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Synthesis of functionalized triphenylenes and dibenzopyrenes Precursor molecules for polymeric discotic liquid crystals

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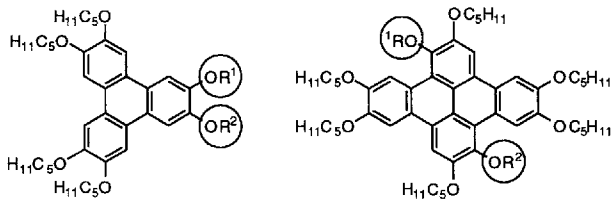
Discotic liquid crystals represent a promising class of new materials, for example, with respect to their photoconductivity properties. To tailor the processability and mesophase behaviour of such materials, specifically functionalized cores are required as precursor molecules for discotic oligomers, polymers and networks. The paper presents a simple synthetic strategy leading to unsymmetrically functionalized triphenylene and dibenzopyrene derivatives. Furthermore new symmetrical discotic octa-alkoxy-substituted dibenzopyrenes have been synthesized applying this route.



1. Introduction

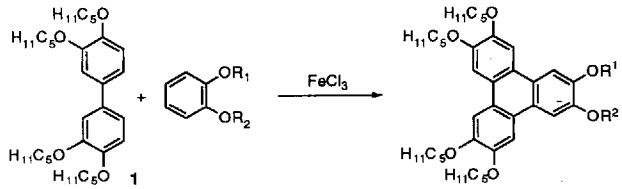
Discotic triphenylenes have been shown to be a new class of fast photoconducting materials. Charge carrier mobilities of $1 \times 10^{-3}\,cm^2\,V^{-1}\,s^{-1}$ (hole mobility) have been determined for hexapentyloxytriphenylene in the hexagonal columnar mesophase (D_h), exceeding the values for commonly used photoconducting polymers by two to three orders of magnitude [1, 2]. To explore further these intriguing features and move towards the aim of processable materials, discotic oligomers, polymers and networks are required. Such materials provide the possibility of studying charge transport phenomena at room temperature and in the solid state, thus elucidating the influence of mobility and defects of the columnar mesophase.

A prerequisite to tailor the properties of such materials is the synthesis of unsymmetrically substituted discotics (for example, triphenylenes and dibenzopyrenes) as precursor molecules for polymeric discotics. So far, the functionalization of triphenylenes has been achieved either by sophisticated synthetic routes [3] or statistical methods involving the partial alkylation of hexa-acetoxy-triphenylene [4] or partial ether cleavage of hexa-alkoxytriphenylenes [5]. However, the resulting mixture of polysubstituted triphenylenes results in tedious separation steps and limits the exploitation of these methods to rather low amounts of material.

To overcome this problem it was necessary to develop a procedure which basically allows the preparation of any specifically functionalized hexa-alkoxysubstituted triphenylene on a large scale.

2. Results and discussion

Symmetrically substituted triphenylenes can be prepared by oxidative trimerization of *o*-dialkoxybenzenes either with chloranil [6] or iron (III) chloride [7–9].



2: $R^1 = R^2 = CH_3$ (80 per cent): C 80 I
3: $R^1 = C_5H_{11}$, $R^2 = CH_3$ (76 per cent): C_1 65 C_2 72 D_h 101 I
4: $R^1 = C_5H_{11}$, $R^2 = (CH_2)_3OH$ (69 per cent): C_1 43 C_2 55 D_h 106 I

Figure 1. Synthesis of functionalized hexa-alkoxytriphenylenes via the biphenyl route (C: crystalline; D_h: discotic hexagonal columnar mesophase; I: isotropic state).

