, 2 H), 3.0 (dd, 2 H), 3.88 (m, 6 H), 5.7 (s, 2 H), 6.55 (s, 2 H); ^{13}C NMR δ 14.11, 22.68, 25.14, 25.67, 26.34, 27.31, 27.55, 28.16, 29.35, 29.51, 29.65, 31.91, 35.36, 38.71, 45.11, 53.92, 68.33, 108.08, 133.89, 140.33, 141.55, 149.10, 199.40. Anal. Calcd for $C_{54}H_{84}O_4$: C, 81.35; H, 10.62. Found: C, 81.40; H, 10.34.

***rel*-(5*R*,6*R*,11*S*,12*S*)-7,10-Bis(dodecyloxy)-5,6,11,12-tetrahydro-5,6:11,12-dihexano-1,4-naphthacenedione (13).** Compound **12** (8.0 g, 10.03 mmol) in 150 mL of EtOH and 5 mL of 24% aqueous HCl were refluxed for 30 min. The solvent was evaporated and the residue dissolved in $CHCl_3$. This solution was washed with water and dried ($MgSO_4$). Then **11** (1.08 g, 10.03 mmol) was added, and the mixture was stirred for 1 h. After removal of the solvent EtOH was added. After being cooled at 2 °C for 1 d the red solid was filtered and dried *in vacuo*: yield 5.84 g (73%); ^1H NMR δ 0.90 (t, 6 H), 1.10−2.20 (m, 64 H), 3.92 (m, 6 H), 4.13 (d, 2 H), 6.65 (s, 2 H), 6.7 (s, 2 H); ^{13}C NMR δ 14.10, 22.67, 26.57, 27.76, 28.43, 29,33, 29.67, 30.29, 31.91, 33.71, 35.01, 36.12, 38.61, 68.08, 107.37, 130.78, 131.54, 136.43, 144.22, 149.44, 187.81. Anal. Calcd for $C_{54}H_{82}O_4$: C, 81.56; H, 10.39. Found: C, 81.58; H, 10.16.

***rel*-(5*R*,12*S*)-7,10-Bis(dodecyloxy)-5,12-dihydro-5,6:11,-12-dihexano-1,4-naphthacenedione (14).** A solution of 200 mg (0.25 mmol) of quinone **13** and 63 mg (0.28 mmol) of DDQ was refluxed in $CHCl_3$ for 2 h. The solvent was removed, and the product was purified by silica gel chromatography (hexane: AcOEt = 30:1). After 1 d the product solidified to give 151 mg (76%) of a purple solid: ^1H-NMR δ 0.90 (t, 6H), 1.1−2.2 (m, 60 H), 2.88 (m, 2 H), 3.99 (m, 4 H), 4.11 (m, 2 H), 4.76 (m, 2 H), 6.77 (s, 4 H); ^{13}C-NMR δ 14.11, 22.68, 24.34, 24.74, 26.54, 29.36, 29.52, 29.63, 29.76, 30.04, 31.91, 32.40, 36.59, 37.82, 69.29, 105.92, 126.65, 135.95, 136.02, 136.19, 146.53, 150.99, 186.68. Anal. Calcd for $C_{54}H_{80}O_4$: C, 81.77; H, 10.17. Found: C, 81.58; H, 9.74.

5,5a,7,8,15,16,17a,18-Octahydro-5,18-epoxy-7,8:15,16-dihexano-6,9,14,17-heptacenetetrone (6). A solution of diquinone **8**[10] (2.0 g, 3.95 mmol) and 1,2,3,4-tetraphenyl-1,4-dicarboxy-9,10-diepoxy-1,4,9,9a,10,10a-hexahydroanthracene[11] (2.087 g, 3.95 mmol) was refluxed for 30 h in 100 mL of dry toluene under exclusion of light. The solvent was removed, and chromatography (silica gel, toluene) provided after separation of tetraphenylbenzene 1.27 g (51.5%) of *exo*-**6** and 1.04 g (42.2%) of *endo*-**6**. Further separation[12] was done by HPLC (silica gel, 15% ethyl acetate in hexane).

***exo(syn)*-6**: ^1H-NMR ($CDCl_3$): δ 1.3−2.3 (m, 24 H), 2.88 (s, 2H), 4.07 (d, 2H), 4.13 (d, 2H), 5.46 (s, 2H), 7.24 (m, 2H), 7.37 (m, 2H), 7.70 (m, 2H), 8.07 (m, 2H); ^{13}C-NMR ($CDCl_3$): δ 27.77, 28.14, 29.20, 30.02, 33.77, 34.61, 36.30, 37.60, 51.56, 71.39, 83.64, 119.52, 126.11, 127.61, 130.16, 132.43, 133.42, 143.94, 146.26, 150.24, 184.81, 196.91; HRMS (EI) calcd for $C_{42}H_{40}O_5$ 624.287 58, found 624.287 35.

