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A Convenient Route to 2-(Hydroxy)-3,6,7,10,11pentaalkyloxytriphenylenes

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A CONVENIENT ROUTE TO 2-(HYDROXY)-3,6,7,10,11-PENTA-ALKYLOXYTRIPHENYLENES

Graeme Cooke,* Franck Hell and Sébastien Violini

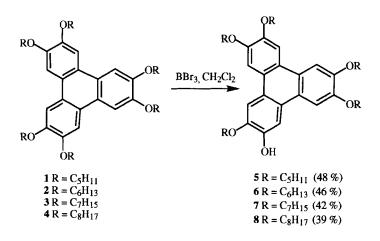
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Abstract: Unsymmetrical monohydroxypentaalkoxytriphenylenes have been conveniently prepared from the readily accessible symmetrical hexa-alkyloxytriphenylenes using boron tribromide mediated ether cleavage.

Arguably the most important class of discotic liquid crystals are those based upon hexa-alkyloxytriphenylenes. It has been proposed that these fascinating discogens may find commercial application as quasi-one-dimensional conductors¹ and photoconducting devices.² However, a major obstacle to their development is the lack of convenient procedures for synthesising suitably functionalised unsymmetrical building blocks. Monohydroxypentaalkoxytriphenylenes are particularly attractive synthetic targets and have been synthesised by selective ether cleavage of both symmetrical³ or unsymmetrical⁴ hexa-alkyloxytriphenylenes. However, these routes suffer from the lack of commercially available deprotection agents and the obvious drawbacks associated with the synthesis of unsymmetrical derivatives using sophisticated synthetic routes.⁴

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Here we describe a convenient method for producing monohydroxypentaalkyloxytriphenylenes in moderate yields from the readily accessible symmetrical hexa-alkyloxytriphenylenes. The synthesis of derivatives (5) - (8) were accomplished by reacting the parent triphenylenes (1) - (4) in high dilution (CH₂Cl₂) with one equivalent of BBr₃ for 5 hours at 25°C (Scheme 1). The pure unsymmetrical derivatives were isolated in moderate yield from the unreacted starting materials using chromatographic separation.⁵



Scheme 1

In conclusion, we have established a general and convenient one-pot synthesis of mono-hydroxypentalkyloxytriphenylenes from readily accessible symmetrical precursors and deprotection agents.

EXPERIMENTAL.

Melting points were recorded on a kofler hot-stage microscope apparatus and are uncorrected ¹H NMR spectra were measured at 90 MHz using a JEOL FX 90 Q

spectrometer. FAB mass spectra (LSIMS) were performed at the University of Wales, Swansea. Satisfactory analytical data were obtained for derivatives **4** - **8**.

General procedure for producing monohydroxypenta-alkyloxy triphenylenes:

Synthesis of 2-(hydroxy)-3,6,7,10,11-pentapentyloxytriphenylene (5).6

To a stirred solution of (5) (0.5 g, 0.67 mmol) in anhydrous CH₂Cl₂ (150 mL) at 25°C was added BBr₃ (0.67 mL of a 1.0 M solution in CH₂Cl₂) dropwise over 2 h. The resultant green solution was stirred for 5 h and then water (150 mL) was added. The organic layer was separated, dried (MgSO₄), filtered and evaporated under reduced pressure to afford a crude product which upon trituration with methanol afforded a white powder. Purification was achieved using column chromatography (silica gel, toluene as eluent) to afford (5) as a white powder; yield mpt 69 - 71 °C (lit.⁸ 70 °C). Calculated for C43H62O6: C, 76.56; H, 9.20; N, 0.00

found: 76.74 9.00 0.00 ¹H NMR (CDC1₃ / TMS): $\delta = 7.90 - 7.70$ (m, 6H), 5.90 (s, 1H), 4.30 - 4.10 (m, 10 H), 2.00 - 1.90 (m, 10H), 1.58 - 1.35 (m, 20H), 0.97 (t, 15H) MS (FAB): m/z = 674 (M⁺)

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- 5 Satisfactory analytical data were obtained for all compounds.
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