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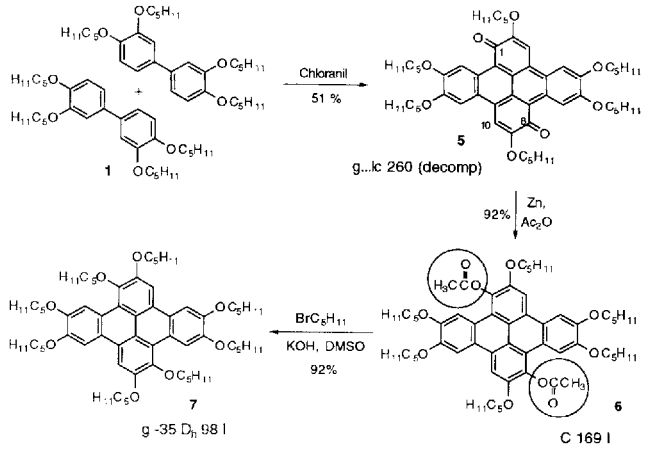
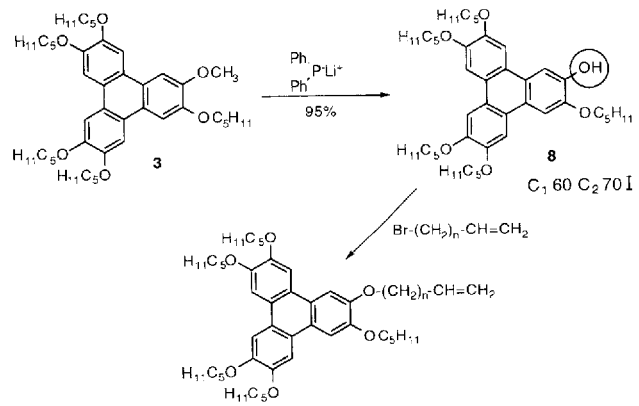


Figure 2. Synthesis of octa-alkoxy-substituted dibenzopyrenes via a difunctionalized precursor molecule (g: glassy state; LC: liquid crystalline; D_h: discotic hexagonal columnar mesophase; I: isotropic state).

The oxidation of a mixture of tetramethoxybiphenyl and veratrole results in an efficient incorporation of the former into the hexamethoxytriphenylene formed, indicating that the trimerization involves three consecutive Scholl reactions involving biphenyl and terphenyl intermediates [10, 11]. Thus the strategy of our synthesis is based on the possibility of preparing highly functionalized triphenylene derivatives by the coupling of tetra-alkoxybiphenyls with *o*-dialkoxybenzenes (see figure 1). Detailed synthetic procedures are given in the experimental part. Bushby *et al.*, have recently described an approach very similar to the one we are pursuing [12]. Additional possibilities for this tailored synthesis of triphenylenes are based on the fact that functionalities like hydroxy-groups can be further derivatized to yield discotic monomers (for example, acrylates or vinyl ethers). Thus by applying functionalized tetra-alkoxybiphenyls and/or *o*-dialkoxybenzenes, various substitution patterns for hexa-alkoxy-substituted triphenylenes may be envisaged.

Similar conditions can be used for the synthesis of discotic dibenzopyrene derivatives—aromatic polycycles with an extended Π-system. As shown in figure 2, the tetrapentyloxybiphenyl **1** can by itself be dimerized to give the hitherto unknown liquid crystalline polycyclic quinone **5**. As demonstrated by Musgrave and Webster for the tetramethoxybiphenyl, this procedure involves repeated Scholl reactions, followed by oxidative dealkylation [13, 14]. Further derivatization of **5** by reduction to the dibenzopyrene diacetate **6** furnishes a precursor molecule which can be modified simply to yield the first discotic octa-alkoxy-substituted dibenzopyrene **7**. According to procedures known for corresponding triphenylenes, the diacetate **6** can also be used to prepare discotic main chain polymers [15] or further converted into monofunctional monomers for the synthesis of discotic side group polymers [4, 16]. These octa-alkoxydibenzopyrenes represent an additional class of electron rich polycyclic discotics. The octapentyloxydibenzopyrene **7** turns out to be an interesting photoconductive system, since it exhibits a columnar mesophase at room temperature and does not crystallize [17]. The homologous series of octa-alkoxydibenzopyrenes [17] is easily accessible by etherification of octa-acetoxydipenzopyrene, which has been obtained by modifying the procedure of Musgrave *et al.* [14].