***exo(anti)*-6**: ^1H-NMR ($CDCl_3$) δ 1.3−2.3 (m, 24H), 2.91 (s, 2H), 4.11 (m, 4H), 5.99 (s, 2H), 7.23 (m, 2H), 7.36 (m, 2H), 7.70 (m, 2H), 8.07 (m, 2H); ^{13}C-NMR ($CDCl_3$) δ 27.69, 28.21, 29.08, 30.06, 33.76, 34.82, 36.19, 37.62, 51.42, 82.46, 119.51, 126.13, 127.35, 130.20, 132.43, 133.44, 143.96, 146.29, 150.37, 184.79, 195.58; HRMS (EI) calcd for $C_{42}H_{40}O_5$ 624.287 58, found 624.287 29.

***endo(syn)*-6**: ^1H-NMR ($CDCl_3$) δ 1.22 (m, 4H), 1.5−2.3 (m, 20H), 3.47 (d, 2H), 3.67 (dd, 2H), 3.95 (d, 2H), 5.78 (dd, 2H), 7.03 (m, 2H), 7.18 (m, 2H), 7.67 (m, 2H), 8.02 (m, 2H); ^{13}C-NMR ($CDCl_3$) δ 27.86, 29.52, 33.98, 34.13, 36.38, 36.63, 49.59, 82.18, 121.08, 126.01, 127.35, 129.91, 132. 37 133.36, 142.25, 145.97, 148.75, 184.83, 195.86; HRMS (EI) calcd for $C_{42}H_{40}O_5$ 624.287 58, found 624.287 78.

***endo(anti)*-6**: ^1H-NMR ($CDCl_3$) δ 0.8 (m, 2H), 1.14 (m, 2H), 1.26 (m, 2H), 1.55−2.2 (m, 18H), 3.63 (dd, 2H), 3.77 (d, 2H), 3.95 (d, 2H), 5.78 (dd, 2H), 7.11 (m, 2H), 7.20 (m, 2H), 7.72 (m, 2H), 8.04 (m, 2H); ^{13}C-NMR ($CDCl_3$) δ 27.77, 27.91, 29.48, 29.72, 33.86, 34.06, 36.84, 48.53, 82.55, 121.04, 126.02, 127.48, 129.96, 132.39, 133.36, 141.94, 146.01, 148.85, 184.87, 195.06; HRMS (EI) calcd for $C_{42}H_{40}O_5$ 624.287 58, found 624.287 90.

9,22-Didodecyl-1,4,14,17-tetrakis(dodecyloxy)-5,6,7a,8,-10,10a,12,13,18,19,20a,21,23,23a,25,26-hexadecahydro-8,-23:10,21-diepoxy-5,6:12,13:18,19:25,26-tetrahexano-7,11,-20,24-undecacenetetrone (7). A solution of quinone **12** (608.2 mg, 0.76 mmol) and 1,4:8,11-dicarboxy-6,13-didodecyl-5,14:7,12-diepoxy-4a,5,7,7a,11a,12,14,14a-octahydro-1,2,3,4,8,9,-10,11-octaphenylpentacene[3] (503.2 mg, 0.38 mmol) in 50 mL of toluene was refluxed for 36 h under exclusion of light. The solvent was evaporated to give the crude product. The tetraphenylbenzene was removed by silica gel column chromatography (toluene:hexane = 1:2), and then the solvent was changed to toluene to eluate the product. Evaporating of the solvent *in vacuo* provided a light yellow solid (777 mg, 98%). A separation of the isomers was done with HPLC (silica gel,

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(12) The separation was done with a nonrepresentative mixture of the four diastereomers.

dichloromethane). ^{1}H and ^{13}C-NMR: see tables 1 and 2. Anal. Calcd for $C_{142}H_{218}O_{10}$: C, 81.79; H, 10.54. Found: C, 81.50; H, 10.36.

Polymer 4. A suspension of diquinone **5**[10] (498.3 mg, 1.091 mmol) and 1,4:8,11-dicarboxy-6,13-didodecyl-5,14:7,12-diepoxy-4a,5,7,7a,11a,12,14,14a-octahydro-1,2,3,4,8,9,10,11-octaphenylpentacene[3] (1.436 g, 1.091 mmol) in 33 mL of decalin was heated to 180 °C for 25 min. After the mixture was cooled to rt the solvent was removed *in vacuo*. The remaining residue was extracted with hot EtOH for 3 d to give a material that after lyophilization using benzene afforded 815 mg (78%) of a white solid. Anal. Calcd for $(C_{64}H_{86}O_6)_n$: C, 80.80; H, 9.11. Found: C, 79.45; H, 8.42.

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