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Liquid Crystals

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New ferroelectric liquid crystals for high-performance optical devices

Roberta Cassano^{a*}, Roman Dąbrowski^b, Jerzy Dziaduszek^b, Sonia Trombino^a, Francesca Iemma^a, Fiore Pasquale Nicoletta^a, Giovanni De Filipo^c and Nevio Picci^a

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The synthesis, characterisation, helical pitch and electro-optical response of new ferroelectric liquid crystals, 4'-(ω -acryloxyalkoxy)-, 4'-(ω -alkanoyloxyalkoxy)- or 4'-(ω -perfluoroalkanoyloxy)alkoxybiphenyl-4-yl (*S*)-4-(1-octyloxy)benzoates, bearing either polymerisable or non-polymerisable terminal groups, are described. The synthesised liquid crystals exhibit both a chiral smectic C phase and a chiral nematic phase. The helical pitch and electro-optical properties of the new compounds were determined as a function of temperature.

Keywords: ferroelectric liquid crystals; liquid crystal polymers; high-performance optical devices

1. Introduction

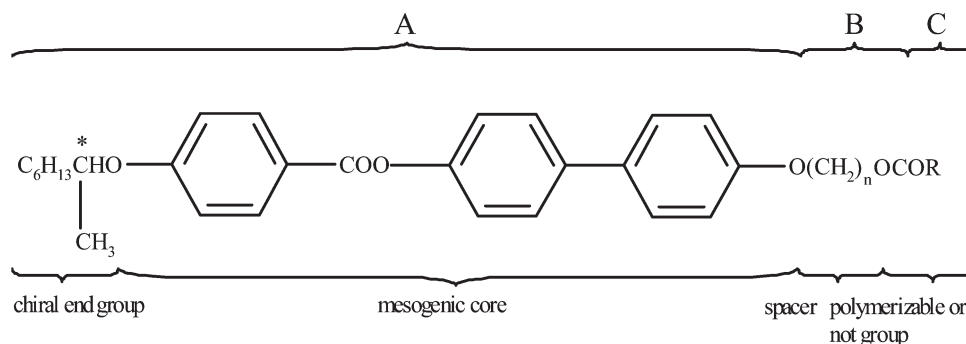
Ferroelectric liquid crystals (FLCs) were discovered in 1975 by Meyer *et al.* (1) and since then both the physics as well as the technology of FLCs have been extensively developed (2–8). Significant interest has been aroused in the chiral smectic C (SmC*) phases owing to their fast electro-optical response, which is useful in many applications. In fact, SmC* phases possess a spontaneous polarisation arising from the presence of a chiral centre in the molecules. Such chirality induces the precession of the direction of the tilt angle and in-plane spontaneous polarisation, leading to a helical structure with an axis perpendicular to the smectic layers. This structure can be deformed by either an external electric field applied perpendicular to the helical axis or by surface treatments of the cell substrates within which the liquid crystal is confined (9). Recently, there has been considerable interest in the synthesis of new liquid crystalline molecules, which may exhibit ferroelectric mesophases in an attempt to promote the development of novel fast-switching electro-optical devices and displays (10). In particular, a great deal of research has been dedicated to the synthesis and polymerisation of ferroelectric monomers (11–14). Liquid crystal polymers (LCPs) exhibit both the properties of liquid crystals and those of polymers, since the mesophase can be frozen in the polymer glass. Since ferroelectricity can be generated by mesogenic side groups bearing chiral carbon atoms, ferroelectric liquid crystal polymers (FLCPs) are side-chain polymers with the mesogen groups in the side chain. Polyacrylates represent the most important group of FLCPs. In this paper, the synthesis and

characterisation are described of the mesogenic compounds **1** and **2** that have the molecular structure shown in Scheme 1. The aim of this work was to obtain a mesogenic monomer with tilted SmC phase and with a broad temperature range of nematic phase. The designed compounds, **1** and **2**, have similar core structure to the ferroelectric compounds described by Inukai *et al.* (15) with the difference that the terminal chain contains a polymerisable group separated with an oxypolymethylene group from a rigid core. Non-polymerisable analogues, i.e. mesogens with an alkanoyloxy or perfluoroalkanoyloxy terminal group instead of an acryloxy group, were also prepared (16). The helical pitch and the electro-optical properties of all the synthesised materials are also discussed.

2. Experimental

The chemical structures of intermediates and target materials were analysed by NMR spectroscopy, using a Bruker 300 AC NMR spectrometer, and infrared and mass spectroscopy, respectively, recorded on a FTIR Perkin Elmer-Paragon 1000 PC and Hewlett Packard GC-MSD 5972. The purity was checked by thin-layer chromatography and further confirmed by elemental analysis. The transition temperatures of the samples were determined by differential scanning calorimetry (DSC) using DSC-NETZSCH 200 and DSC-SETARAM 141 instruments. The heating and cooling rates were 10°C min⁻¹ and 5°C min⁻¹, respectively; the mass of the sample was 4.0 mg. Mesophases were identified by observation of the microscopic textures using a Leitz Laborlux 12 Pol

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Scheme 1. Molecular structure of mesogenic compounds **1** and **2**. In particular, $n=4$, $R=-CH=CH_2$, (**1a**); $n=6$, $R=-CH=CH_2$, (**1b**); $n=6$, $R=-OCH_3$, (**2a**); $n=6$, $R=-C_3H_7$, (**2b**); $n=6$, $R=-C_3F_7$ (**2c**).

polarising optical microscope connected with Linkam PR 600 heating stage.

