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Synthesis of Discotic Columnar Side-Chain Liquid Crystalline Polymers by Ring-Opening Metathesis Polymerization (ROMP)

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ABSTRACT: Discotic liquid crystalline polymers bearing alkoxy-substituted triphenylene moieties in the side-chain were synthesized by ring-opening metathesis polymerization using a well-defined ruthenium initiator. To elucidate the effect of backbone flexibility on the mesomorphism, norbornene and cyclobutene monomers containing triphenylene moieties were synthesized, which yielded the relatively rigid poly(norbornene)s and the more flexible poly(butadiene)s, respectively, after polymerization. To further increase the backbone flexibility, the poly(butadiene)s were hydrogenated using Crabtree's catalyst to yield triphenylene-substituted poly(butane)s. The mesomorphic behavior of the polymers has been investigated by differential scanning calorimetry (DSC) and powder diffraction X-ray scattering (wide angle X-ray scattering, WAXS). All polymers bearing a 2,3,6,7,19-pentakis(decyloxy)triphenylene-based mesogenic unit exhibit enantiotropic discotic hexagonal mesophases, while the pentoxy analogues do not display liquid crystalline behavior. No effect of backbone rigidity on the mesomorphism could be detected.

Introduction

Liquid crystals are promising materials for electronic applications, e.g., in displays and photoconducting materials, because they can be oriented by applied electric and magnetic fields.[1−3] In particular, discotic liquid crystals, which bear a flat, rigid core molecule as the mesogenic unit and are able to self-assemble into highly ordered columnar phases (Figure 1),[4,5] have been studied intensely over the last few years for potential applications in these fields.[2,3]

A variety of mesogens such as 2,3,6,7,10,11-hexaalkoxytriphenylenes,[2,6−31] phthalocyanines,[3,32−43] cyclotetraveratrylenes,[44] sugars,[45,46] transition metal complexes,[47−49] hydrogen-bonded oligoheterocycles,[50−52] and diaza-crowns[53] have been used to obtain discotic liquid crystalline materials. Some of these materials show high charge carrier mobilities; e.g., triphenylenes in the highly ordered hexagonal columnar mesophases (D_h) display charge carrier mobilities of 0.1 cm^2 V^{-1} s^{-1}, which are higher than any organic material except single crystal phases.[2,21−23,54−56] This enhanced photoconductivity has been attributed to long-range ordering along the columns of the discotic hexagonal and helical phases.

To move toward possible industrial applications, polymeric materials are required because discotic liquid crystalline polymers provide a mechanically stable arrangement of the columnar assembly. Only a few examples of such oligomeric or polymeric discotic liquid crystalline materials are known to date.[7−14,31,32,41,44,45,57−59] Structural variations of these systems bear the mesogens as a side group (Figure 2A),[7−9,11] in the main chain (Figure 2B),[10,13,14,31,32,41,44,45,58,59] or in a polymeric network (Figure 2C).[12] Most materials in these examples were synthesized in step polymerizations or polymer-analogous reactions, thus limiting control over molecular weight and polydispersity. We herein present ring-opening metathesis polymerization (ROMP), which is able to overcome these limitations, as an alternative method to obtain polymeric discotic liquid crystalline materials.

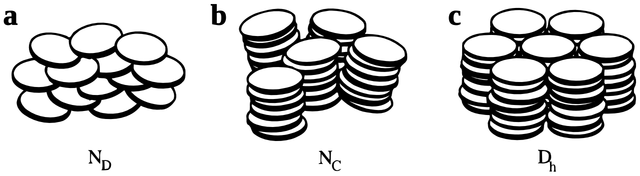


Figure 1. Possible alignment of the discotic core molecules in discotic liquid crystals: (a) N$_D$; discotic nematic mesophase; (b) N$_C$, discotic columnar mesophase; (c) D$_h$, discotic hexagonal mesophase.

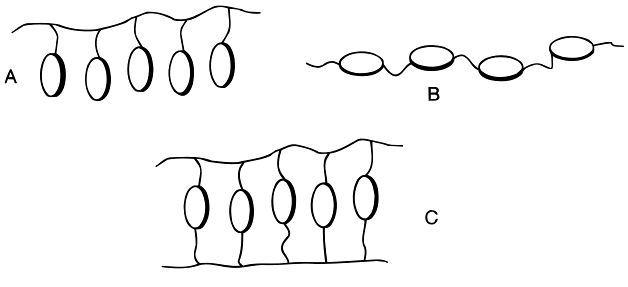
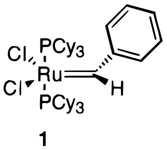


Figure 2. Possible molecular architectures for discotic liquid crystalline polymers: (A) side-chain liquid crystalline polymer; (B) main-chain liquid crystalline polymer; (C) discotic network.

In the last decade, living ROMP has been established as an efficient method to control the polymer's molecular structure, size, and bulk properties by variations at the molecular level.[60−63] Recently, we reported the synthesis of a new class of well-defined ruthenium complexes that are highly active in ROMP and ring-closing metathesis (RCM).[64−67] Initiator **1** $(PCy_3)_2Cl_2Ru(=CHC_6H_5)$ in particular has been shown to polymerize a large variety of strained cyclic olefins in a living fashion as a result of its remarkable stability toward functional groups.[64,65] For example, it was shown that cyclobutenes and norbornenes, bearing a large variety of side groups, can be polymerized in a living fashion using initiator **1**, yielding polymers and block copolymers with low polydispersities.[68,69]



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We herein present a new strategy for the synthesis of discotic side-chain liquid crystalline materials using

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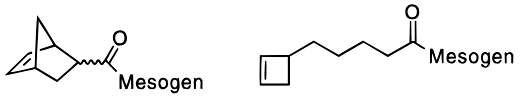
ring-opening metathesis polymerization. Utilizing cyclobutene or norbornene monomers **2** and **3**, we obtained the corresponding poly(norbornene)s and poly(butadiene)s bearing pentoxy- or decyloxy-substituted triphenylenes. To investigate the role of backbone rigidity on the phase behavior, hydrogenation of the poly(butadiene)s was performed using Crabtree's catalyst [Ir(COD)(Cy$_3$)(py)]PF$_6$ to further increase the flexibility of this system. The phase behavior of the polymers was examined by differential scanning calorimetry (DSC) and powder diffraction X-ray scattering. Different routes for the synthesis of the monomers—depending on the length of the alkoxy chains and the polymerizable unit—are presented, and a new strategy for the aryl−aryl coupling to obtain alkoxy-substituted triphenylenes is introduced.