The combination of the coupling reactions described above with a selective cleavage of methoxy groups next to alkoxy groups [18] extends the versatility of the procedure and provides additional possibilities for the synthesis of unsymmetrically substituted discotics. In this context, the synthesis of monohydroxypentaalkoxytriphenylene **8** is of special interest, since it represents a central precursor molecule for monofunctional discotic monomers. Selective cleavage of the methoxy group of **3** with lithium diphenylphosphide is an efficient route to get **8** in high yield (see figure 3). Alkylation of **8** with functionalized ω-bromoalkanes allows the tailoring of the mesophase behaviour of discotic monomers and their corresponding oligomers or polymers. As an example, the discotic triphenylenes **9**, **10** and **11** carrying one terminal olefinic group were prepared from **8**. They can be linked via hydrosilylation to tetramethylcyclosiloxane providing discotic oligomers as glassy model compounds for photoconductivity studies [17]. Furthermore it is possible not only to remove selectively the methyl group (see figure 3), but also to do it successively, starting from the dimethoxy derivative **2**, in such a way that each methyl



9; $n = 2$ (90 per cent): C 55·5 D_h 126·5 I
10: $n = 4$ (87 per cent): C 60 D_h 114 I
11; $n = 6$ (81 per cent); C 51 D_h 100 I

Figure 3. Synthesis of discotic monomers via monohydroxypentapentyloxytriphenylene as precursor molecule (C: crystalline; D_h: discotic hexagonal columnar mesophase; I: isotropic state).

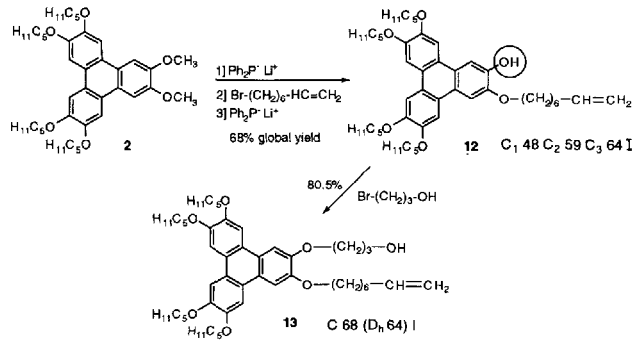


Figure 4. Synthesis of discotic triphenylenes carrying different functional groups (C: crystalline; D_h: discotic hexagonal columnar mesophase; I: isotropic state).

group can be replaced by a specific and different function as shown for **12** and **13** (see figure 4).

All the procedures described in this paper proceed in high yields and thus open possibilities for the synthesis of a variety of unsymmetrically functionalized triphenylene and dibenzopyrene derivatives interesting as new materials, for example, for molecular electronics [19].

3. Experimental

Phase transitions have been determined using a DSC 7 (Perkin–Elmer) with heating and cooling rates of $10\,K\,min^{-1}$. The phase type was characterized using polarizing microscopy and X-ray investigations. A detailed discussion of the phase behaviour of the synthesized compounds, in particular of the mesogenic dibenzopyrenes, will be presented in a forthcoming publication. This will also include the detailed description of the intracolumnar correlation length to characterize the extent of order within the columns; at present, the notation D_h will be used, referring to a hexagonal columnar mesophase.

Synthesis of **1**: 1,2-dipentyloxybenzene [8] is iodinated to give 1-iodo-3,4-dipentyloxybenzene (88 per cent) as described for veratrole [20], and subsequent Ullman-coupling [21] yields the 3,4,3′,4′-tetrapentyloxybiphenyl **1** (82 per cent, m.p. 84°C); ^{1}H NMR (200 MHz, $CDCl_3$: δ (ppm) = 7·07–6·89 (m, 6 H, Ar–H), 4·05, 4·02 (2 × t, 8 H, $OC\underline{H}_2$, J = 6·8, 6·7 Hz), 1·93–1·78 (m, 8 H, $OCH_2C\underline{H}_2$), 1·50–1·38 (m, 16 H, $OCH_2CH_2(C\underline{H}_2)_2$); 0·93 (t, 12 H, $C\underline{H}_3$, J = 6·8 Hz); FD-MS: m/z (per cent) 498 (100) $[M]^{\bullet+}$.