The helical pitch, p , was measured by the Grandjean–Cano method (17, 18) using wedge-shaped cells with an angle lower than 0.5° . Cells substrates were coated with a thin layer of buffed polyvinyl alcohol (PVA) in order to achieve a planar orientation for the measurements in the chiral nematic phase, and with a thin layer of *N,N*-dimethyl-*n*-octadecyl-3-aminopropyltrimethoxysilyl chloride (DMOAP) in order to gain a homeotropic orientation in the SmC* phase. In these geometries, the average direction of the long axis of the molecules in the chiral nematic (N*) phase is parallel to the substrates, whereas it is perpendicular in the SmC* phase. The electro-optical response of the SmC* phase, including tilt angle and response time as a function of temperature, was investigated with the experimental setup described in a previous work (19). Liquid crystals were confined in conducting cells (Linkam, $5\ \mu\text{m}$ thick, antiparallel planar alignment), mounted between crossed polarisers. Tilt angles were measured by finding the extinction positions of the two fully switched states obtained by reversing the applied electric field (0.2 Hz square wave with electric field strength of $5\ \text{V}\ \mu\text{m}^{-1}$).

The switching time, defined as the time for the transmittance to change from 10% to 90% of its maximum value, was determined by monitoring the drive signal (1 Hz square wave with electric field strength of $5\ \text{V}\ \mu\text{m}^{-1}$) and the response of the photodiode.

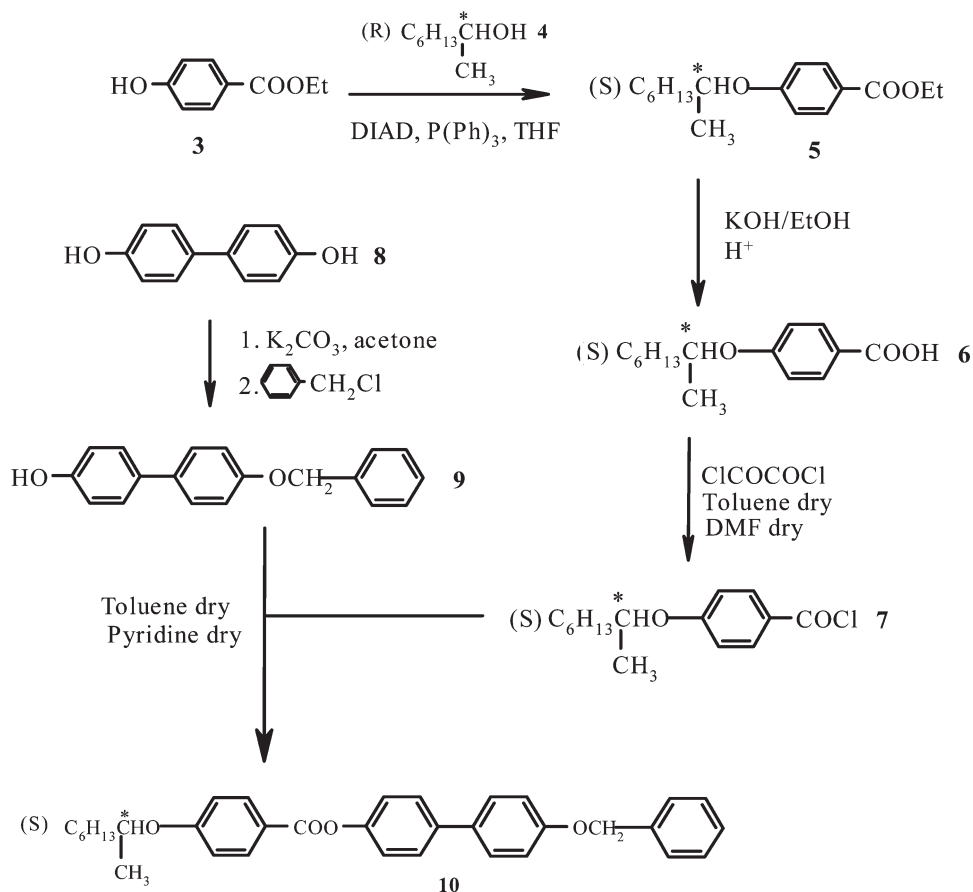
3. Results and discussion

Synthesis

The starting chiral materials were purchased from Aldrich Chemical Co. USA (purity greater than 99%). All remaining starting materials were commercial products from Aldrich of high purity and

used without further purifications. Thin-layer chromatography (TLC) was performed with TLC sheets coated with silica; spots were detected by UV irradiation. Silica gel (Merk Kieselgel 60, 70–230 mesh) was used for column chromatography. Anhydrous organic solvents were obtained by distillation before use. Detailed procedures for the syntheses of intermediates and final compounds are as follows. The synthetic procedures for the target compounds were carried out as outlined in Schemes 2–4. The synthesis of the mesogenic monomer **1** is outlined in Scheme 2 (linkage of the chiral group to the mesogenic core, part A) and further in Scheme 3, (introduction of the spacer, part B, and of the polymerisable group, part C). The introduction of the other terminal groups (not polymerisable) is shown in Scheme 4.