2 **3**

Results and Discussion

Monomer Synthesis. Alkoxy-substituted triphenylenes represent the most widely studied class of discotic mesogens.[2,6−18,20−31,70] Synthetic strategies for these triphenylenes are based on the trimerization of *o*-dialkylbenzene derivatives,[17] terphenyl intermediates,[71] or the biphenyl route.[15,18,27,70] The *o*-dialkylbenzene approach provides a statistical mixture of triphenylenes carrying two different alkoxy groups, while the reaction conditions (67% sulfuric acid) are incompatible with a large number of functionalities.[17] The terphenyl strategy permits the preparation of highly functionalized triphenylenes, especially for the synthesis of triphenylenes with less than six alkoxy groups.[71] The recently developed biphenyl route is based on the aryl−aryl coupling of a biphenyl with a catechol derivative using FeCl$_3$ as coupling agent, which allows large scale preparations in good yields.[15,18,27,70] However, functionalities have to be introduced in a later step by a selective ether cleavage because of the incompatibility of most functional groups with FeCl$_3$.[15,24−27,70] Another approach introduces the functionality by an electrophilic aromatic substitution after the coupling.[16] We herein present a modified strategy for the synthesis of functionalized triphenylenes based on the biphenyl route, using VOF$_3$ as coupling agent. This route is compatible with most functional groups, allowing their introduction prior to the coupling reaction.

Schemes 1 and 2 show the synthesis of the triphenylene precursors **15** and **16** by oxidative aryl−aryl coupling of the tetraalkoxy-substituted biphenyls **8** or **10** with the bis-alkylated catechols **13** or **14** using VOF$_3$ in the presence of boron trifluoride−diethyl ether.[72−74] **13** and **14** were obtained in high yields by monoalkylation of catechol using the corresponding 1-bromoalkanes, followed by treatment with 1-bromo-12-dodecanol. Biphenyl **8** was synthesized by Suzuki-coupling of the aryl bromide **6** with the arylboronic acid **7**.[75] **7** was derived from the conversion of **6** into the boronic acid. Alkylation of catechol followed by bromination yielded **6** in high yield. Biphenyl **10**, by contrast, was obtained in quantitative yield from the Ulman coupling of the aryl iodide **9**, which was obtained by alkylation and subsequent iodination of catechol.

Norbornene monomers **17** and **18** were synthesized by esterfication of norborn-2-ene-5-carbonyl chloride[76] with the hydroxy-functionalized triphenylenes **15** and **16**.[68] The reaction was carried out in tetrahydrofuran in the presence of triethylamine to yield **17** and **18** in nearly quantitative yields (Scheme 3).

It was anticipated that a more flexible backbone such as a poly(butadiene) might facilitate a better alignment of the mesogenic units. To obtain a poly(butadiene) backbone, the corresponding monomers **19** and **20** were synthesized in analogy to literature procedures[68,69] by the initial conversion of [5-(2-cyclobutenyl)pentanoic acid[69] to the corresponding acid chloride and subsequent esterfication with the triphenylene precursors **15** and **16** in high yields (Scheme 4).

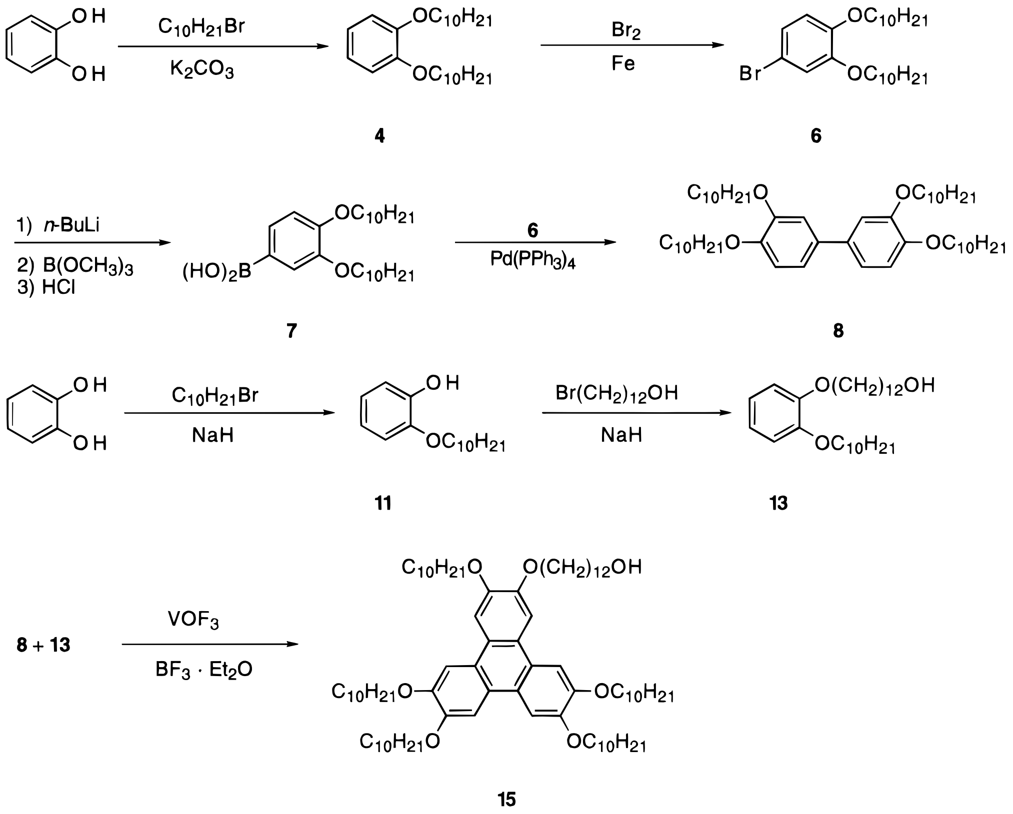
Polymer Synthesis. Polymerization of the norbornene monomers **17** and **18** was carried out using initiator **1**. The monomers were polymerized for 6 h at room temperature in dichloromethane followed by treatment with an excess of ethyl vinyl ether to cleave the initiator from the polymer chain (Scheme 5). Repeated precipitation from methanol yielded the polymers **21** and **22** as white solids in quantitative yield. The polymerizations resulted in material with low PDI(s) between 1.09−1.17 (Table 1).

The polymerizations of the cyclobutene monomers **19** and **20** were performed with initiator **1** in dichloromethane at 45 °C in a sealed vial for 1.5 h (Scheme 6). The initiator was then cleaved from the polymer chain by precipitation into methanol containing 5% 1 N hydrochloric acid, as previously described for poly(butadiene)s[68,69] to yield the polymers **23** and **24** in quantitative yields. In analogy to the poly(norbornenes), **21** and **22**, low polydispersities of 1.11−1.19 were observed, as presented in Table 1.