Synthesis of **2**: 3·14 g (19·3 mmol) or iron(III) chloride is added to a solution of 1 g (2 mmol) of **1** and 0·83 g (6 mmol) of veratrole in 10 ml of dichloromethane and three drops of concentrated sulphuric acid. The mixture is heated for 1·5 h at 50°C. Work-up is performed by addition of 2 ml of methanol before the mixture is subjected to a short flash chromatography on silica gel (CH_2Cl_2/PE 4 : 1). Recrystallization from acetonitrile affords 1·02 g of **2** (80 per cent); ^{1}H NMR (400 MHz, $CDCl_3$: δ (ppm) = 7·83, 7·78 (2 × s, 4 H, 2 H, Ar–H), 4·22 (t, 8 H, $OC\underline{H}_2$, J = 6·6 Hz), 4·10 (s, 6 H, $OC\underline{H}_3$), 1·97–1·90 (m, 8 H, $OCH_2C\underline{H}_2$), 1·58–1·41 (m, 16 H, $OCH_2CH_2(C\underline{H}_2)_2$), 0·96, 0·95 (2 × t, 12 H, $CH_2C\underline{H}_3$, J = 7·3 Hz); FD-MS: m/z (per cent) 633 (100) $[M]^{\bullet+}$.

Synthesis of **3**: 9·41 g (589 mmol) of iron(III) chloride, 3 g (6·02 mmol) of **1** and 3·51 g (18·5 mmol) of 1-pentyloxy-2-methoxybenzene are subjected to the procedure described for **2**. After flash chromatography on silica gel (CH_2Cl_2/PE 1 : 1), 3·15 g of **3** (76 per cent) are isolated after recrystallization from ethanol; ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) = 7·83–7·79 (m, 6 H, Ar–H), 4·25–4·19 (m, 10 H, $OC\underline{H}_2$), 4·09 (s, 3 H, $OC\underline{H}_3$), 1·98–1·91 (m, 10 H, $OCH_2C\underline{H}_2$), 1·59–1·39 (m, 20 H, $OCH_2CH_2(C\underline{H}_2)_2$), 0·96 (t, 15 H, $CH_2C\underline{H}_3$, J = 7·0 Hz); FD-MS: m/z (per cent) 688 (100) $[M]^{\bullet+}$.

Synthesis of **4**: 9·41 g (58 mmol) of iron(III) chloride, 3 g (6·02 mmol) of **1** and 3·51 g (18·5 mmol) of 1-(3-hydroxypropyloxy)-2-pentyloxybenzene are subjected to the procedure described for **2**. Following the flash chromatography (CH_2Cl_2/MeOH 300 : 1), 3·1 g of **4** (69 per cent) are isolated after recrystallization from ethanol and petroleum ether; ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) = 7·83–7·79 (m, 6 H, Ar–H), 4·43 (t, 2 H, $OC\underline{H}_2CH_2CH_2OH$, J = 5·6 Hz), 4·26 (t, 10 H, $OC\underline{H}_2$, J = 6·4 Hz), 3·99–3·96 (m, 2 H, $OCH_2CH_2C\underline{H}_2OH$), 2·96 (s, 1 H, $O\underline{H}$), 2·27–2·15 (m, 2 H, $OCH_2C\underline{H}_2CH_2OH$), 2·01–1·88 (m, 10 H, $OCH_2C\underline{H}_2$), 1·60–1·39 (m, 20 H, $OCH_2CH_2(C\underline{H}_2)_2$), 0·96 (t, 15 H, $C\underline{H}_3$, J = 7·1 Hz); FD-MS: m/z (per cent) 732 (100) $[M]^{\bullet+}$.