The synthetic steps can be summarised as follows. Ethyl 4-hydroxybenzoate (**3**, 2.0 g, 0.012 mol) was treated with (*R*)-2-octanol (**4**, 1.7 g, 0.013 mol) in the presence of diisopropyl azodicarboxylate (DIAD) and triphenylphosphine ($\text{P}(\text{C}_6\text{H}_5)_3$) in dry tetrahydrofuran (THF) and the resulting ester, (*S*)-4-(2-octyloxy)ethyl benzoate (**5**), was hydrolysed without purification to the (*S*)-4-(2-octyloxy)benzoic acid (**6**) with melting point of 66.1°C . The latter (**6**, 5.8 g, 0.022 mol) was transformed into the liquid crystalline ester (*S*)-4'-(benzyloxy)biphenyl 4-(1-octyloxy)benzoate (**10**) after initial conversion in the (*S*)-4-(2-octyloxy)benzoyl chloride (**7**), via reaction with oxalyl chloride (2.1 ml, 0.024 mol) in dry toluene and few drops of dry *N,N*-dimethylformamide (DMF), and subsequent coupling in dry toluene with compound **9** (6.1 g, 0.022 mol), using dry pyridine to quench HCl. Compound **9**, with a melting point of 189.0°C , was prepared through monosubstitution of the commercial 4,4'-dihydroxybiphenyl (**8**) (8.0 g, 0.043 mol) with benzyl chloride (5.4 g, 0.043 mol) in a potassium carbonate (K_2CO_3) (8.9 g, 0.064 mol) solution of dry acetone. Ester **10** (3.6 g, 0.071 mol), treated in dry THF with H_2 and



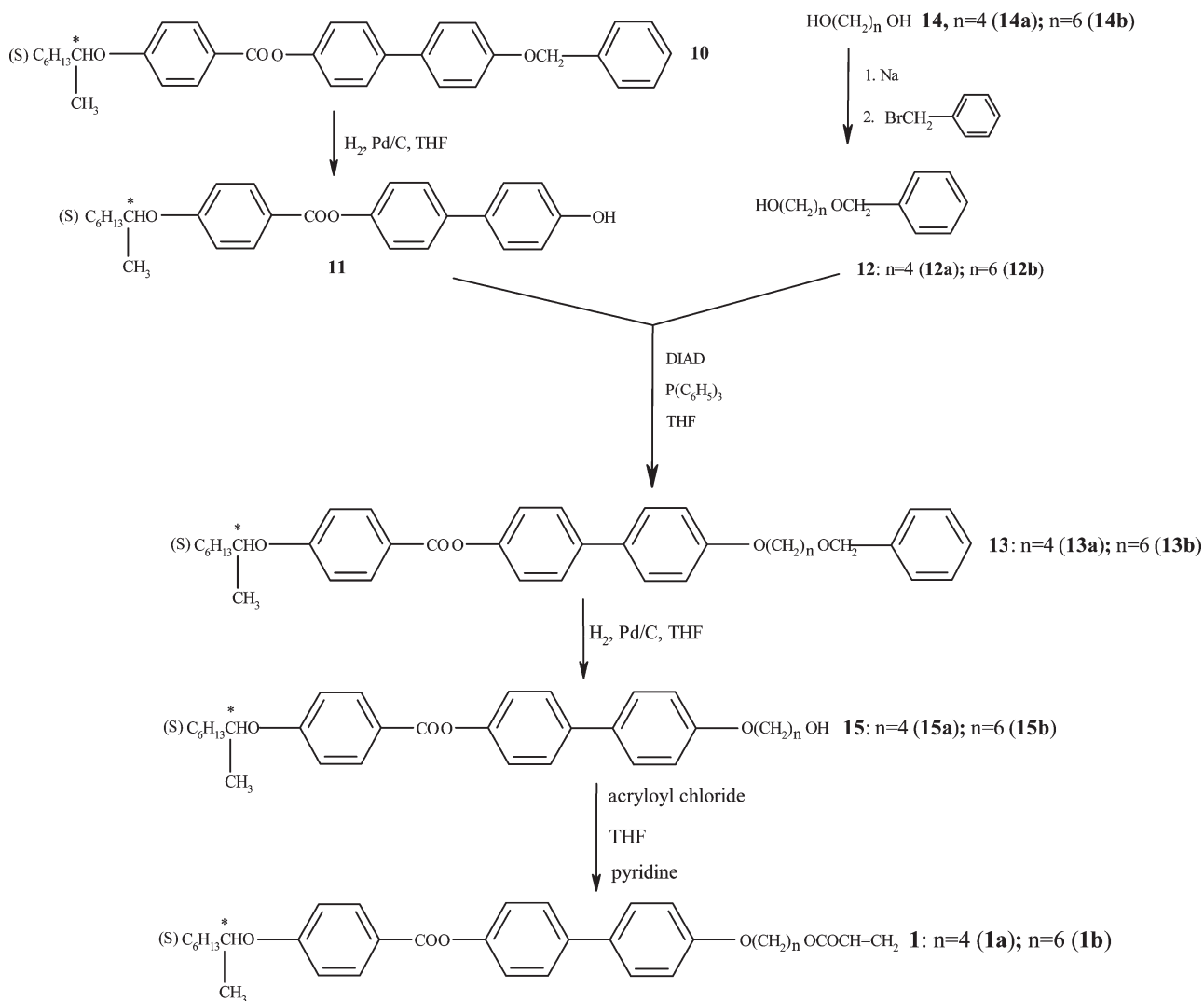
Scheme 2. Synthetic route for intermediate **10**.