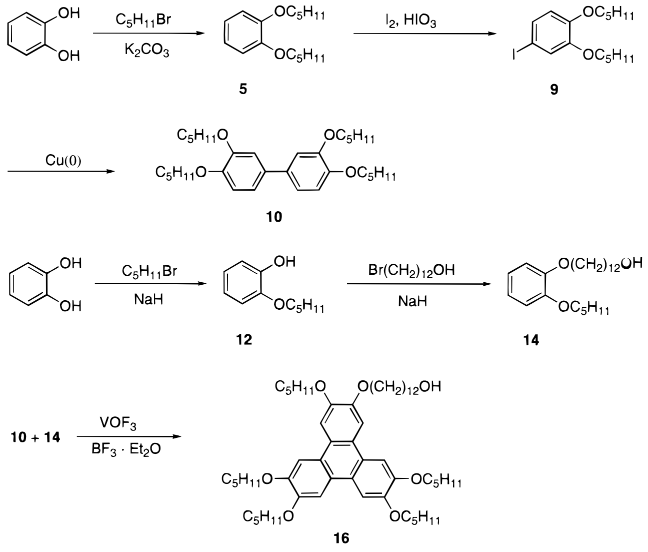
We recently investigated the influence of backbone rigidity on the thermotropic behavior of calamitic side-chain liquid crystalline polymers synthesized by ROMP.[68] In these studies we compared the relatively rigid poly(norbornene) backbone with the more flexible poly(butadiene) using a nitrostilbene as the mesogenic unit. Our investigations showed that, as a consequence of an increase in backbone flexibility, a better alignment of the mesogens was possible, resulting in a nematic mesophase behavior for the poly(norbornenes) and a smectic mesophase for the poly(butadiene)s. To further investigate the influence of backbone flexibility in the systems presented herein, we carried out a hydrogenation of the butadiene backbone of polymers **23** and **24** using Crabtree's catalyst [Ir(COD)(Cy$_3$)(py)]PF$_6$ to obtain poly(ethylene)-based polymers **25** and **26**, which have the most flexible backbone possible. The hydrogenation was performed in dichloromethane at 55 °C at 120 psi for 16 h with 5−10 mol % catalyst to yield polymers **25** and **26** in quantitative yields, as shown in Scheme 7. The molecular weights and the polydispersities for polymers **25** and **26** are summarized in Table 1.

Thermal Characterization of the Polymers. Analysis of the phase behavior of polymers **21**−**26** was accomplished using DSC and powder diffraction X-ray scattering (wide angle X-ray scattering; WAXS). For the DSC measurements all heating and cooling scans were completely reversible for each of the polymers. The second heating and first cooling scans are reported in all cases. It was not possible to obtain optical polarized microscopical pictures of these compounds due to the formation of a homogeneous film, as previously de-

Scheme 1



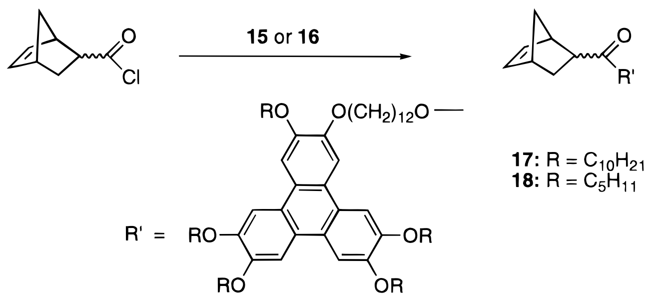
Scheme 2



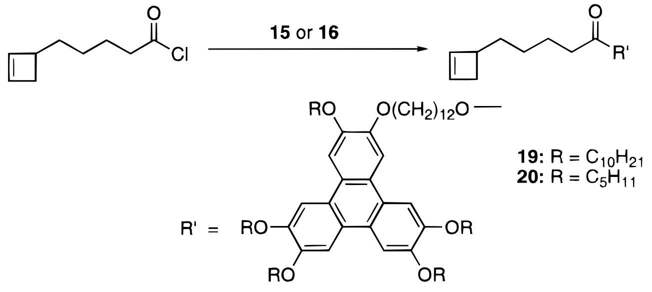
scribed for discotic polymers.[7,10]

DSC Measurements. All monomers exhibit only a single phase transition from the crystalline into the isotropic state. The DSC results of the polymers **21**–**26** are summarized in Table 1. Figure 3 shows the DSC traces of polymer **25** as a characteristic example of results obtained for polymers **21**, **23**, and **25**, all bearing a decyloxy-substituted triphenylene. These polymers display a broad glass transition and two first-order transitions during heating, while only one exothermic signal and a broad glass transition are observed during cooling. Similar behavior has been observed previously for discotic liquid crystalline polymers.[7,14,32,33,44,45] The glass transition temperature (T_g) decreases from −3 °C for the poly(norbornene)s to −17 and −18 °C for the more flexible poly(butadiene)s and poly(butane)s, respectively. For all polymers, the first disordering endotherms are observed between 34 and 37 °C while the transition into the isotropic melt is found between 42 and 45 °C. Due to a supercooling effect during the cooling scans, the transitions from the isotropic melt into the liquid crystalline phase and the broad glass transitions (over temperature ranges of 35 °C) are shifted to lower temperatures for polymers **21**, **23**, and **25**. As a result, the exotherms can be observed at 20, 28, and 26 °C and the glass transitions at −4, −34, and −34 °C, respectively.

Scheme 3



Scheme 4



For polymers **22**, **24**, and **26**, which bear pentoxy-substituted triphenylenes, only a glass transition was

Scheme 5

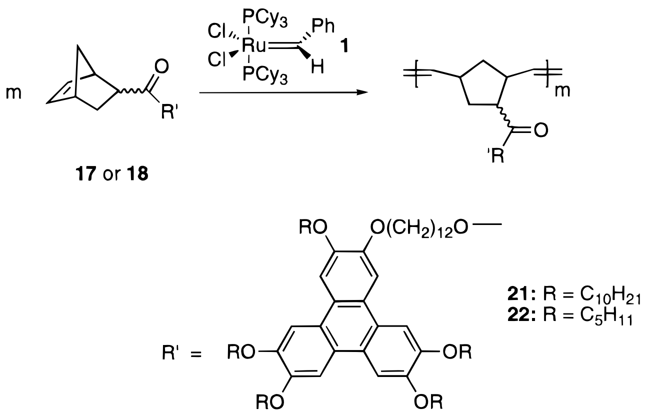
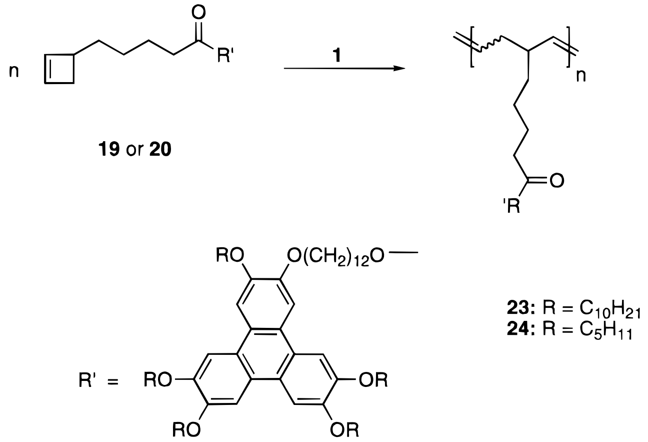


