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## Liquid Crystals

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### Photoinduced phase transitions and helix untwisting in the SmC\* phase of a novel cinnamoyl-based liquid crystal

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## Photoinduced phase transitions and helix untwisting in the SmC\* phase of a novel cinnamoyl-based liquid crystal

Alexey Bobrovsky<sup>a\*</sup>, Valery Shibaev<sup>a</sup>, Věra Hamplová<sup>b</sup>, Miroslav Kašpar<sup>b</sup>, Vladimira Novotná<sup>b</sup>, Milada Glogarová<sup>b</sup> and Evgeniy Pozhidaev<sup>c</sup>

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A photoinduced phase transition and helix untwisting in a new liquid crystal forming the SmC\* phase were studied in detail. The compound consists of a cinnamoyl photosensitive fragment with C=C double bond capable of photoisomerisation and photocycloaddition. It was shown that ultraviolet (UV) irradiation (365 nm) induces an extreme decrease in phase transitions temperatures (SmC\*–SmA\*, SmA\*–N\*, N\*–I). Vertically aligned samples in the SmC\* phase cause selective light reflection in the visible spectral range. The light action results in a noticeable helix untwisting that causes a shift in the selective light reflection peak to the long-wavelength spectral region. The temperature dependence of spontaneous polarisation  $P_s$  was measured and it was found that UV irradiation induces a decrease in the values of  $P_s$ . Photo-optical phenomena taking place in the liquid crystal are attributed to the formation of photoproducts having low anisotropy, which disrupts mesophases.

**Keywords:** photoinduced phase transition; SmC\* phase; helix untwisting

### 1. Introduction

The combination of unique optical properties of liquid crystals (LCs) with photosensitivity provides a great possibility for the creation of a large variety of photochemical switches, which can be used in optics and optoelectronics (1–3). One of the most well-known photo-optical phenomena in LC systems is the so-called photoinduced isothermal phase transition. Compounds and blends containing photosensitive fragments capable of photoisomerisation and changing their shape can undergo a phase transition under the light action (4–9). Most studied substances of this type consist of an azobenzene photochrome unit (1–9) and *E–Z* isomerisation of such photochromes is well understood. In most cases this process leads to the formation of a *Z*-form having low anisotropy and can be used for photochemical switching of the phase behaviour of liquid crystalline individual compounds or mixtures containing azobenzene fragments.

Another promising class of photochromic compounds are cinnamic acid derivatives having photo-reactive C=C double bonds (10). In this case, not only can *E–Z* isomerisation of cinnamoyl derivatives take place, but also {2+2} photocycloaddition reactions and, as for azobenzenes, the formation of products having low anisotropy takes place. These types of

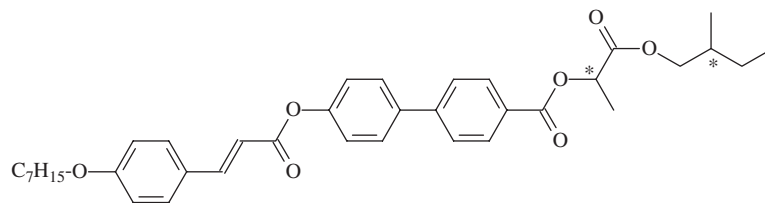
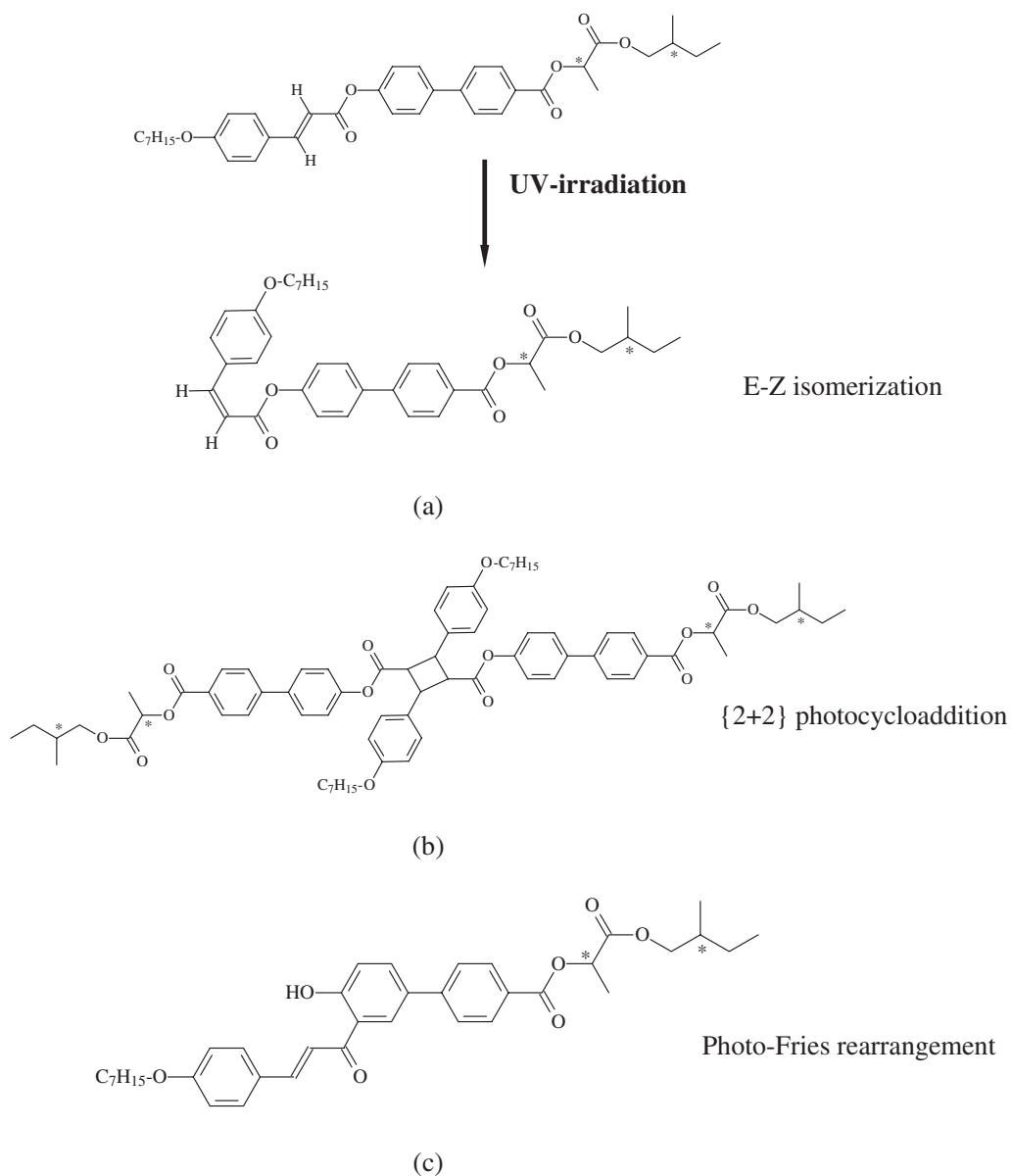
photochromes embedded in LC media were studied to much less an extent (11–17) and mostly from the point of view of LC photoalignment.

