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## Liquid Crystals

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# Preliminary communication

## Synthesis and mesomorphic properties of two semi-fluorocarbon chain liquid crystals

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Two chiral molecules, each having a long fluoroalkylalkoxy chain, have been synthesized. Their phase transition temperatures have been measured by texture observation in a polarizing microscope and confirmed by differential scanning calorimetry. Very broad SmC\* phase is found in one of these two compounds

Perfluorinated alkanes are more rigid and linear than unsubstituted alkanes, and due to their extremely low surface energy are less miscible [1–5]. Therefore, the replacement of a perfluorinated alkyl group in the tail of a rigid rod-like molecular liquid crystal enhances the formation of a lamellar phase. In particular, LCs whose molecules have semi-fluorinated chains, exhibit tilted smectic properties. Since the electro-optical properties of ferroelectric chiral smectic C liquid crystals were first reported by Clark and Lagerwall, there has been considerable interest in the synthesis of new SmC\* materials. In the search for new compounds with broad SmC phases chiral groups were introduced into compounds with long semi-fluorocarbon chains. As yet, only a few papers have been published in this field [6–9].

The synthesis of the target molecules is outlined in the scheme. A semi-fluorocarbon chain was first synthesized. The acid **1** was then prepared by hydrolysis from the corresponding methyl ester. The protected 4-hydroxybenzoic acid was esterified with the chiral alcohol using the DCC/DMAP system in dry THF solution. The benzyl ether thus produced was then converted to the corresponding phenol ester **3** by removal of the benzyl protecting group by hydrogenation at room temperature in the presence of palladium on active carbon in ethyl acetate. The phenol **4** was obtained from the mono-protected 1,2'-biphenyl-4,4'-diol by a two-step process. Finally, the phenols **3** and **4** were esterified with the *para*-semi-fluoroalkoxy benzoic acid **1** using DCC/DMAP system in dry THF, to yield, respectively, the target

compounds **A** and **B**. All the intermediates and the final compounds were characterized by MS, EA, IR, <sup>1</sup>H NMR and <sup>19</sup>F NMR spectroscopy.

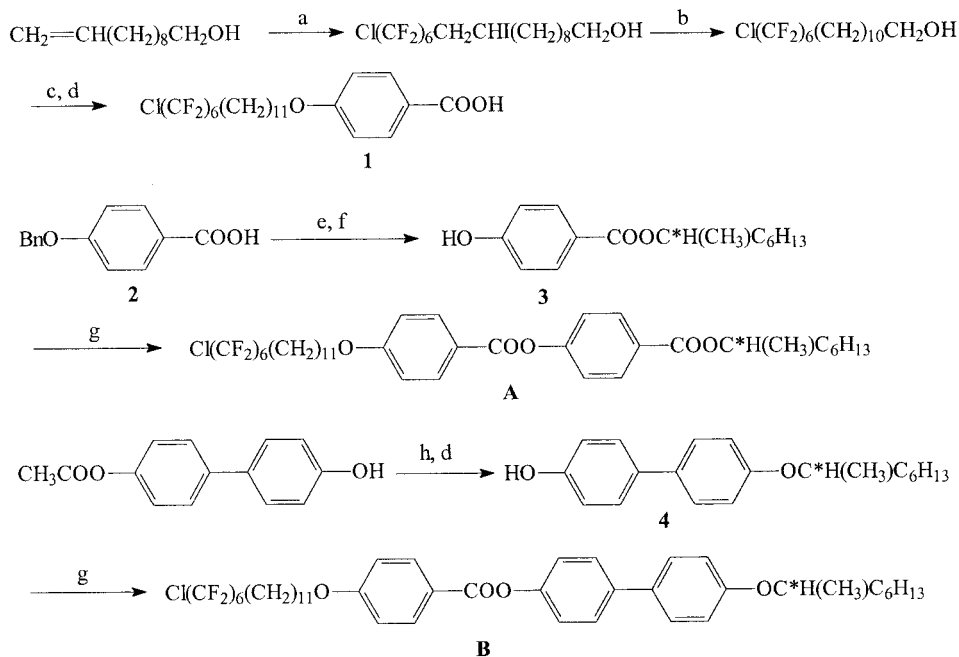
The phase transition temperatures of the target compounds were measured visually by optical microscopy using a polarizing microscope (Olympus PM-6) fitted with a heating stage (Mettler FP-80) and a control unit (FP-82), and by differential scanning calorimetry (DSC, Shimadzu DSC-50 calorimeter with a data system, heating and cooling rate 5°C min<sup>-1</sup>). The transition temperatures reported in this paper were the peak values of the transition from DSC traces, and are summarized in the table.

The compound **A** shows a monotropic SmA phase. In the compound **B** the carboxy group of compound **A** is changed to a 1,4-phenylene group. Due to this change the clearing point of compound **B** increases by 82°C and melting point by 21°C. Compound **B** shows enantiotropic SmA and SmC\* phases, and a monotropic SmB phase. It is interesting that such a broad (54°C) SmC\* phase was found in compound **B**; this compound may be useful in FLC mixtures. Further studies on this type of compound are planned.

Table. Transition temperatures of the compounds synthesized: Cr = crystal, S = smectic phase, I = isotropic phase, Recr = recrystal.

Compound	Transition temperatures/°C
<b>A</b>	Cr 61.9 I 49.5 SmA 38.4 Recr
<b>B</b>	Cr 83.1 SmC* 137.5 SmA 144.0 I 142.2 SmA 135.5 SmC* 67.4 SmB 54.7 Recr

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Scheme. Synthesis route for compounds **A** and **B**. Reagents and condition: (a)  $\text{Cl}(\text{CF}_2)_6\text{I}$ ,  $\text{Na}_2\text{S}_2\text{O}_4$ ,  $\text{NaHCO}_3$ ,  $\text{CH}_3\text{CN}$ ,  $\text{H}_2\text{O}$ ; (b)  $\text{Zn}$ ,  $\text{CH}_3\text{COOH}$ ; (c) methyl *p*-I-benzoate,  $\text{DEAD}/\text{PPh}_3$ ,  $\text{THF}$ ; (d)  $\text{NaOH}$ ,  $\text{THF}/\text{H}_2\text{O}$ ; (e)  $(S)\text{-C}_6\text{H}_{13}\text{C}^*\text{H}(\text{CH}_3)\text{OH}$ ,  $\text{DCC}/\text{DMAP}$ ; (f)  $\text{H}_2$ , 10%  $\text{Pd}/\text{C}$ ; (g) **1**,  $\text{DCC}/\text{DMAP}$ ,  $\text{THF}$ ; (h)  $(S)\text{-C}_6\text{H}_{13}\text{C}^*\text{H}(\text{CH}_3)\text{OH}$ ,  $\text{DEAD}/\text{PPh}_3$ .

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