Table 1. Polymerization Results

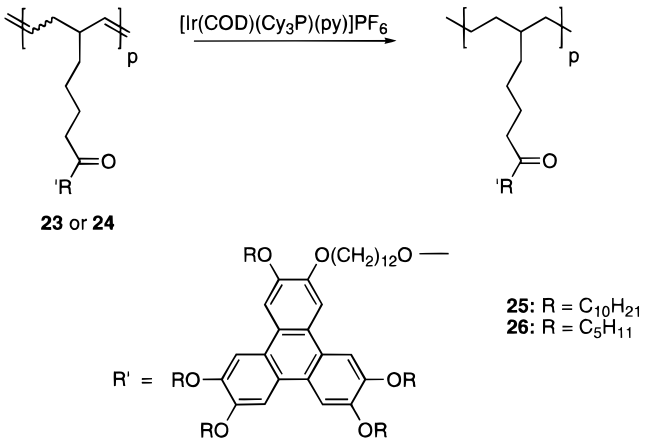
polymer	$\bar{M}_n$[a]	PDI[a]	phase transition[b] (°C) heating	phase transition[b] (°C) cooling
21	46 500	1.09	g-3D$_{hd1}$36D$_{hd2}$42i	i20D$_{hd}$-4g
22	48 500	1.17	g-4i	i-11g
23	33 000	1.11	g-17D$_{hd1}$37D$_{hd2}$45i	i28D$_{hd}$-34g
24	157 000	1.19	g-12i	i-12.5g
25	50 000	1.11	g-18D$_{hd1}$34D$_{hd2}$43i	i26D$_{hd}$-34g
26	125 000	1.32	g-17i	i-19g

[a] Determined by gel permeation chromatography in CH_2Cl_2 relative to monodispersed polystyrene standards. [b] Analysis by differential scanning calorimetry with a scan rate of 10 K/min.

Scheme 6



Scheme 7



observed, but disordering peaks were not, clearly showing that these polymers do not exhibit liquid crystalline behavior.

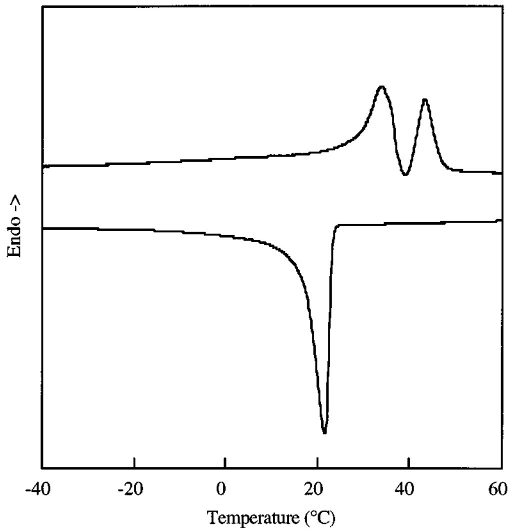


Figure 3. Normalized DSC thermograms for the poly(butadiene) **25** with a scan rate of 10 °C/min. (The first cooling and the second heating scans are shown; all heating and cooling scans were completely reversible for each of the polymers.) The phase transition enthalpies of the first disordered transitions ($\Delta H(\mathrm{D_h} \rightarrow \mathrm{i})$) range from 7.5 J g^{-1} (in the case of polymer **25**) to 10 J g^{-1} (in the case of polymer **21**).

X-ray Investigations. Diffraction data were collected on nonoriented samples of the polymers at temperatures ranging from −73 to +77 °C. Figure 4 displays the diffraction patterns obtained for compound **25** at 0, 38, and 77 °C. The *d*-spacings in Table 2 were calculated from 2θ values according to the Bragg equation ($n\lambda = 2d \sin \theta$).

The X-ray diagrams for polymers **21**, **23**, and **25** between −73 and 30 °C display a high-intensity reflection at low Bragg angles, a set of weak, secondary reflections in the small angle region, and an amorphous halo and broad reflection at higher angles, as reported before for discotic columnar side-chain liquid crystalline polymers (Figure 4a).[9] The intense reflection corresponding to a spacing of 19.64 Å for **21**, 19.81 Å for **23**, and 19.13 Å for **25** (at 0 °C) can be attributed to the (100) reflection of a hexagonal columnar (D_h) mesophase. Small peaks observed around 12 Å and 10 Å for all three polymers result from the (110) and (200) reflections respectively, while the broad peak around 7 Å could not be unequivocally assigned at the present state. These three peaks at small angles with values of Q in the ratio $1{:}\sqrt{3}{:}2$ indicate that a hexagonal lattice exists from low temperatures to the isotropic phase with intercolumnar distances of 22.68 Å for **21**, 22.78 Å for **23**, and 22.00 Å for **25** (at 0 °C) in the mesophase. The diffuse halo at around 4.3 Å is characteristic for a liquidlike order of the alkyl chains. The lack of an ordered peak between 3 and 4 Å may indicate a disordered stacking of the core molecules but it is also possible that this peak, which is normally weak, cannot be clearly observed above the background noise. In fact, the spectra of polymers **23** and **25** display an extremely weak peak on the top of the diffuse halo, which could correspond to the ordered peak. These data indicate that a discotic hexagonal mesophase of polymers **21**, **23**, and **25** exists that is best identified as D$_{hd}$ (discotic hexagonal disordered, labeled as D$_{hd1}$ in Table 1), but a D$_{ho}$ (discotic hexagonal ordered) mesophase cannot be entirely excluded.