A relatively new area of research in the field of photosensitive LC materials concerns the possibility of photoregulation of the helix pitch of the cholesteric structure (and selective light reflection wavelength) by means of isomerisation of the chiral photochromic dopants (2, 18–23), which also can be included as side groups of copolymers (2). The principle of this photoregulation is based on changing the chiral fragment anisotropy leading to a decrease (or increase) in the helical twisting power resulting in helix untwisting (or twisting).

Despite the large number of papers devoted to photosensitive cholesterics with photovisible helix pitch, there are no publications regarding investigations of chiral smectic LC materials changing the selective light reflection under light irradiation. This is a quite surprising fact because there is a large amount of data devoted to the detailed study of photomodulation of spontaneous polarisation and electro-optical characteristics in photosensitive materials forming the SmC\* phase (24–27).

Taking into account the above-mentioned facts, a novel chiral substance **7SZL** (Scheme 1), containing the cinnamoyl-based photochromic fragment that forms

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Scheme 1. Compound **7SZL**.Scheme 2. Main photoprocesses taking place under ultraviolet irradiation of **7SZL** liquid crystal films.

the SmC\* phase with selective light reflection in the visible range, was synthesised and studied.

Ultraviolet (UV) irradiation of thin liquid crystalline films of this compound may lead to three main processes:

(a) *E-Z* isomerisation, (b) {2+2} photocycloaddition and (c) photo-Fries rearrangement. Possible products of these photoreactions are presented in Scheme 2. (In the case of photocycloaddition several isomers can

be produced, which are not shown here.) It is noteworthy that for all photoreactions the anisometry of the products is much lower than for the initial compound. This fact allows one to expect a decrease in the phase transition temperatures as well as helix untwisting of supramolecular (helical) structure of the SmC\* phase.

One of the important advantages of the cinnamoyl derivatives is related to the irreversibility of the above-mentioned photoprocesses which makes the investigation of the thermal behaviour and the determination of phase transitions more convenient from the experimental point of view. In contrast to azobenzene derivatives there is no competing thermal back isomerisation leading to returning the system to the initial state during heating.

In this paper we have focused our attention on the study of photochemically induced changes in phase behaviour and phase transition temperatures of the chosen chiral photochromic LC compound **7SZL**.

Special attention is paid to the investigation of the photo-optical properties of the helical structure of the SmC\* phase including a study of the kinetics of the helix untwisting under UV irradiation at different temperatures.