Pd/C, was transformed to liquid crystalline intermediate phenol **11**. Compounds **13a** and **13b** were obtained by Mitsunobu etherification of **11** (2.0 g, 0.005 mol), using 4-benzyloxy-1-butanol (**12a**) (0.91 g, 0.005 mol) or 6-benzyloxy-1-hexanol (**12b**) (1.0 g, 0.005 mol), respectively, in the presence of equimolar amount of $P(C_6H_5)_3$ and DIAD. Compounds **12a** and **12b**, were obtained from the commercially available 1,4-butanediol (**14a**, 1.0 g, 0.011 mol) or 1,6-hexandiol (**14b**, 1.4 g, 0.011 mol), respectively, treating them first with metallic sodium and then with benzyl bromide. Debenzylation of **13a** or **13b**, with H_2 and Pd/C in dry THF, gave the liquid crystalline compounds **15a** or **15b**. The latter, after reaction with freshly distilled acryloyl chloride (in the molar ratio of 1:2) in dry THF and in the presence of anhydrous pyridine, afforded final polymerisable mesogens **1a** and **1b**. Esterification of **15b** (0.25 g, 0.480 mmol) with methyl chloroformate (0.06 g, 0.640 mmol), butyryl chloride (0.07 g, 0.640 mmol) and heptafluorobutyric acid (0.14 g, 0.640 mmol) gave the compounds **2a**, **2b** and **2c** (Scheme 4). The formation of all intermediates and final compounds was unambiguously confirmed by

FT-IR and 1H NMR spectroscopy. All the target materials were also analysed by elemental analysis for purity (Tables 1 and 2).

Mesomorphic properties

The liquid crystalline properties of the target molecules were investigated primarily with polarising optical microscopy (POM) and DSC. The results are summarised in Table 3. The microscopic patterns, confirming the mesophases, were identified through comparison with known reference textures (20). The compounds have both N^* and SmC^* phases. Representative DSC traces of these mesogens are shown in Figure 1. The cooling scan looks non-identical to the heating scan. The SmC^* and cholesteric phases of compounds **2b–2c** were similarly detected and confirmed by POM and DSC. As shown in Table 3, the N^* phase temperature range of sample **1a** is greater than that exhibited by sample **1b**; in contrast, the SmC^* temperature range is larger in **1b** and is dependent on the spacer length (spacer B in Scheme 1). Both phase temperature ranges depend on the type of

Scheme 3. Procedure for the synthesis of **1a–1b**.

chiral end group (samples **2a–2c**) due to their different physicochemical properties.

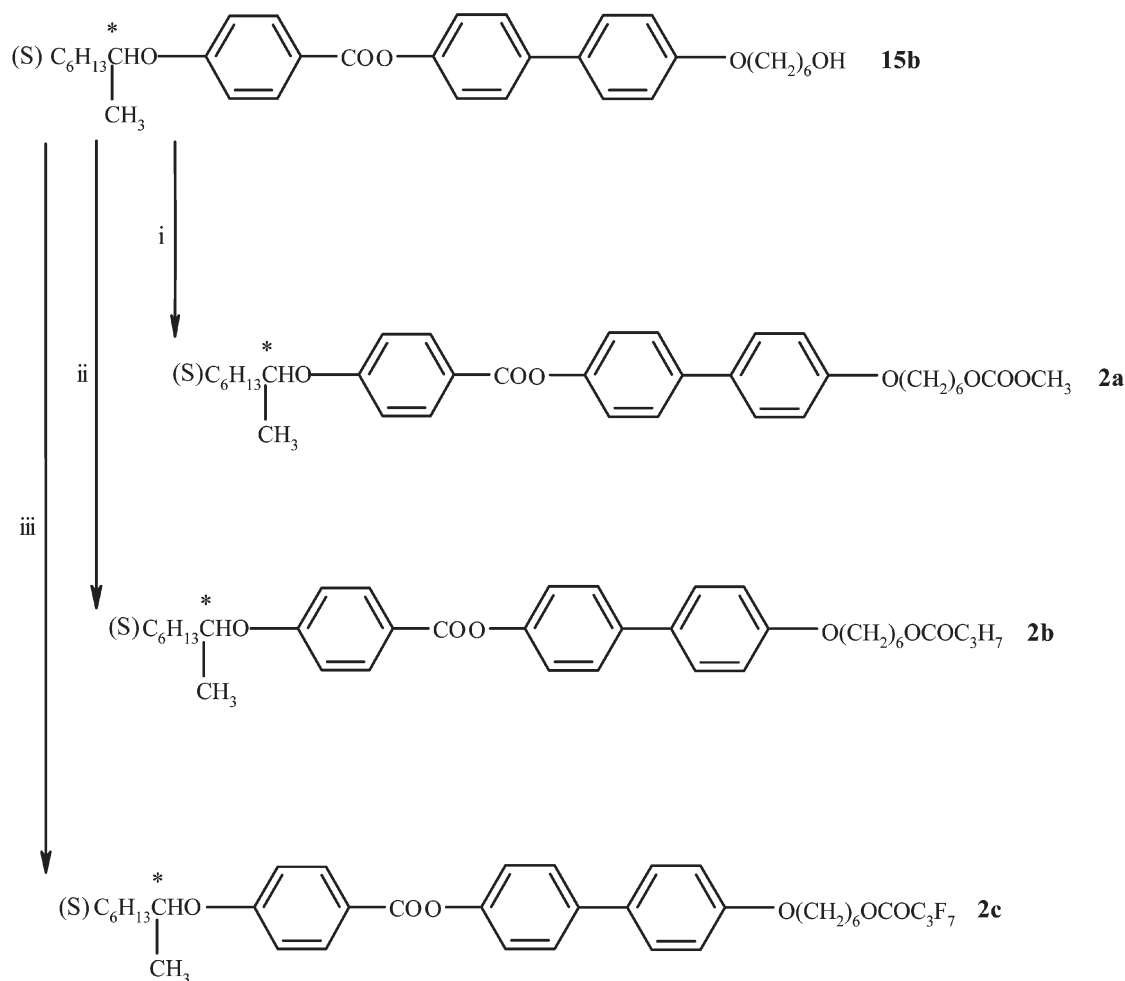
Structural and electro-optical properties