No significant difference in the X-ray patterns of **21**, **23** and **25** could be observed on heating the polymers

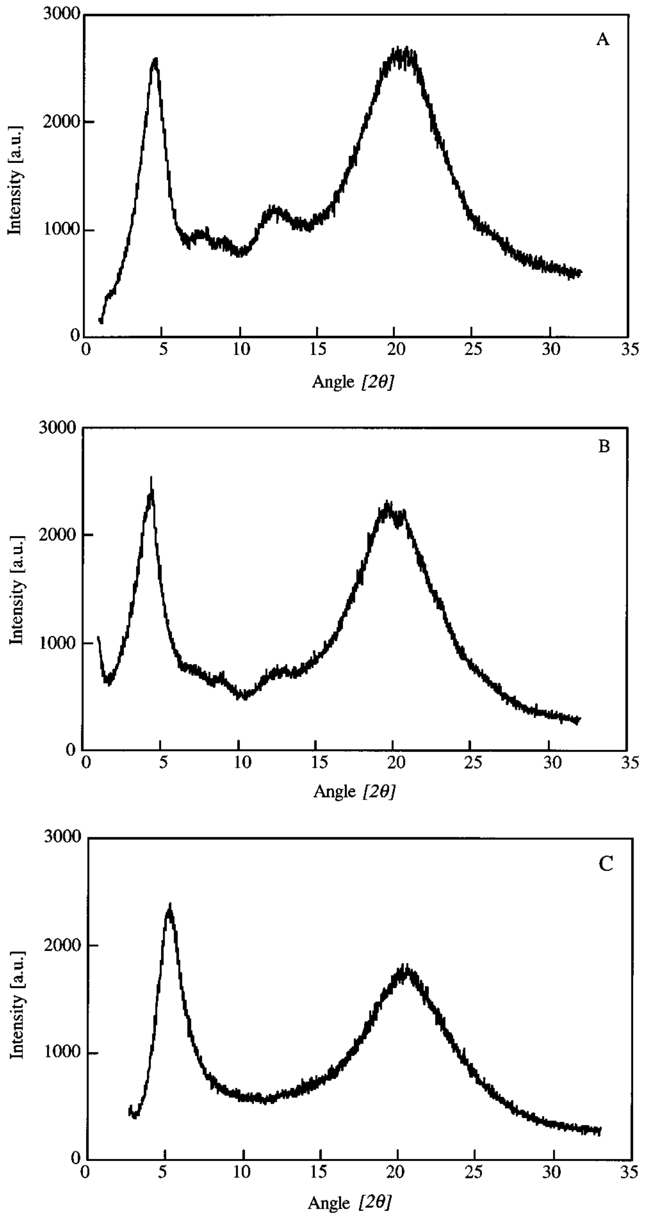


Figure 4. Powder X-ray diffractograms of **25** at (A) 0 °C, (B) 38 °C, and (C) 77 °C.

Table 2. Selected 2*θ* (deg) and Derived *d*-Spacing Values (Å) and Miller Indices (*hkl*) of Polymers 21, 23 and 25 at 0 °C and 77 °C from Powder X-ray Diffraction Measurements

polymer	*T* (°C)	2*θ* (deg)	d_{100} (Å)	2*θ* (deg)	d_{110} (Å)	2*θ* (deg)	d_{200} (Å)
21	0	4.50	19.64	7.64	11.57	8.92	9.91
21	77	4.20	21.04				
23	0	4.46	19.81	7.08	12.49	8.80	10.05
23	77	4.18	21.14				
25	0	4.62	19.13	7.26	12.18	8.84	10.00
25	77	4.18	21.14				

from 25 to 37−42 °C (Figure 4b), although a first-order transition in the DSC could be observed between 34 and 37 °C. Therefore, a discotic hexagonal mesophase, which is assigned as D_{hd2} in Table 1, was identified in this temperature range as well.

As shown in Table 2, a trend to longer distances between the columns with increasing temperatures can be observed in all polymers as a result of increased flexibility. When the temperature is increased above 43 °C (the transition temperature from the liquid crystalline mesophase into the isotropic melt as observed by DSC), only the (100) peak and the diffuse halo were observed in all cases while the higher ordered reflections (110 and 200) were no longer present, indicating the existence of a lower order in the isotropic melt as previously observed.[77] Figure 4c shows the diffraction pattern of polymer **25** at 77 °C. In analogy to the DSC results, no difference in the X-ray patterns of polymers **21**, **23**, and **25** was observed, suggesting that the backbone rigidity does not play a role in the discotic liquid crystalline phase behavior of these polymers.

Unlike compounds **21**, **23**, and **25**, no diffraction pattern corresponding to a discotic mesophase was observed for polymers **22**, **24**, and **26**, indicating that these polymers exist only as amorphous material, which is consistent with the DSC results. Therefore, the DSC and X-ray measurements prove that the pentoxy substitution on triphenylenes is too short to stabilize a liquid crystalline mesophase.

Conclusions

Ring-opening metathesis polymerization of norbornene- and cyclobutene-based monomers bearing alkoxy-substituted triphenylenes to obtain poly(norbornene)s and poly(butadiene)s with low polydispersities has been demonstrated. Poly(butane)-based polymers have been synthesized by hydrogenation of the poly(butadiene)s using Crabtree's catalyst. The examination of the phase behavior of these polymers has been accomplished by DSC and X-ray diffraction. DSC measurements show that all polymers bearing decyloxy-substituted triphenylenes display mesogenic behavior up to 45 °C, while the polymers containing pentoxy-substituted triphenylenes do not exhibit a mesophase behavior. The same trend can be substantiated by X-ray diffraction measurements, which showed a discotic hexagonal mesophase in the case of the decyloxy-substituted triphenylene containing polymers but not for the pentoxy-substituted polymers. In contrast to previous studies on similar systems, no dependency of the mesophase on the backbone rigidity could be established. For the mesogen synthesis, a rational, universally applicable strategy using vanadium oxyfluoride as the aryl−aryl coupling reagent has been developed. Because this is compatible with a large variety of functionalities, this route should provide convenient access to functionalized triphenylenes. The synthesis introduced herein presents a facile and efficient route to discotic, columnar, side-chain liquid crystalline polymers, which are promising material for use as hole transporters in light emitting diodes (LED) or as photoconducting materials. Photoconductivity measurements for these systems are currently under investigation.

Experimental Section

General Information. Argon was purified by passage through columns of BASF R-11 catalyst (Chemalog) and 4 Å molecular sieves (Linde). NMR spectra were recorded on a GE QE-300 Plus (300.1 MHz; 75.49 MHz ^{13}C) spectrometer; IR spectra were recorded on a Perkin-Elmer 1600 series FT-IR spectrometer. Gel permeation chromatographs were obtained on a HPLC system using an Altex model 110A pump, a Rheodyne model 7125 injector with a 100 μL injection loop, two American Polymer Standards 10 μm mixed bed columns, and a Knauer differential-refractometer using CH_2Cl_2 as eluent at a 1.0 mL/min flow rate. Molecular weights and polydispersities were reported versus monodispersed polystyrene standards. Differential scanning calorimetry was carried out on a Perkin-Elmer DSC-7 with a scan rate of 10 °C/min. Wide angle X-ray scattering analysis was performed by employing a goniometer from Siemens (D 500) with Cu-Kα radiation of 1.5418 Å.