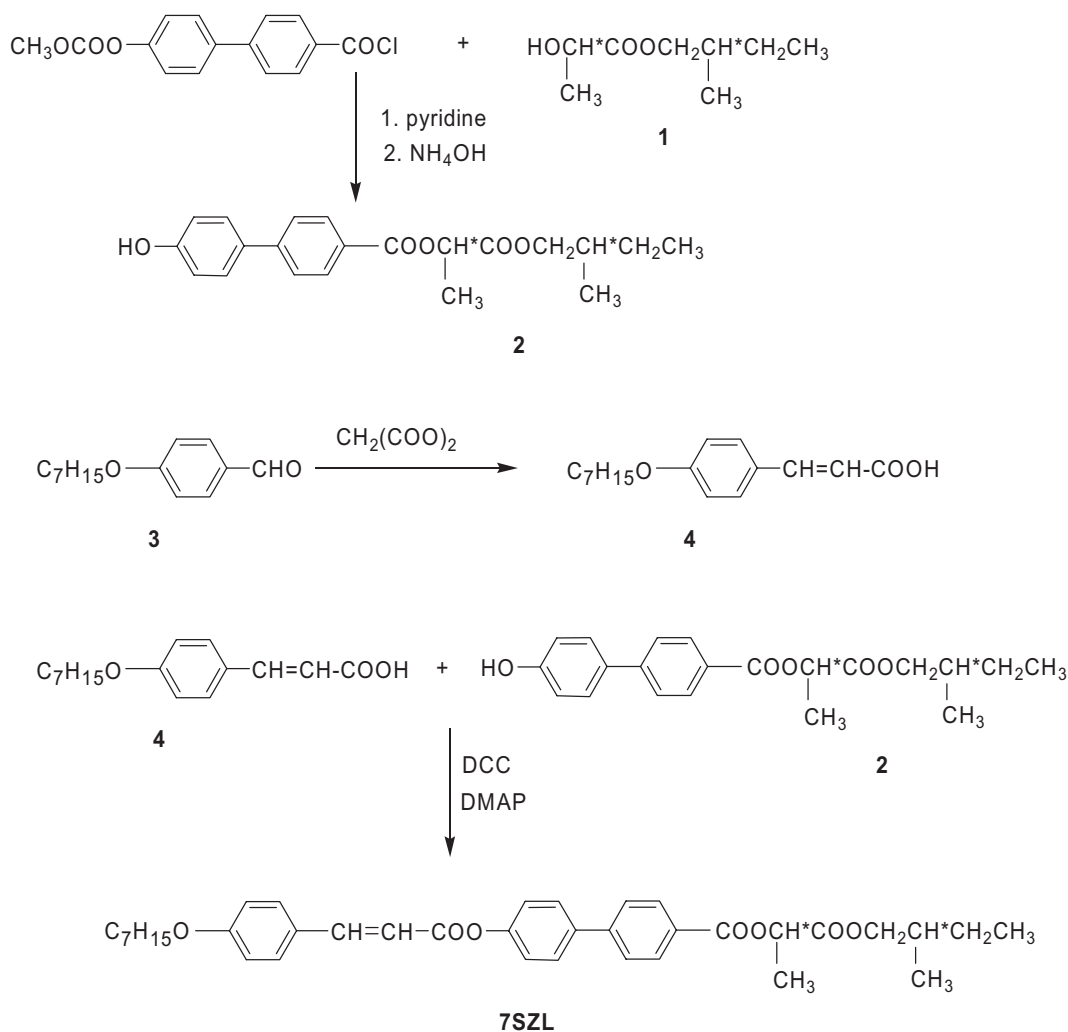
## 2. Experimental details

### 2.1 Synthesis

The synthesis of **7SZL** was carried out according to Scheme 3.

#### 2.1.1 Synthesis of chiral alcohol (**1**)

The process of synthesis of (S)-(-)-2-methylbutylacrylate (**1**) has been described in detail in (28). Optical rotation of this product was  $[\alpha]_D^{20} = -11,1$  (neat), which is the same value as described in (28).



Scheme 3. Scheme of the synthesis of **7SZL**.

### 2.1.2 Synthesis of phenol (2)

The mixture of protected 4-hydroxy-4'-biphenylcarbonylchloride and chiral alcohol (**1**) was boiled in pyridine/dichloromethane for several hours. Then the reaction mixture was washed with diluted hydrochloric acid and water. The organic layer was separated, solvents were evaporated and the solid remainder dissolved in tetrahydrofuran/chloroform (1:1) mixture and ammonolysed with concentrated  $\text{NH}_4\text{OH}$  at  $10^\circ\text{C}$ . The conversion was monitored by thin layer chromatography. Then the reaction mixture was washed with water and evaporated to dryness. After separation by column chromatography on silica gel (using a mixture of dichloromethane/acetone (99:1) as an eluent) compound **2** was obtained in 50% yield.

$^1\text{H}$  nuclear magnetic resonance (NMR) of compound **2**,  $\text{CDCl}_3$ , 300 MHz: 8.12d (2H, ortho to  $-\text{COO}$ ); 7.59d (2H, meta to  $-\text{COO}$ ); 7.42d (2H, meta to  $-\text{OH}$ ); 6.91d (2H, ortho to  $-\text{OH}$ ); 5.41q (1H,  $\text{ArCOOCH}^*$ ); 4.0 (2H,  $\text{COOCH}_2$ ); 1.6d (3H,  $\text{CH}_3\text{CHCOO}$ ); 1.2–1.7m (3H,  $\text{CH}-\text{CH}_2$ ); 0.9m (6H,  $\text{CH}_3$ ).

### 2.1.3 Synthesis of 4-alkyloxycinnamic acid (4)

4-alkoxybenzaldehyde (**3**) was prepared by a Williamson method using  $\text{K}_2\text{CO}_3$  as a base and acetone as a solvent. The mixture was boiled with stirring for 48 hours. The product was then purified by distillation in vacuo ( $160^\circ\text{C}/5$  torr) and condensed with malonic acid in dry pyridine (**29**). The product, 4-alkyloxycinnamic acid (**4**) was purified by repeated recrystallisation from ethanol. White crystals were obtained having with a melting point of  $149^\circ\text{C}$ .