Synthesis of **5**: 4 g (8·02 mmol) of **1** and 7·89 g (32·08 mmol) of chloranil in a mixture of 25 ml of acetic acid and 25 ml of concentrated sulphuric acid are heated to 60°C for 24 h while stirring under argon. The mixture is poured into water and filtered. The solid is extracted into dichloromethane which is washed with sodium bicarbonate solution and water. Evaporation of the solvent and subsequent short flash chromatography on silica gel (CH_2Cl_2/PE 2 : 1) provides 2·4 g (71 per cent) of a purple solid which is a mixture of two quinones with the quinonoid groups in the 1,8 positions (see figure 2) (**5**, CH_2Cl_2, R_f = 0·53) and the 1,10 positions (CH_2Cl_2, R_f = 0·61). From ^{1}H NMR, a ratio of 3 : 1 favouring the desired product **5** has been determined. Further repeated chromatography (CH_2Cl_2) provides 1·71 g (51 per cent) of **5**. It should be noted that the conditions applied for the synthesis of the triphenylene derivatives **2**, **3** and **4** ($FeCl_3$, CH_2Cl_2) have also been used for the dimerisation of **1**; however, the major product formed is not **5**, but the 1,10-quinone which can also be used as a precursor molecule for further functionalization to discotic dibenzopyrenes. **5**: ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) = 9·37 (s, 2 H, $H_{quin.}$), 6·86, 6·71 (2 × s, 4 H, Ar–H), 4·25–4·01 (m, 12 H, $OC\underline{H}_2$), 2·0–1·89 (m, 12 H, $OCH_2C\underline{H}_2$), 1·58–

1·40 (m, 24 H, $OCH_2CH_2(C\underline{H}_2)_2$) 1·03–10·94 (m, 18 H, $C\underline{H}_3$); FD-MS: *m/z* (per cent) 849 (100) $[M]^{\bullet+}$.

Synthesis of **6**: 1 g (1·18 mmol) of **5**, 1 g of zinc dust, 0·5 ml of triethylamine and 30 ml of acetic anhydride are heated under reflux for 15 min under argon. After cooling to room temperature, the mixture is poured into water and filtered. The solid is dissolved in 1 ml of dichloromethane and precipitated in 100 ml of methanol, affording 1·01 g of **6** as a white powder (92 per cent); ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) = 8·64, 8·1, 7·94 (3 × s, 6 H, Ar–H), 4·30–4·17 (m, 12 H, $OC\underline{H}_2$), 2·47 (s, 6 H, $OCOC\underline{H}_3$), 2·03–1·86 (m, 12 H, $OCH_2C\underline{H}_2$), 1·59–1·32 (m, 24 H, $OCH_2CH_2(C\underline{H}_2)_2$), 1·03 0·96 (m, 18 H, $C\underline{H}_3$); FD-MS: *m/z* (per cent) 935 (100) $[M]^{\bullet+}$.

Synthesis of **7**: 200 mg (0·21 mmol) of **6**, 0·5 ml of 1-bromopentane and 100 mg of KOH in 10 ml of dry DMSO are stirred at 60°C for 2 h under argon. The mixture is poured into 100 ml of water to precipitate **7**. After filtering, the solid is washed with methanol and recrystallized from ethyl acetate/methanol (1 : 1) affording 195 mg (92 per cent) of white product **7**; ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) = 9·24, 8·07, 7·95 (3 × s, 2 H, Ar–H), 4·33–4·20 (m, 12 H, $OC\underline{H}_2$) 3·95 (t, 4 H, $OC\underline{H}_2$, J = 6·9 Hz), 2·0–1·84 (m, 16 H,)$CH_2C\underline{H}_2$, 1·61–1·37 (m, 32 H, $OCH_2CH_2(C\underline{H}_2)_2$), 1·03–0·87 (m, 24 H, $C\underline{H}_3$); FD-MS: *m/z* (per cent) 991 (100) $[M]^{\bullet+}$; Elemental analysis (per cent) [found (calculated)]: C [77·53 (77·32)]; H [9·56 (9·63)].