Materials. CH_2Cl_2 was distilled from calcium hydride and degassed by repeated freeze−pump−thaw cycles. All other solvents were used without further purification unless noted otherwise. Chemicals were purchased from the Aldrich Chemical Co. unless noted otherwise and used without further purification. Crabtree's catalyst ($[Ir(COD)(Cy_3P)(py)]PF_6$) was purchased from Strem Chemical Co. Norborn-2-ene-5-carbonyl chloride and 5-(2-cyclobutenyl)pentanoic acid were synthesized as previously reported.[68,69]

Preparation of 11-((12′-Hydroxydodecyl)oxy)-2,3,6,7,-10-pentakis(decyloxy)triphenylene (15). A solution of **8** (0.7 g, 0.9 mmol) in 50 mL of dichloromethane was treated sequentially with vanadium oxytrifluoride (0.335 g, 2.7 mmol) and boron trifluoride−diethyl ether (0.27 g, 1.9 mmol), during which the solution turned dark purple. **13** (0.51 g, 1.2 mmol) was added dropwise over a period of 10 min, and the solution was stirred at room temperature for an additional 20 min. Workup was performed by adding 200 mL of water, extracting the mixture several times with ether until the water phase was clear, drying the organic phase with magnesium sulfate, and evaporating the solvent followed by short flash column chromatography (silica gel/CH_2Cl_2). Yield: 0.512 g (47%) of a white crystalline solid. ^{1}H-NMR ($CDCl_3$): δ 7.86 (s, 6H), 4.25 (t, 12H, $J = 6.3$ Hz), 3.64 (t, 2H, $J = 6.6$ Hz), 1.99 (q, 12H, $J = 6.9$ Hz), 1.58 (m, 12H), 1.31 (m, 76H), 0.91 (t, 15H, $J = 4.2$ Hz); ^{13}C-NMR ($CDCl_3$): δ 149.0, 123.6, 107.3, 69.7, 63.1, 32.9, 32.1, 29.8, 29.8, 29.7, 29.6, 29.5, 26.3, 25.9, 22.8, 14.3; IR (thin film on NaCl plate): 3103, 2928, 2859, 1617, 1518, 1468, 1389, 1340, 1264, 1171, 1054, 980, 894, 836, 757, 730, 666, 617, 602 cm^{-1}; Anal. Calcd for $C_{80}H_{136}O_7$: C, 79.41; H, 11.32. Found: C, 78.93; H, 10.82. HRMS (FAB) calcd for $C_{80}H_{136}O_7$ $(MH)^+$: 1209.9717. Found: 1209.9712.

Preparation of 11-((12-Hydroxydodecyl)oxy)-2,3,6,7,-10-pentapentoxytriphenylene (16). **16** was synthesized according to the procedure described above using **13** (0.7 g, 1.4 mmol) and **10** (0.7 g, 1.9 mmol) in dimethylformamide to give 0.38 g (32%) of a white crystalline solid. ^{1}H-NMR ($CDCl_3$): δ 7.82 (s, 6H), 4.22 (t, 12H, $J = 6.6$ Hz), 3.60 (t, 2H, $J = 6.3$ Hz), 1.94 (q, 12H, $J = 6.9$ Hz), 1.51 (m, 12H), 1.28 (m, 26H), 0.96 (t, 15H, $J = 7.2$ Hz); ^{13}C-NMR ($CDCl_3$): δ 148.8, 123.4, 107.2, 69.5, 62.8, 32.6, 32.3, 29.4, 29.3, 28.9, 28.1, 25.9, 25.5, 22.3, 13.8. IR (thin film on NaCl plate): 3104, 2925, 2857, 1617, 1518, 1468, 1453, 1389, 1341, 1263, 1172, 1054, 980, 894, 871, 836, 757, 730, 666, 616, 601 cm^{-1}. Anal. Calcd for $C_{55}H_{86}O_7$: C, 76.87; H, 10.09. Found: C, 75.99; H, 9.82. HRMS (FAB) calcd for $C_{55}H_{86}O_7$ $(MH)^+$: 858.6352. Found: 858.6366.

Preparation of 5-{[(((2,3,6,7,10-Pentakis(decyloxy)-triphenylen-11-yl)oxy)dodecyl)oxy]carbonyl}bicyclo[2.2.1]-hept-2-ene (17). Norborn-2-ene-5-carbonyl chloride (50 mg, 0.3 mmol) in 10 mL of dry tetrahydrofuran was added to a solution of **15** (190 mg, 0.16 mmol) and triethylamine (30 mg) in 50 mL of dry tetrahydrofuran. The resulting solution was stirred under argon under reflux for 16 h, during which a white solid formed. The mixture was filtered and dried over magnesium sulfate, and the solvent was removed *in vacuo*. Final purification was achieved by column chromatography (silica gel/CH_2Cl_2) to yield 0.205 g of a white crystalline solid (96%). ^{1}H-NMR ($CDCl_3$): δ 7.82 (s, 6H), 6.17 (d of d, 1H, $J = 3$ Hz), 5.90 (dd, 1H, $J = 3$ Hz), 4.21 (t, 12H, $J = 6.6$ Hz), 3.99 (m, 2H), 3.18 (m, 1H), 2.95−2.88 (m, 2H), 1.92 (m, 14H), 1.55 (m, 12H), 1.27 (m, 78H), 0.87 (t, 15H, $J = 5.1$ Hz). ^{13}C-NMR ($CDCl_3$): δ 148.8, 137.5, 132.2, 125.3, 123.4, 107.2, 69.5, 64.0, 49.4, 45.5, 43.1, 42.3, 32.7, 30.1, 29.5, 29.4, 29.3, 29.2, 29.1, 28.9, 28.5, 26.0, 25.7, 22.5, 13.9. IR (thin film on NaCl plate): 3044, 2922, 2852, 1842, 1738, 1617, 1517, 1468, 1435, 1388, 1262, 1174, 1072, 1048, 1033, 977, 835, 802, 722, 699, 602 cm^{-1}. Anal. Calcd for $C_{88}H_{144}O_8$: C, 75.52; H, 10.84, found: C, 75.41; H, 10.77. HRMS (FAB) calcd for $C_{88}H_{144}O_8$ $(MH)^+$: 1330.1241. Found: 1330.1238.