$^1\text{H}$ -NMR of compound **4**,  $\text{CDCl}_3$ , 300 MHz: 7.74d (1H,  $=\text{CHAr}$ ); 7.50d (2H, meta to  $-\text{OR}$ ); 6.91d (2H, ortho to  $-\text{OR}$ ); 6.32d (1H,  $=\text{CH}-\text{COO}$ ); 4.5brs (1H,  $\text{COOH}$ ); 4.0t (2H,  $\text{CH}_2\text{OAr}$ ); 1.80q (2H,  $\text{OCH}_2\text{CH}_2$ ); 1.2–1.55m (8H,  $\text{CH}_2$ ); 0.91t (3H,  $\text{CH}_3$ ).

### 2.1.4 Synthesis of final product 7SZL

The final product **7SZL** was prepared by standard method of esterification with dicyclohexylcarbodiimid (DCC) in the presence of dimethylaminopyridine (DMAP) in dichloromethane. The crude product was purified by column chromatography on silica gel using a mixture (99.5:0.5) of dichloromethane and acetone as an eluent and crystallised twice from ethanol. The structure of the final product was confirmed by  $^1\text{H}$ -NMR (300 MHz, Varian). The chemical purity of material was checked by high-performance liquid chromatography (HPLC), which was carried out with an Ecom HPLC chromatograph using a silica

gel column (Separon 7  $\mu\text{m}$ ,  $3 \times 150$ , Tessek) with a mixture of 99.9% of toluene and 0.1% of methanol as an eluent, and detection of the eluting products by a ultraviolet–visible (UV–Vis) detector ( $\lambda = 290$  nm). The chemical purity was found to be better than 99% under these conditions.

$^1\text{H}$ -NMR of **7SZL**,  $\text{CDCl}_3$ , 300 MHz: 8.15d (2H, ortho to  $-\text{COO}$ ); 7.86d (1H,  $\text{ArCH}=\text{}$ ); 7.68dd (4H, ortho to  $-\text{Ar}$ ); 7.55d (2H, ortho to  $-\text{CH}=\text{}$ ); 7.30d (2H, ortho to  $-\text{OCO}$ ); 6.94d (2H ortho to  $-\text{OR}$ ); 6.52d (1H,  $=\text{CHCOO}$ ); 5.40q (1H,  $\text{COOCH}^*$ ); 4.03m (4H,  $\text{CH}_2\text{O}$ ); 1.7d (3H,  $\text{CH}_3\text{CHCOO}$ ); 1.2–1.9m (13H,  $\text{CH}_2, \text{CH}$ ); 0.9m (9H,  $\text{CH}_3$ ).

## 2.2 Physical and chemical investigations

The phase transition temperatures have been evaluated by differential scanning calorimetry (DSC) and polarising optical microscopy (POM). For DSC the small amount of substance (about 2–5 mg) was hermetically closed in the aluminium pans and placed in the nitrogen atmosphere of a calorimeter (Perkin–Elmer Pyris Diamond). The cooling and heating rate of  $5 \text{ K min}^{-1}$  was applied. The phases have been identified from POM texture observations carried out in cells with planar and homeotropic alignment (3 or 6  $\mu\text{m}$  thick) using polarising microscopes (Nikon Eclipse or LOMO P112). We prepared cells from glass plates with transparent ITO electrodes with area of  $5 \times 5 \text{ mm}^2$ . The glasses were glued together with Mylar sheets as a spacer. The cells were filled in the isotropic phase. Temperature was stabilised with an accuracy of  $\pm 0.1^\circ\text{C}$  in the hot stage (Linkam) placed on the table of a polarising microscope.

Photochemical investigations were performed using a special optical setup equipped with DRSh-250 ultra-high-pressure mercury lamp. Light at a wavelength of 365 nm was selected by a filter. A water filter was used to prevent heating of the samples due to the infrared (IR) irradiation of the lamp. To obtain the plane–parallel light beam, a quartz lens was applied. During the irradiation, the constant temperature of the test samples was maintained using a Mettler FP-80 hot stage. The intensity of light was measured by a LaserMate-Q (Coherent) intensity meter.

Circularly polarised spectra of samples were studied with a TIDAS spectrometer (J&M) equipped with rotating polariser (Glan–Taylor prism controlled by a computer) and broad-band quarter wave plate.

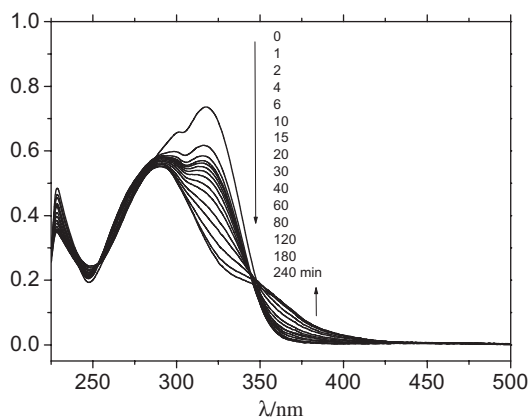
The spontaneous polarisation was measured using the very well-known polarisation reversal currents integration method (*30*) that was modified (*31*) especially for ferroelectric liquid crystals to distinguish clearly between induced and spontaneous polarisation.