The helical pitch, tilt angle and response time exhibited by the samples revealed very similar behaviour; hence, detailed results are presented only for **2a**.

Figure 2 shows the variation of the helical pitch, p , in the SmC* and N* phases for compound **2a**. In the N* phase, p is very short ($p \sim 0.3 \mu m$). On cooling, the pitch increases its value, reaching a maximum for the SmC* phase. In the SmC* phase, the helical pitch decreases from values of about 1.5–2.0 μm (1.7 μm for **2a** at the SmC*–N* transition temperature) to around 1.0 μm (at the Cr–SmC* transition temperature).

The tilt angle, θ , of the different samples as a function of temperature exhibits a rather regular decrease from its starting values ($20^\circ < \theta < 30^\circ$), as shown in Figure 3 for compound **2a**. The tilt angle behaves according to theory and saturates at roughly 25° , as found for many compounds.

As shown in Figure 4, the response time increases once the tilt angle saturates, maybe due the temperature dependence of the rotational viscosity in the SmC* phase, which obeys to an Arrhenius-like law (21). The response times are of the order of hundreds of microseconds ($\sim 250 \mu s$ in the middle of the SmC* range), which would make such compounds useful for new technological applications. If we compare the performance of our compounds with the similar ones studied by Inukai *et al.* (15), it is possible to observe that our liquid crystals exhibit lower tilt angles in their SmC* phases, but larger helical pitches. In



Scheme 4. The synthesis of **2a–2c**: (i) $\text{CH}_3\text{OCOC}\text{Cl}$, Py, CH_2Cl_2 ; (ii) $\text{C}_2\text{H}_5\text{COCl}$, Py, CH_2Cl_2 ; (iii) $\text{C}_2\text{F}_5\text{COOH}$, THF, DEAD, $\text{P}(\text{Ph})_3$.

Table 1. Characterisation data of final compounds.

Compound	Yield/%	Elemental analysis/%		IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$	$^1\text{H NMR } \delta/\text{ppm}$
		Found	Calculated		
1a	49	C 75.37, H 7.44	C 74.97, H 7.40	3063, 3015, 2932, 2860, 1708, 1607, 1072, 963, 930	CD_3OD : 0.90 (m, 3H), 1.40–1.71 (m, 17H), 4.02 (t, 2H), 4.10 (m, 2H), 4.50 (m, 1H), 5.80 (dd, 1H), 6.11 (dd, 1H), 6.43 (dd, 1H), 6.90 (m, 2H), 7.01 (m, 2H), 7.24 (m, 2H), 7.53 (m, 2H), 7.61 (m, 2H), 8.20 (m, 2H)
1b	45	C 75.58, H 7.80	C 75.49, H 7.74	3069, 3042, 2926, 2856, 1716, 1603, 1076, 966, 931	CDCl_3 : 1.25–2.54 (m, 24H), 4.01–4.54 (m, 5H), 5.84 (dd, 1H), 6.14 (dd, 1H), 6.42 (dd, 1H), 6.97 (m, 2H), 6.99 (m, 2H), 7.26 (m, 2H), 7.52 (m, 2H), 7.6 (m, 2H), 8.17 (m, 2H)
2a	72	C 71.05, H 7.74	C 70.81, H 7.70	3070, 2927, 2856, 1740, 1727, 1605, 1268	CDCl_3 : 0.83 (t, 3H), 1.24–1.86 (m, 21H), 3.61 (t, 2H), 3.78 (s, 3H), 3.94 (t, 2H), 4.43 (sextuplet, 1H), 6.89 (m, 2H), 6.92 (m, 2H), 7.18 (m, 2H), 7.45 (m, 2H), 7.52 (m, 2H), 8.09 (m, 2H)
2b	82	C 75.11, H 8	C 75.48, H 8.22	3064, 2930, 2856, 1723, 1275, 1078	CDCl_3 : 0.83 (t, 3H), 0.89 (t, 3H), 1.24–1.86 (m, 23H), 2.21 (t, 2H), 3.94 (t, 2H), 4.03 (t, 2H), 4.44 (sextuplet, 1H), 6.89 (m, 2H), 7.15 (m, 2H), 7.44 (m, 4H), 7.52 (m, 2H), 8.10 (m, 2H)
2c	82	C 61.81, H 5.74	C 62.18, H 5.78	2911, 2854, 1732, 1700, 1276, 1218, 1076	CDCl_3 : 1.47–1.85 (m, 24H), 3.67 (t, 2H), 4.01 (t, 2H), 4.49 (m, 1H), 6.95 (d, 2H), 6.98 (d, 2H), 7.25 (d, 2H), 7.51 (d, 2H), 7.58 (d, 2H), 8.15 (d, 2H)