Synthesis of **8**: The general procedure followed a previously described method [18]. Starting with 516 mg (0·75 mmol) of 3 and 1·5 eq. of BuLi and diphenylphosphine, the cleavage is achieved by boiling the mixture until complete reaction is achieved (~2 h). The reaction is monitored by TLC (aluminium oxide 60 F_{254} E, CH_2Cl_2/Hex.: 3/2, R_f(**3**) = 0·35, R_f(**8**) = 0·05). The purification by silica gel chromatography (CH_2Cl_2/Hex.: 3/2) yields 480 mg (95 per cent) of **8** as a white powder (m.p. 70°C); ^{1}H NMR (200 MHz, $CDCl_3$: δ (ppm) = 7·94–7·75 (m, 6 H, Ar–H), 5·90 (s, 1 H, O$\underline{H}$), 4·30, 4·16 (m, 10 H, $OC\underline{H}_2$), 1·97–1·87 (m, 10 H, $OCH_2C\underline{H}_2$), 1·567–1·35 (m, 20 H, $OCH_2CH_2(C\underline{H}_2)_2$), 0·96 (t, 15 H, CH_3, J = 7·0 Hz); FD-MS: *m/z* (per cent) 674 (100) $[M]^{\bullet+}$.

Synthesis of **9**: The procedure used was that described for **7** (DMF instead of DMSO), starting from 4 g (5·93 mmol) of **8**, 1·22 ml (11·9 mmol) of 4-bromobut-1-ene and 1 g (17·86 mmol) of KOH. Recrystallization from ethanol affords 3·89 g of **9** as a white solid (90 per cent); ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) = 7·96, 7·91 (2 × s, 6 H, Ar–H), 6·02 (tdd, 1 H, C$\underline{H}$=CH_2, J_{cis} = 10·2 Hz, J_{trans} = 17·1 Hz, J_{CH_2} = 6·8 Hz), 5·25 (d, 1 H, *trans* CH=C$\underline{H}_2$, J = 17·2 Hz), 5·17 (d, 1 H, *cis* CH=C$\underline{H}_2$, J = 10·3 Hz), 4·28, 4·23 (2 × t, 12 H, $OC\underline{H}_2$, J = 6·8, 6·5 Hz), 2·71 (td, 2 H, $C\underline{H}_2CH$=CH_2, J = 6·7, 6·7 Hz), 2·02–1·88 (m, 10 H, $OCH_2C\underline{H}_2$) 1·63–1·39 (m, 20 H, $OCH_2CH_2(C\underline{H}_2)_2$) 1·01–0·91 (m, 10 H, $CH_2C\underline{H}_3$); FD-MS: *m/z* (per cent) 728 (100) $[M]^{\bullet+}$.

10 and **11** have been obtained by a similar procedure in comparable yields (**10**: 87 per cent and **11**: 81 per cent). NMR and mass spectroscopy data are consistent with the proposed structure. **11**: Elemental analysis (per cent) [found (calculated)]: C [77·85 (78·02)]; H [9·69 (9·76)].

Synthesis of **12**: The first ether cleavage was carried out by the procedure described for **8**, starting with 264 mg (0·42 mmol) of **2** and 1·4 eq. of BuLi and diphenylphosphine. The purification carried out by silica gel chromatography (CH_2Cl_2/Hex.: 3/2) yields 237 mg 92 per cent) of the compound as a white powder (m.p. 116°C); ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) = 7·93, 7·81, 7·76 (3 × s, 6 H, Ar–H), 5·86 (broad s, 1 H, OH), 4·21, 4·19 (2 × t, 8 H, $OC\underline{H}_2$, J = 6·6 Hz), 4·11 (s, 3 H, OCH_3), 2·05–1·85 (m, 8 H, $OCH_2C\underline{H}_2$), 1·65–1·35 (m, 16 H, $OCH_2CH_2(C\underline{H}_2)_2$), 0·95 (t, 12 H, $CH_2C\underline{H}_3$, J = 7·0 Hz); MS-E.I.: *m/z* (per cent) 619 (100) $[M + 1]^+$.