### 3. Results and discussion

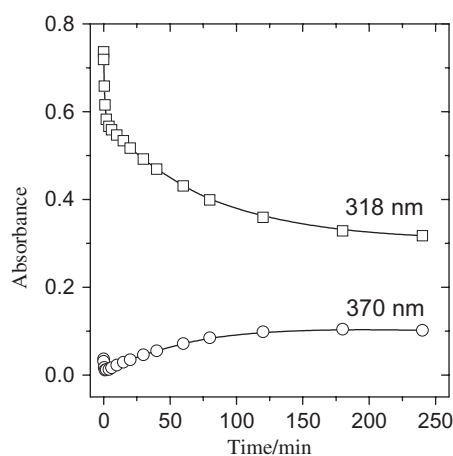
#### 3.1 Photochemical behaviour in solution

To obtain primary information on the possible photochemical reaction we first studied changes in the electronic spectra of **7SZL** in a dilute solution. In Figure 1(a), the time evolution of spectra taken after irradiation by UV light (365 nm) are shown for dilute solutions of LC substance **7SZL** in dichloromethane. From these spectra, the kinetics of the changes was evaluated for two selected wavelengths (see Figure 1(b)).

Analysis of these spectra and their comparison with those shown in (10) allow us to conclude that at least two processes, *E-Z* isomerisation and photo-Fries rearrangement, take place simultaneously, the latter being weak. The decrease in absorbance at 318 nm corresponds to overall changes in cinnamoyl chromophore, whereas the increase at 370 nm (see also Figure 1(b))



(a)



(b)

Figure 1. (a) Absorbance spectra changes under ultraviolet irradiation (365 nm,  $1.6 \text{ mW cm}^{-2}$ ) and (b) its kinetics at two wavelengths for a solution of **7SZL** in dichloromethane ( $8.8 \times 10^{-3} \text{ mg ml}^{-1}$ ). Arrows indicate the directions of the absorbance changes.

corresponds to the appearance of a photo-Fries rearrangement product (aromatic ketone; see Scheme 2). The probability of {2+2} photocycloaddition of **7SZL** for such a dilute solution is quite low.

During the process of UV irradiation of the sample by a wavelength of 365 nm in films or concentrated solutions, photocycloaddition mainly occurs (Scheme 2, product (b)). Evidence of this fact can be found from  $^1\text{H-NMR}$  measurements. In  $^1\text{H-NMR}$  spectra signals of protons on C=C double bond (doublets 7.86 and 7.55) after irradiation practically disappeared.

To a smaller extent, degradation of the molecule also occurs during irradiation, which can be also recognised from NMR spectra.

#### 3.2 Mesomorphic properties

The DSC curves show the phase transition temperatures on the second cooling and subsequent heating runs (Figure 2). The phase transition temperatures are determined from the onset of the transitions. On cooling, two phase transitions are clearly seen above crystallisation. On heating, only one peak is seen above the melting point, but as it still in the low temperature phase, which is identified on cooling, it is not monotropic. It exists within 3 K above the melting point, which is confirmed by observation with a polarising microscope. A low peak connected with the transition to this phase is overwhelmed by the high melting peak. In addition, a high-temperature transition exists, which is seen on both cooling and heating runs as a broad bump (see the inset in Figure 2). Observation in the polarising microscope has shown that this bump

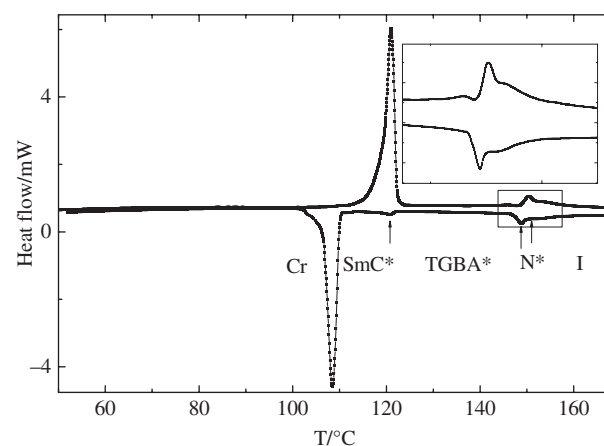
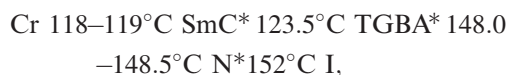


Figure 2. Differential scanning calorimetry curves taken for **7SZL** obtained during the second heating and cooling runs (the upper and lower curve, respectively) at a rate of  $5 \text{ K min}^{-1}$ . The phases are indicated, and the arrows show the phase transitions. In the inset the part within the demarked rectangle is enlarged in the vicinity of the clearing point.



corresponds to the phase transition from the isotropic phase and is accompanied with a rather broad coexistence of phases. The sequence of phases and phase transitions evaluated at heating were



and at cooling were



The SmC\* phase is easily overcooled by about 10 K below the melting point at 117°C.