Table 2. Characterisation data of intermediate compounds.

Compound	Yield/%	Elemental analysis/%		GC/MS, m/z	IR (KBr) $\nu_{\max}/\text{cm}^{-1}$	^1H NMR δ/ppm
		Found	Calculated			
6	50	C 72.06, H 8.82	C 71.97, H 8.86	250 (M^+ , 12%), 138 (100%)	3038, 3023, 2928, 2856, 1699, 1255, 1076	CD_6SO : 0.73 (t, 2H), 1.14–1.54 (m, 13H), 4.42 (m, 1H), 6.88 (m, 2H), 7.78 (m, 2H), 12.52 (s, 1H)
9	56	C 82.32, H 5.86	C 82.58, H 5.83	276 (M^+ , 52%), 91 (100%)	3241, 2902, 2859, 1602, 1244, 1082	CD_6SO : 4.47 (s, 2H), 5.1 (s, 1H), 6.96 (m, 2H), 6.75 (m, 2H), 7.22–7.45 (m, 9H)
10	32	C 79.92, H 7.20	C 80.28, H 7.13	508 (M^+ , 8%), 233 (28%), 121 (100%)	3034, 2955, 2855, 1722, 1603, 1282, 1073	CDCl_3 : 0.83 (t, 3H), 1.24–1.79 (m, 13H), 3.73 (s, 2H), 4.43 (sextuplet, 1H), 4.47 (s, 2H), 6.90 (m, 2H), 7.18 (m, 2H), 7.44–7.54 (m, 11H), 8.1 (m, 2H)
11	98	C 77.11, H 7.18	C 77.48 H 7.22	418 (M^+ , 2%), 233 (14%), 121 (100%)	3443, 3038, 3020, 2929, 2856, 1698, 1602, 1497, 1250	CDCl_3 : 0.85 (t, 3H), 1.20–1.80 (m, 13H), 4.45 (m, 2H), 6.77 (m, 2H), 6.90 (m, 2H), 7.15–7.66 (m, 6H), 8.10 (m, 2H)
12a	59	C 73.06, H 9	C 73.30 H 8.95	180 (M^+ , 4%), 91 (100%)	3391, 3063, 3030, 2937, 2862, 1099, 1064	CD_3OD : 1.40 (m, 4H), 3.50 (s, 2H), 3.60 (s, 2H), 4.20 (m, 1H), 4.53 (s, 2H), and 7.45 (m, 5H)
12b	91	C 75.05, H 9.64	C 74.96 H 9.68	208 (M^+ , 2%), 107 (45%), 91 (100%)	3029, 2950, 2855, 1495, 1099	CD_6SO : 1.41–1.53 (m, 8H), 3.34 (m, 2H), 3.35 (m, 2H), 4.36 (s, 2H), 4.39 (s, 1H), 7.25 (m, 5H)
13a	61	C 78.23, H 7.72	C 78.59 H 7.64	91 (100%)	3037, 2846, 1728	CD_3OD : 0.90 (m, 3H), 1.40–1.55 (m, 10H), 1.61–1.80 (m, 7H), 3.40 (s, 2H), 4.10 (m, 2H), 4.42 (s, 2H), 4.50 (m, 1H), 7.01 (m, 2H), 7.08–7.18 (m, 9 H), 7.21 (m, 2H), 7.45 (m, 2H), 8.30 (m, 2H)
13b	80	C 79.00, H 8.04	C 78.91 H 7.95	91 (100%)	3033, 2841, 1730, 1280	CDCl_3 : 0.85 (t, 3H), 1.25–1.79 (m, 21H), 3.45 (t, 2H), 3.95 (t, 2H), 4.45 (m, 1H), 4.47 (s, 2H), 6.92 (m, 2H), 7.15–7.45 (m, 9H), 7.46 (m, 2H), 7.53 (m, 2H), 8.1 (m, 2H)
15a	89	C 75.38, H 7.55	C 76.04 H 7.61	77 (17%), 277 (100%)	3430, 3075, 3038, 2933, 1730, 1602, 1497, 1250	CD_3OD : 0.90 (m, 3H), 1.41–1.62 (m, 17H), 3.60 (s, 2H), 4.10 (s, 2H), 4.21 (m, 1H), 4.53 (m, 1H), 7.01 (m, 2H), 7.08 (m, 2H), 7.21 (m, 2H), 7.45 (m, 2H), 7.48 (m, 2H), d 8.30 (m, 2H)
15b	87	C 76.06, H 8.13	C 76.41 H 8.16	418 (5%), 233 (32%), 121 (100%)	3423, 3078, 3036, 2929, 2856, 1735, 1604, 1255, 1076	CD_6SO : 0.77 (t, 3H), 1.18–1.39 (m, 21H), 3.32 (m, 2H), 3.92 (t, 2H), 4.31 (t, 1H), 4.53 (sextuplet, 1H), 6.94 (m, 2H), 7.02 (m, 2H), 7.22 (m, 2H), 7.53 (m, 2H), 7.56 (m, 2H), 7.99 (m, 2H)