Alkylation was carried out following the previously described procedure [22] starting from 440 g (0·71 mmol) and 2 eq. of the bromoalkane. The purification by silica gel chromatography (CH_2Cl_2/Hex.: 3/2) yields 442 mg (85 per cent) of the intermediate olefin as a yellow powder (m.p. 60°C); ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) = 7·82, 7·80, 7·78 (3 × s, 6 H, Ar–H), 5·82 (tdd, 1 H, C$\underline{H}$=CH_2, J_{cis} = 10·2 Hz, J_{trans} = 17·0 Hz, J_{CH_2} = 6·5 Hz), 5·01 (d, 1 H, *trans* CH=C$\underline{H}_2$, J = 17·0 Hz), 4·93 (d, 1 H, *cis* CH=C$\underline{H}_2$, J = 10·2 Hz), 4·23 (t, 10 H, OCH_2, J = 6·5 Hz), 4·09 (s, 3 H, OCH_3), 2·15–1·85 (m, 12 H, $OCH_2C\underline{H}_2$, $C\underline{H}_2CH$=CH_2), 1·65–1·35 (m, 22 H, $OCH_2CH_2(C\underline{H}_2)_2$), $OCH_2CH_2(C\underline{H}_2)_3$), 0·97 (t, 12 H, $CH_2C\underline{H}_3$, J = 7·0 Hz); MS-E.I.: *m/z* (per cent) 728 (100) $[M]^{\bullet+}$; Elemental analysis (per cent) [found (calculated)]: C [77·24 (77·43)]; H [9·50 (9·40)].

The final ether cleavage was made by the procedure described for **8**, starting with 370 mg (0·508 mmol) of the intermediate olefin. The purification by chromatography (CH_2Cl_2/Hex.: 3/2) yields 315 mg (87 per cent) of **12** as a yellow powder; ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) = 7·97, 7·82, 7·78 (3 × s, 6 H, Ar–H), 5·84 (tdd + broad s, 1·5 H, C$\underline{H}$=CH_2, OH, J_{cis} = 9·5 Hz, J_{trans} = 15·5 Hz, J_{CH_2} = 7·0 Hz), 5·02 (d, 1 H *trans* CH=C$\underline{H}_2$, J = 15·5 Hz), 4·96 (d, 1 H, *cis* CH=C$\underline{H}_2$, J = 9·5 Hz), 4·35–4·15 (m, 10 H, OCH_2), 2·20–1·85 (m, 12 H, $OCH_2C\underline{H}_2$, $C\underline{H}_2CH$=CH_2), 1·65–1·35 (m, 22 H, $OCH_2CH_2(C\underline{H}_2)_2$, $OCH_2CH_2(C\underline{H}_2)_3$), 0·99 (t, 12 H, $CH_2C\underline{H}_3$, J = 7·0 Hz); IR (KBr) 3550 cm^{-1} (sharp) [free OH st.] and 3300–3600 cm^{-1} (broad) [bonded OH st.]; MS-E.I.: *m/z* (per cent) 714 (100) $[M]^{\bullet+}$.

Synthesis of **13**: Following the previously described procedure [21], starting from 340 mg (0·48 mmol) of **12** and 1·5 eq. of the bromoalkane. The purification by silica gel chromatography (CH_2Cl_2) yields 298 mg (80·5 per

cent) of **13** as a white powder; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7·82, 7·80, 7·78 (3 × s, 6 H, Ar–H), 5·81 (tdd, 1 H, CH=CH_2, J_{cis} = 10·3 Hz, J_{trans} = 17·1 Hz, J_{CH_2} = 6·6 Hz), 4·99 (d, 1 H, *trans* CH=CH_2, J = 17·1 Hz), 4·93 (d, 1 H, *cis* CH=CH_2, J = 10·1 Hz), 4·42 (t, 2 H, O$CH_2CH_2CH_2$OH, J = 5·7 Hz), 4·22, 4·21 (2 × t, 10 H, O$CH_2(CH_2)_n$ (n = 3 or 5), J = 6·4, 6·4 Hz), 3·96 (t, 2 H, CH_2OH, J = 5·2 Hz), 3·05–2·80 (s broad, 0·6 H, OH), 2·18 (tt, 2 H, $OCH_2CH_2CH_2$OH, J = 5·3, 5·5 Hz), 2·12–2·04 (m, 2 H, CH_2CH=CH_2), 1·98–1·88 (m, 10 H, $OCH_2CH_2(CH_2)_n$ (n = 2 or 4), 1·60–1·49, 1·49–1·38 (2 × m, 10, 12 H, $O(CH_2O_2(CH_2)_n$ (n = 2 or 3), 0·96 (t, 12 H, CH_3, J = 7·3 Hz); FD-MS: m/z (per cent) 773 (100) $[M]^{\bullet+}$.

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