The phases have been assigned by texture observation on samples in cells with homeotropic and planar alignment conditions (molecules are preferentially perpendicular and parallel to the sample plane, respectively), as shown in Figure 3. The studied compound **7SZL** exhibits an unusual tendency to form homeotropic orientation in samples placed between glass plates without any surface treatment. On the opposite, rubbing of polyimide-coated glasses was applied to achieve the planar orientation. Also a treatment with an electric field induces the planar orientation, which might slowly relax to the homeotropic orientation after the field is switched off. The textures in all phases

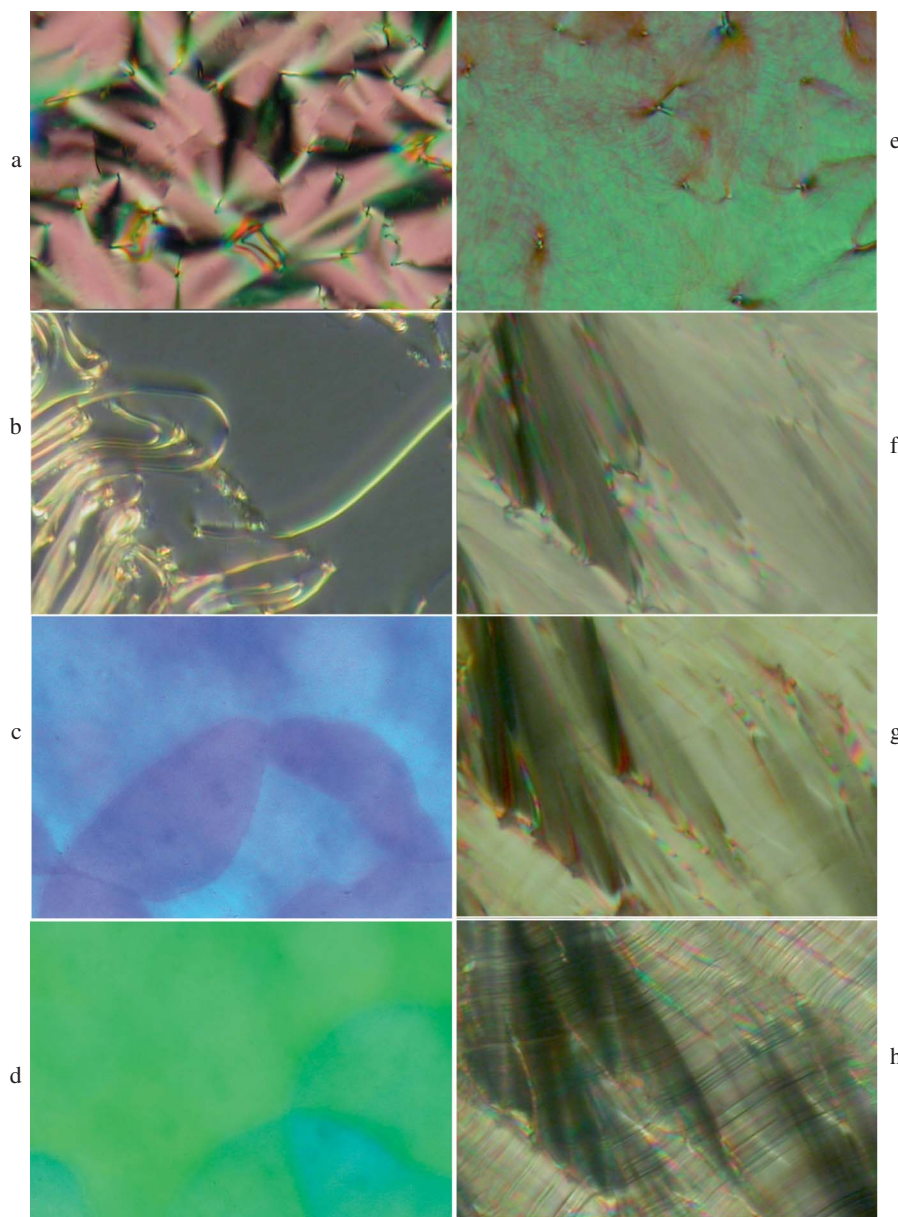


Figure 3. Textures observed in cells with (a)–(d) homeotropic and (e)–(h) planar alignment; (a), (e) N\* at  $T = 151^\circ\text{C}$ ; (b), (f) TGBA\* at  $147^\circ\text{C}$ ; (c), (g), (h) SmC\* at  $120^\circ\text{C}$ ; (d) SmC\* at  $113^\circ\text{C}$ ; (h)  $E = 0.5 \text{ V } \mu\text{m}^{-1}$ .

for both planar as well as homeotropic structures exhibit typical features so that the assignment of phases is unequivocal (see Figure 3). Below the cholesteric phase (Figures 3(a) and (c)) there is the TGBA\* phase, which is recognised principally by typical filaments in the homeotropic structure (Figure 3(b)). In the SmC\* phase with homeotropic orientation, selective reflection of visible light is observed which is associated with the helical structure. The colours of selective light reflection are varied with changing temperature, which is a consequence of the change of the helical pitch magnitude (Figures 3(c) and (d)). In the SmC\* phase with planar orientation, the typical fan-shaped texture occurs, and the dechiralisation lines spaced by the pitch are not seen (Figure 3(g)). This confirms the unusually short pitch of the helical structure. When a weak electric field is applied the pitch becomes longer and thus the dechiralisation lines appear (Figure 3(h)).