addition, our SmC* phases are characterised by intermediate spontaneous polarisation values (around 30 nC cm^{-2} in the middle of chiral smectic range). Pitch behaviour is similar to that shown by ferroelectric liquid crystals of a biphenyl alkyloxybenzoate series

studied by Hemine *et al.* (22), which are characterised by larger tilt angles and spontaneous polarisation values (larger than 100 nC cm^{-2}), faster electro-optical response times, but higher SmC* temperature ranges.

Table 3. Transition temperatures ($^{\circ}\text{C}$) of mesogenic compounds.

Compound	Heating	Cooling
10	Cr 128.9 N* 151.0 I	I 145.9 N* 115.4 Cr
11	Cr 78.9 SmC* 155.3 I	I 150.9 SmC* 67.0 Cr
13b	Cr 48.1 SmC* 56.0 N* 79.8 I	I 77.3 N* 53.5 SmC* 47.4 Cr
15b	Cr 104.2 N* 130.9 I	I 130.3 N* 87.3 SmC* 83.9 Cr
1a	Cr 95.9 I	I 96.6 N* 81.6 SmC* 80.3 Cr
1b	Cr 92.8 I	I 95.2 N* 83.7 SmC* 78.0 Cr
2a	Cr 94.0 I	I 94.3 N* 81.2 SmC* 51.1 Cr
2b	Cr 62.4 SmC* 78.4 N* 86.0 I	I 85.4 N* 77.6 SmC* 47.5 Cr
2c	Cr 99.6 N* 123.1 I	I 121.6 N* 90.3 SmC* 88.2 Cr

Cr=crystal; SmC*=chiral smectic C; N*=chiral nematic; I=isotropic phase.

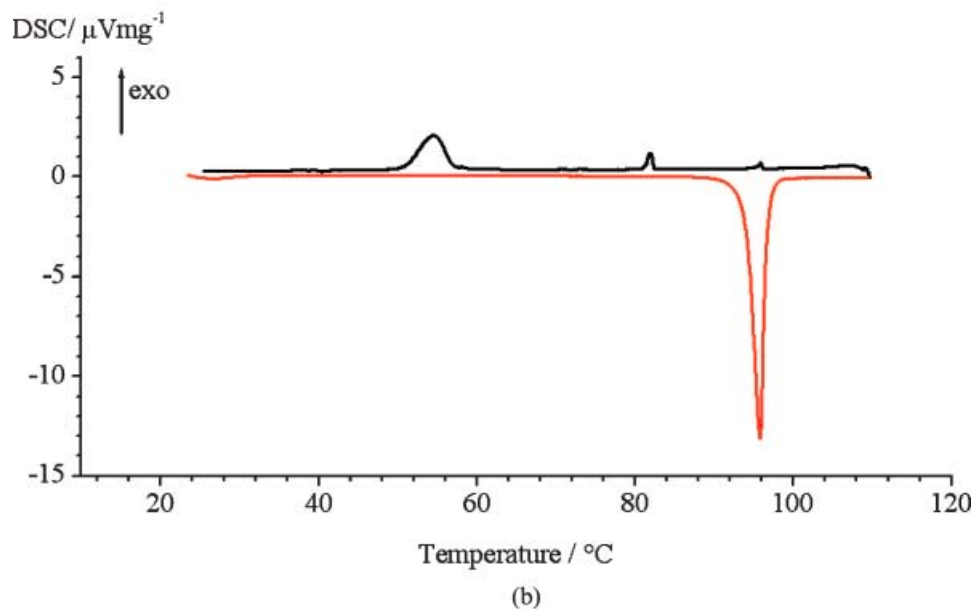
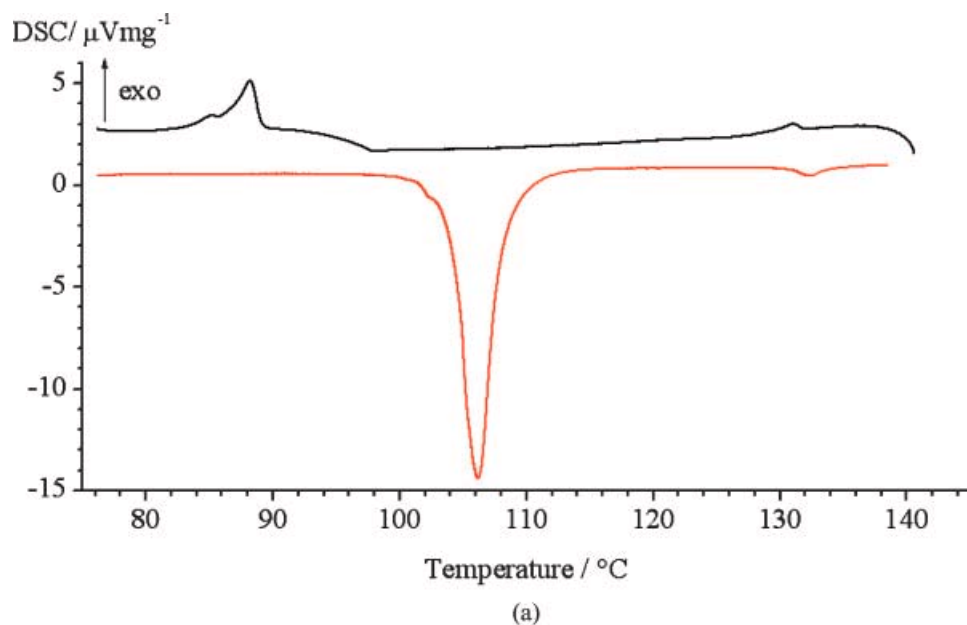