Preparation of 5-{[(((2,3,6,7,10-Pentapentoxytriphenylen-11-yl)oxy)dodecyl)oxy]carbonyl}bicyclo[2.2.1]hept-2-ene (18). **18** was synthesized according to the procedure described above using norborn-2-ene-5-carbonyl chloride (0.1 g, 0.64 mmol) and **16** (0.35 g, 0.41 mmol) to give 0.37 g (91%) of a white crystalline solid. ^{1}H-NMR ($CDCl_3$): δ 7.82 (s, 6H), 6.17 (d of d, 1H, $J = 3.1$ Hz), 5.91 (d of d, 1H, $J = 3.1$ Hz), 4.22 (t, 12H, $J = 6.3$ Hz), 3.99 (m, 2H), 3.19 (m, 1H), 2.95−2.88 (m, 2H), 1.94 (m, 14H), 1.55 (m, 12H), 1.29 (m, 28H), 0.97 (t, 15H, $J = 7.2$ Hz). ^{13}C-NMR ($CDCl_3$): δ 148.7, 137.5, 135.5, 132.1, 1223.4, 107.1, 69.4, 64.1, 49.4, 45.5, 43.1, 42.3, 31.4, 30.1, 29.5, 29.4, 29.3, 29.0, 28.9, 28.5, 28.2, 26.0, 25.7, 22.4, 13.9. IR (thin film on NaCl plate): 3048, 2934, 2857, 1842, 1733, 1619, 1510, 1468, 1430, 1389, 1262, 1174, 1071, 1033, 977, 938, 838, 774, 699, 605 cm^{-1}; Anal. Calcd for $C_{63}H_{94}O_8$: C, 77.26; H, 9.67. found: C, 76.81; H, 9.52. HRMS (FAB) calcd for $C_{63}H_{94}O_8$ $(MH)^+$: 978.4468. Found: 978.6919.

Preparation of ((2,3,6,7,10-Pentakis(decyloxy)triphenylen-11-yl)oxy)dodecyl 5-(2-Cyclobutenyl)pentanoate (19). 5-(2-Cyclobutenyl)pentanoic acid[69] (0.26 g, 1.8 mmol) was combined with thionyl chloride (0.35 g) in a 25 mL round bottom flask. This mixture was then heated to 45 °C for 1.5 h, at which point gas evolution had ceased. The solution was then concentrated *in vacuo*. The resulting orange oil was added over a period of 5 min to a solution of **15** (0.35 g, 0.3 mmol) and 1 mL of triethylamine in 50 mL of tetrahydrofuran. The reaction was stirred under argon at room temperature for 16 h, during which a white solid precipitated. After filtration the solvent was removed *in vacuo*. Final purification was achieved by column chromatography (silica gel/CH_2Cl_2) to yield 0.55 g of a white crystalline solid (99%). ^{1}H-NMR ($CDCl_3$): δ 7.82 (s, 6H), 6.04 (dd, 2H, $J_1 = 3.2$ Hz, $J_2 = 10.2$ Hz), 4.22 (t, 12H, $J = 6.3$ Hz), 4.04 (t, 2H, $J = 6.6$ Hz), 2.76 (m, 1H), 2.62 (dd, 1H, $J_1 = 4.0$ Hz, $J_2 = 13.8$ Hz), 2.29 (t, 2H, $J = 7.5$ Hz), 1.94 (m, 12H), 1.57−1.28 (m, 75H), 0.88 (t, 15H, $J = 6.6$ Hz). ^{13}C-NMR ($CDCl_3$): δ 148.9, 140.5, 134.9, 123.5, 107.5, 69.6, 64.1, 43.8, 36.6, 34.1, 34.0, 31.7, 31.3, 29.4, 29.3, 29.2, 29.1, 28.5, 27.3, 26.0, 25.7, 24.9, 22.4, 13.8. IR (thin film on NaCl plate) 3045, 2922, 2851, 1868, 1732, 1616, 1519, 1466, 1436, 1388, 1262, 1174, 1070, 1048, 977, 933, 836, 800, 774, 722, 699, 602 cm^{-1}; Anal. Calcd for $C_{89}H_{148}O_8$: C, 79.41; H, 10.99. Found: C, 79.39; H, 10.61. HRMS (FAB) calcd for $C_{89}H_{148}O_8$ $(MH)^+$: 1345.152. Found: 1345.117.

Preparation of ((2,3,6,7,10-Pentapentoxytriphenylen-11-yl)oxy)dodecyl 5-(2-Cyclobutenyl)pentanoate (20). **20** was synthesized in analogy to **19** using 3-oxa-4-(2-cyclobutene)-butyric acid[69] (0.26 g, 1.8 mmol) and **16** (0.5 g, 0.05 mmol) to yield 0.54 g of **20** as a colorless liquid (93%). ^{1}H-NMR ($CDCl_3$): δ 7.82 (s, 6H), 6.05 (dd, 2H, $J_1 = 3.2$ Hz, $J_2 = 10.4$ Hz), 4.22 (t, 12H, $J = 6.3$ Hz), 4.04 (t, 2H, $J = 6.6$ Hz), 2.75 (m, 1H), 2.62 (dd, 1H, $J_1 = 4.0$ Hz, $J_2 = 14.0$ Hz), 2.28 (t, 2H, $J = 7.5$ Hz), 1.94 (m, 12H), 1.56−1.25 (m, 45H), 0.97 (t, 15H, $J = 7.2$ Hz). ^{13}C-NMR ($CDCl_3$): δ 148.8, 140.6, 135.0, 123.4, 107.1, 69.5, 64.1, 43.7, 36.6, 34.1, 34.0, 31.4, 29.4, 29.3, 29.0, 28.9, 28.4, 28.2, 27.3, 26.0, 25.7, 25.0, 23.7, 22.3, 13.9; IR (thin film on NaCl plate): 3045, 2929, 2857, 1769, 1719, 1644, 1616, 1576, 1509, 1458, 1435, 1387, 1262, 1172, 1102, 1052, 837, 699 cm^{-1}. Anal. Calcd for $C_{64}H_{98}O_8$: C, 77.15; H, 9.92. Found: C, 77.02; H, 9.63. HRMS (FAB) calcd for $C_{64}H_{98}O_8$ $(MH)^+$: 994.7262. Found: 994.7281.

General Polymerization Procedure for the Norbornene Monomers 17 and 18. Under an inert atmosphere, the catalyst and the monomer were weighed into vials and dissolved in dichloromethane (~1 mL of CH_2Cl_2 for every 100 mg of monomer). The reaction was initiated by adding the catalyst solution to the vigorously stirred monomer solution. The reaction mixture was allowed to stir under argon for 6 h. It was terminated by adding a small amount of ethyl vinyl ether and poured into ~50 mL of methanol to precipitate the polymer. The resulting white polymer was purified by dissolving in dichloromethane and reprecipitating from methanol several times and drying *in vacuo*. Isolated yields ranged from 90 to 99%.

Polymer 21: ^{1}H-NMR ($CDCl_3$) δ 7.78 (bs, 6H), 5.31 (bm, 2H), 4.20 (bs, 12H), 3.99 (bs, 2H), 2.89−2.44 (bm, 3H), 1.91 (bm, 14H), 1.56−1.25 (bm, 90H), 0.85 (bs, 15H); ^{13}C-NMR

($CDCl_3$) δ 149.1, 134.4−131.9 (backbone C, *olefin*), 123.6, 107.4, 69.8, 29.8−26.1 (backbone C, *alkyl*), 29.2, 28.3; IR (thin film on NaCl plate) 2929, 2856, 1730, 1617, 1509, 1468, 1437, 1389, 1263, 1174, 1052, 971, 836, 722, 605 cm^{-1}. Anal. Calcd for $C_{88}H_{144}O_8$: C, 75.52; H, 10.84. Found: C, 76.02; H, 10.52.