### 3.3 Phase diagram under irradiation

The effect of UV irradiation on the mesomorphic properties of 7SZL has been studied in several equivalent cells with the same thickness of 10  $\mu\text{m}$ . The cells have been irradiated at 125°C (in the TGBA\* phase) for different periods of time and subsequently the phases and phase transition temperatures have been determined by observation with a polarising optical microscope. Figure 4 shows the phase diagram of 7SZL against the irradiation time. One can see the UV irradiation up to about 100 minutes results in a significant decrease in SmC\*–TGBA\* and TGBA\*–N\* phase transition temperatures, while longer irradiation has a less dramatic impact. The melting transition is

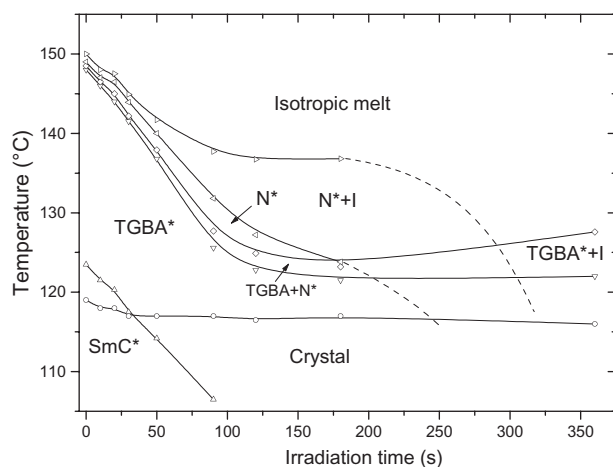


Figure 4. Changes in the phase transitions temperatures of 7SZL under ultraviolet irradiation (365 nm, 1.5 mW cm<sup>-2</sup>). Data obtained using polarising optical microscopy under heating.

changed only slightly. After about 25 s of irradiation, SmC\* becomes monotropic, i.e. the SmA\*–SmC\* transition temperature detected on cooling shifts below the melting point. All of the observed phase transitions are of the first order, which implies a coexistence of phases at these phase transitions (see Figure 4). From about 50 s of irradiation the biphasic (coexistence) region of the N\* and isotropic phases becomes extremely large.

The above-mentioned changes are associated with photoreactions leading to the formation of photoproducts with a low anisotropy (Scheme 2). It should be emphasised that in contrast to the dilute solution of 7SZL, in its liquid crystalline state the dominant process is {2+2} photocycloaddition because the probability of bimolecular reactions in this state is very high due to direct contacts.

### 3.4 Selective reflection in the SmC\* phase

The SmC\* phase of this substance exhibits selective light reflection in the visible spectral range, which is evident from the measurements of spectral transmittance of circularly polarised light on homeotropically oriented films. Spectra corresponding to the transmittance of the left-handed circularly polarised light show a well-developed peak (see Figure 5(a)), which indicates the light reflection. For the right-handed light, no peak is detected. This means namely that the left-handed helical structure is formed in the whole temperature range of the SmC\* phase.

The wavelength of the selective reflection maximum is highly dependent on temperature (Figure 5(b)). An increase in the temperature by 15°C leads to a shift of selective the reflection band position from the blue to the red spectral region. This phenomenon reflects an increase of the pitch of the helical structure under heating. Similar behaviour was found in the papers devoted to the study of optics of the SmC\* phase (32–37).

To assess the effect of UV irradiation on the helical structure in the SmC\* phase the transmission spectra have been detected on samples irradiated for various time periods. It was found that the peak corresponding to the selective reflection is shifted to longer wavelengths (Figure 6(a)) with prolongation of the irradiation, showing gradual untwisting of the helix. In addition a decrease in the peak intensity occurs with irradiation. After irradiation for several tens of seconds (less than one minute) the peak of selective light reflection disappears completely. Investigation of the kinetics of this process at different temperatures (Figure 6(b)) showed that a more significant shift is observed in the slightly overcooled SmC\* phase (at 116°C) and is almost absent at high temperatures.



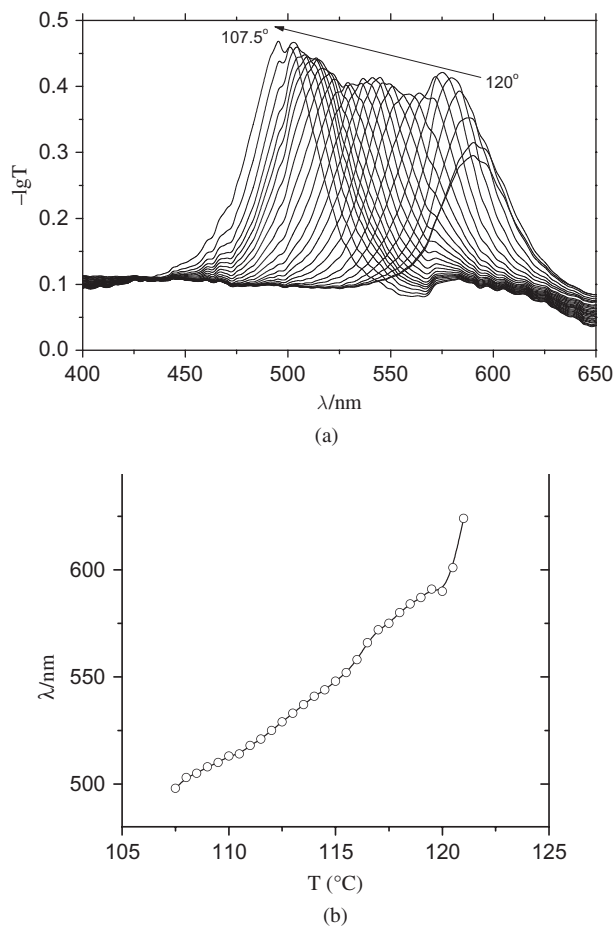


Figure 5. (a) Selective light reflection spectra of **7SZL** in SmC\* measured under slow cooling ( $1^{\circ}\text{C min}^{-1}$ ). Spectra (logarithm of transmittance for left-handed circularly polarized light) were recorded each  $0.5^{\circ}\text{C}$ . (b) Temperature dependence of selective light reflection of **7SZL** in SmC\* (determined as maxima of the curves in (a)).