Figure 1. DSC thermogram of mesogens (a) **15b** and (b) **2a**.

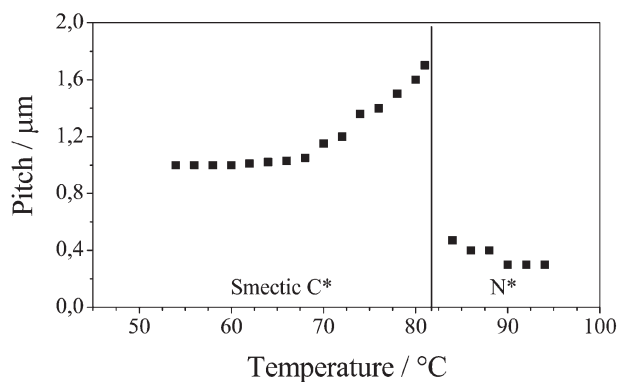


Figure 2. Temperature dependence of the helical pitch in the SmC* and N* phases of **2a**.

4. Conclusions

Chiral compounds **1a–1b** and **2a–2c** derived from (*R*)-2-octanol have been shown to possess both N* and SmC* phases. The compounds have been characterised from a thermal, structural and electro-optical point of view. These materials, which are characterised by small pitches and rather fast response times, could provide a molecular design for ferroelectric liquid crystals for future studies. Mesogenic monomers could be used to obtain polymers within which the molecular orientation is frozen. Non-polymerisable analogue mesogens with alkanoyloxy or perfluoroalkanoyloxy terminal groups could be used in order to adjust the polymer

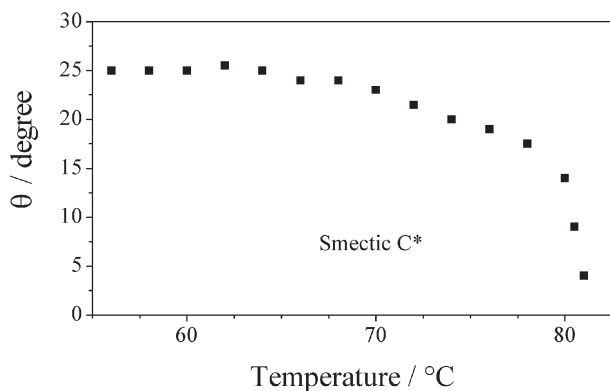


Figure 3. Tilt angle as a function of temperature in the SmC* phase of **2a**.

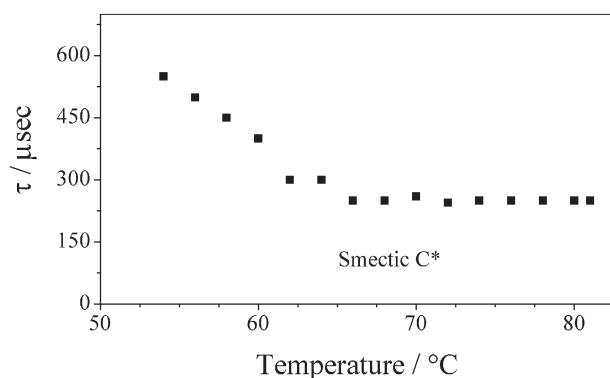


Figure 4. Electro-optical response time as a function of temperature for **2a**.

optical properties and then to obtain devices with a high optical contrast. In particular, the acrylic liquid crystalline monomers should allow polymeric networks to be obtained via UV photopolymerisation.

Acknowledgements

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