Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

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

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To cite this article: Graeme Cooke , Franck Hell & Sébastien Violini (1997) A Convenient Route to 2-(Hydroxy)-3,6,7,10,11-pentaalkyloxytriphenylenes, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 27:21, 3745-3748, DOI: 10.1080/00397919708007297

To link to this article: http://dx.doi.org/10.1080/00397919708007297

SYNTHETIC COMMUNICATIONS, 27(21), 3745-3748 (1997)

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

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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 conductors[1] and photoconducting devices.[2] However, a major obstacle to their development is the lack of convenient procedures for synthesising suitably functionalised unsymmetrical building blocks. Monohydroxypenta-alkoxytriphenylenes are particularly attractive synthetic targets and have been synthesised by selective ether cleavage of both symmetrical[3] or unsymmetrical[4] 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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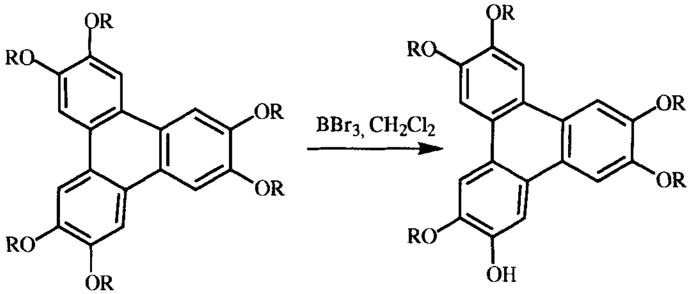
Here we describe a convenient method for producing mono-hydroxypentaalkyloxytriphenylenes in moderate yields from the readily accessible symmetrical hexa-alkyloxytriphenylenes. The synthesis of derivatives (**5**) - (**8**) were accomplished by reacting the parent triphenylenes (**1**) - (**4**) in high dilution (CH_2Cl_2) with one equivalent of BBr_3 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]



1 R = C_5H_{11}
2 R = C_6H_{13}
3 R = C_7H_{15}
4 R = C_8H_{17}

5 R = C_5H_{11} (48 %)
6 R = C_6H_{13} (46 %)
7 R = C_7H_{15} (42 %)
8 R = C_8H_{17} (39 %)

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 CH_2Cl_2 (150 mL) at 25°C was added BBr_3 (0.67 mL of a 1.0 M solution in CH_2Cl_2) 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 ($MgSO_4$), 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 $C_{43}H_{62}O_6$:	C, 76.56;	H, 9.20;	N, 0.00
found:	76.74	9.00	0.00

^1H NMR ($CDCl_3$ / TMS): δ = 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^+)

Acknowledgements

We thank the EPSRC Mass Spectrometry Service Centre Service Centre, University of Wales, Swansea.

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5 Satisfactory analytical data were obtained for all compounds.

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(Received in the UK 26th March, 1997)