The colour of the films changes from green to red in the latter case.

The helix untwisting in the SmC\* phase might be related mostly to the decrease of the SmC\*–SmA\* transition temperature under UV irradiation that is associated with the formation of non-mesogenic photoproducts (Scheme 2). A decrease in the selective light reflection wavelength at the final stages of irradiation (after 30 s; Figure 6(b)) is probably associated with the coexistence of two phases. Another possible explanation of the helix untwisting is the decrease in helical twisting power of the chiral fragment due to the decrease of its anisometry, which was frequently observed for chiral-photochromic cinnamoyl- and azobenzene-containing dopants (2).

Figure 7 shows the temperature dependence of spontaneous polarisation before and after UV irradiation. One can see clearly that the spontaneous polarisation

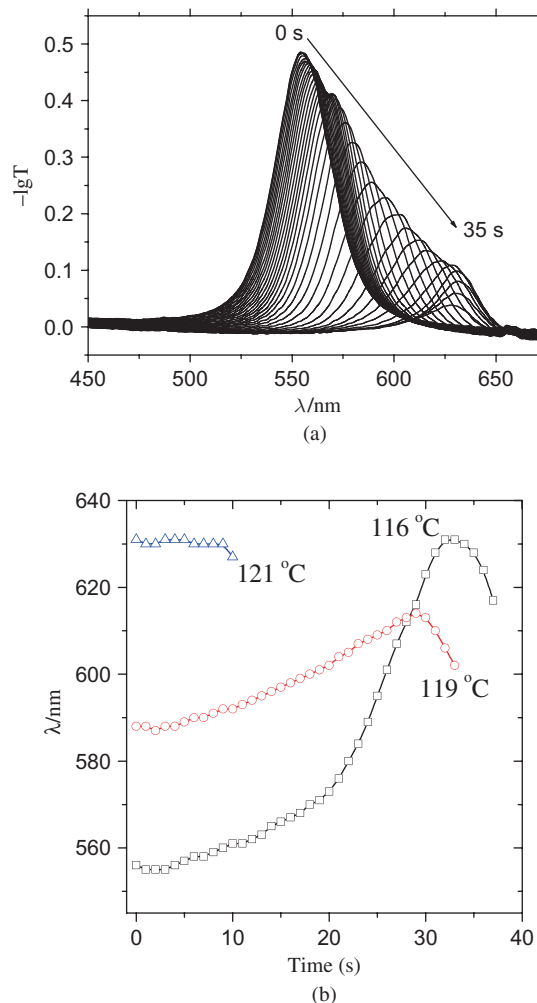


Figure 6. (a) Shift of the selective light reflection peak of **7SZL** during irradiation by ultraviolet light ( $365\text{ nm}$ ,  $1.5\text{ mW cm}^{-2}$ ), spectra were recorded every 1 s of irradiation. Temperature of the sample  $116^{\circ}\text{C}$ , cell thickness  $10\text{ }\mu\text{m}$ . (b) Kinetics of the selective light reflection shift at different temperatures.

magnitude decreases after irradiation due to the photo-processes resulting in the formation of non-mesogenic derivatives.

#### 4. Conclusion

In conclusion, for the first time the helix untwisting induced by UV irradiation has been studied for a LC substance forming the SmC\* mesophase. It has been demonstrated that the cinnamoyl-containing compound has important advantages in comparison with most studied azobenzene derivatives: photoprocesses leading to changes in molecular anisometry are completely thermally irreversible, which provides better experimental conditions for the study of phase transitions. The novel synthesised and investigated compound

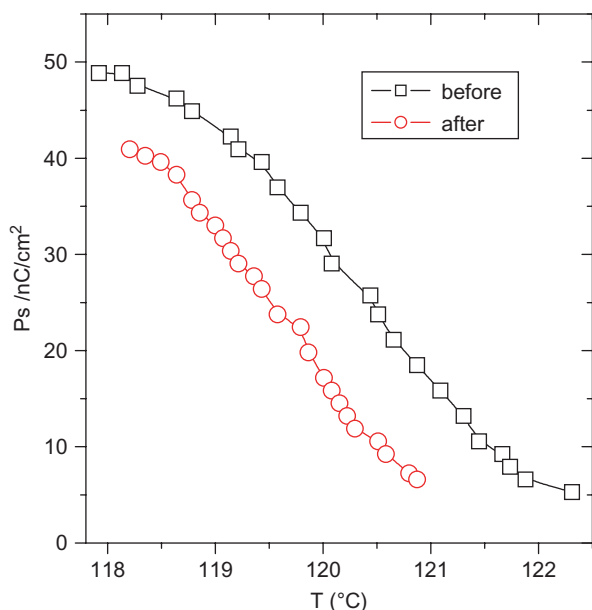


Figure 7. Temperature dependence of spontaneous polarization before and after ultraviolet irradiation that was performed with a light-emitting diode; its wavelength is 365 nm, the light power density is about  $0.01 \text{ mW cm}^{-2}$ , and the exposure time is 10 min.

**7SZL** has good potential for application in photo-, chiro- and electrooptical switches and photorecording media.

#### Acknowledgment

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