Polymer 22: ^{1}H-NMR ($CDCl_3$) δ 7.78 (bs, 6H), 5.31 (bm, 2H), 4.19 (bs, 12H), 3.99 (bs, 2H), 2.87−2.46 (bm, 3H), 1.91 (bm, 14H), 1.57−1.26 (bm, 12H), 0.94 (bd, 28H, $J = 5.1$). ^{13}C-NMR ($CDCl_3$) δ 149.0, 134.5−132.1 (backbone C, *olefin*), 123.6, 107.3, 69.7, 29.8−26.0 (backbone C, *alkyl*), 29.2, 28.5; IR (thin film on NaCl plate) 2930, 2857, 1730, 1617, 1510, 1468, 1436, 1389, 1263, 1171, 1052, 971, 836, 730, 600 cm^{-1}. Anal. Calcd for $C_{63}H_{94}O_8$: C, 77.26; H, 9.67. Found: C, 77.01; H, 9.61.

General Polymerization Procedure for the Cyclobutene Monomers 19 and 20. Under an inert atmosphere, the catalyst and the monomer were weighed into vials and dissolved in CH_2Cl_2 such that the overall [M] was approximately 0.3 M. The reaction was initiated by adding the catalyst solution to the vigorously stirred monomer solution. The reaction mixture was allowed to stir under argon for 2 h at room temperature. The reaction mixture was poured into methanol (~150 mL) containing 1 N HCl (5% v/v) to precipitate the polymer and left to stir for 30 min. The polymer was redissolved in CH_2Cl_2 and reprecipitated into methanol (~150 mL). The polymer was then isolated by filtration and dried *in vacuo*. Isolated yields ranged from 82 to 98%.

Polymer 23: ^{1}H-NMR ($CDCl_3$) δ 7.78 (s, 6H), 5.36−5.11 (bm, 2H), 4.23 (bs, 12H), 4.01 (bs, 2H), 3.44 (bs, 1H), 2.80−2.60 (bm, 2H), 2.25 (bm, 4H), 1.90 (bs, 14H), 1.57−1.29 (bm, 90H), 0.89 (bt, 15H, $J = 6.7$); ^{13}C-NMR ($CDCl_3$) δ 149.0, 134.0−126.0 (backbone C, *olefin*), 123.6, 107.3, 69.7, 64.5, 34.4−24.6 (backbone C, *alkyl*), 32.0, 29.8, 29.7, 29.6, 29.5, 29.4, 28.8, 26.3, 22.8, 14.2; IR (thin film on NaCl plate) 2922, 2852, 1739, 1617, 1518, 1468, 1436, 1388, 1262, 1174, 1071, 1033, 836, 802, 722, 602 cm^{-1}. Anal. Calcd for $C_{89}H_{148}O_8$: C, 79.41; H, 9.99. Found: C, 79.33; H, 10.82.

Polymer 24: ^{1}H-NMR ($CDCl_3$) δ 7.80 (bs, 6H), 5.30−5.10 (bm, 2H), 4.20 (bs, 12H), 4.01 (bs, 2H), 3.46 (bs, 1H), 2.90−2.60 (bm, 2H), 2.25 (bm, 4H), 1.92 (bs, 14H), 1.56−1.27 (bm, 50H), 0.95 (bt, 15H, $J = 6.9$); ^{13}C-NMR ($CDCl_3$) δ 148.9, 134.7−127.8 (backbone C, *olefin*), 123.6, 107.6, 69.6, 64.1, 42.5−24.8 (backbone C, *alkyl*), 34.1, 29.4, 29.1, 29.0, 28.5, 26.0, 25.7, 22.3, 13.7; IR (thin film on NaCl plate) 2926, 2854, 1739, 1618, 1518, 1468, 1436, 1388, 1262, 1174, 1072, 1033, 836, 803, 722, 601 cm^{-1}. Anal. Calcd for $C_{64}H_{98}O_8$: C, 77.15; H, 9.92. Found: C, 77.26; H, 10.04.

General procedure for the Hydrogenation of Polymers 23 and 24. Polymer **23** or **24** was dissolved in dichloromethane followed by the addition of a catalytic amount of Crabtree catalyst $[Ir(COD)(Cy_3)(py)]PF_6$. The flask was placed in a hydrogenation bomb and heated at 55 °C for 16 h under 120 psi of hydrogen pressure. The polymers were isolated by repeated precipitation from methanol.

Polymer 25: ^{1}H-NMR ($CDCl_3$) δ 7.80 (bs, 6H), 4.20 (bs, 12H), 4.03 (bs, 2H), 2.27 (bm, 4H), 1.92 (bs, 14H), 1.57−1.28 (bm, 95H), 0.89 (bt, 15H); ^{13}C-NMR ($CDCl_3$) δ 149.0, 123.6, 107.3, 69.7, 64.5, 34.5−26.1 (backbone C, *alkyl*), 32.0, 29.8, 29.7, 29.6, 29.5, 29.4, 26.3, 14.2; IR (thin film on NaCl plate) 2923, 2852, 1738, 1617, 1518, 1467, 1438, 1388, 1263, 1174, 1071, 1048, 836, 801, 722, 602 cm^{-1}. Anal. Calcd for $C_{89}H_{150}O_8$: C, 79.53; H, 11.14. Found: C, 79.12; H, 11.30.

Polymer 26: ^{1}H-NMR ($CDCl_3$) δ 7.79 (bs, 6H), 4.20 (bs, 12H), 4.02 (bs, 2H), 2.25 (bm, 4H), 1.92 (bs, 14H), 1.54−1.18 (bm, 55H), 0.95 (bt, 15H, $J = 6.9$); ^{13}C-NMR ($CDCl_3$) δ 148.9, 123.6, 107.2, 69.7, 64.5, 51.0, 38.2−25.5 (backbone C, *alkyl*), 34.5, 29.8, 29.7, 29.6, 29.5, 29.2, 28.8, 28.5, 26.3, 26.0, 22.7, 14.2; IR (thin film on NaCl plate) 2923, 2852, 1738, 1617, 1518, 1467, 1436, 1387, 1262, 1173, 1071, 1033, 836, 802, 722, 602 cm^{-1}. Anal. Calcd for $C_{64}H_{100}O_8$: C, 77.11; H, 10.04. Found: C, 76.87; H, 10.03.

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Supporting Information Available: Experimental procedures for the synthesis of compounds **4**−**14** are available (6 pages). Ordering information is given on any current masthead page.

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