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(*S*)-Isoleucine and (*R*)-2-octanol as chiral precursors of new chiral liquid crystalline thiadiazoles: synthesis, mesomorphic and ferroelectric properties

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New chiral Schiff bases with a 1,3,4-thiadiazole unit in the rigid core were synthesized (**SB1**–**SB4**). These compounds contain a chiral chain derived from (*S*)-isoleucine and (*R*)-2-octanol. Their liquid crystalline properties were studied by polarizing optical microscopy, differential scanning calorimetry and X-ray diffraction. Thereby it was found that most of the new compounds exhibit a chiral Smectic C phase. A study of the ferroelectric properties is described.

1. Introduction

It is well-known that mesogens made from optically active molecules can form chiral mesophases showing ferroelectricity [1]. Ferroelectric liquid crystals (FCLs) have attracted considerable interest due to their unique properties and their potential technical applications [2–4]. Before the present work, research has been carried out on mesogens incorporating a thiadiazole ring [5–7], because it produces wide smectic C (SmC) ranges. The incorporation of a thiadiazole moiety in a mesogen structure is conducive to the formation of smectic phases, in particular the SmC phase. More recently, we have reported the mesomorphic and ferroelectric properties of chiral Schiff bases containing the 1,3,4-thiadiazole ring in their rigid core, in which one terminal group is a chiral ester chain derived from (*S*)-ethyl lactate [8].

In a continuation of our work on heterocyclic mesogens, we now describe the synthesis, and mesomorphic and ferroelectric properties of news Schiff bases incorporating the 1,3,4-thiadiazole moiety. In order to achieve the non-centrosymmetry in the SmC phase neccesary for ferroelectric behaviour, one of the terminal groups is a chiral ester chain or a chiral alkoxy chain derived from (*S*)-isoleucine and (*R*)-2-octanol. The other terminal substituent is an alkoxy group with the number of carbon atoms kept constant at $n=10$. The structures of these Schiff bases (**SB1**–**SB4**) are shown in figure 1.

The main aim of this work was to study the effect of the nature of the chiral chain on the mesomorphic properties of the chiral Schiff bases. The ester chains used in the previous study (derived from (*S*)-ethyl lactate) [8] have been replaced by either alkoxy or ester chains derived from (*S*)-isoleucine and (*R*)-2-octanol. As a result, the mesomorphic behaviour strongly depends on the nature of the chiral chain in the molecule.

2. Synthesis

This study involved the synthesis and characterization of new chiral Schiff bases containing the thiadiazole ring in the rigid core (see figure 1) and the comparison of these compounds with the analogous Schiff bases previously reported [8]. These compounds were obtained by reaction of the 5-(4-*n*-decyloxy)phenyl-2-amino-1,3,4-thiadiazole with the corresponding chiral benzaldehydes. The alcohols were chosen as chiral precursors were: (2*S*, 3*S*)-2-chloro-3-methylpentanol and (*R*)-2-octanol. The latter was purchased from Merck while the former was prepared using (*S*)-isoleucine as chiral precursor. The synthesis of all the compounds is outlined in schemes 1 and 2.

The chiral alcohol (2*S*, 3*S*)-2-chloro-3-methylpentanol was obtained by diazotization of (*S*)-isoleucine with sodium nitrite and HCl followed by reduction

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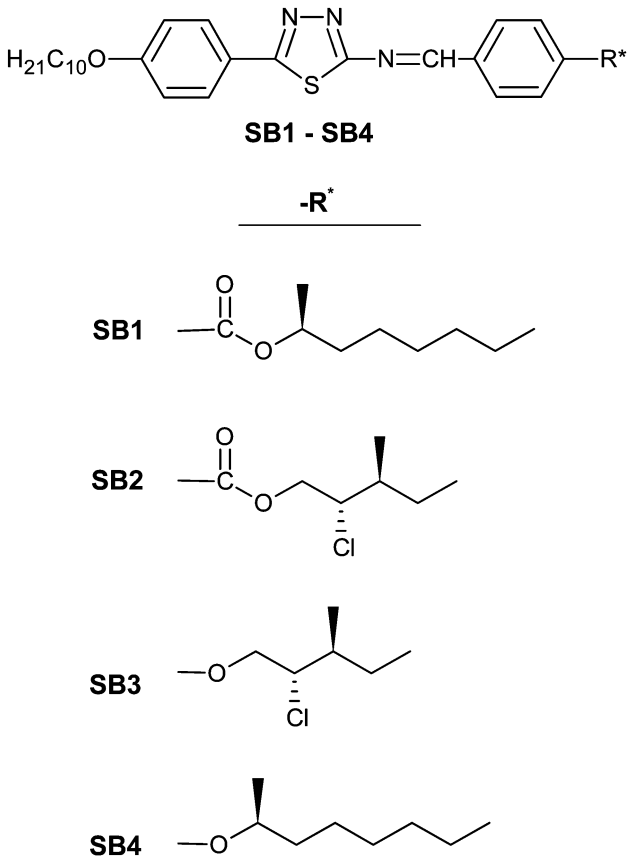


Figure 1. Structures of the chiral compounds **SB1**–**SB4**.

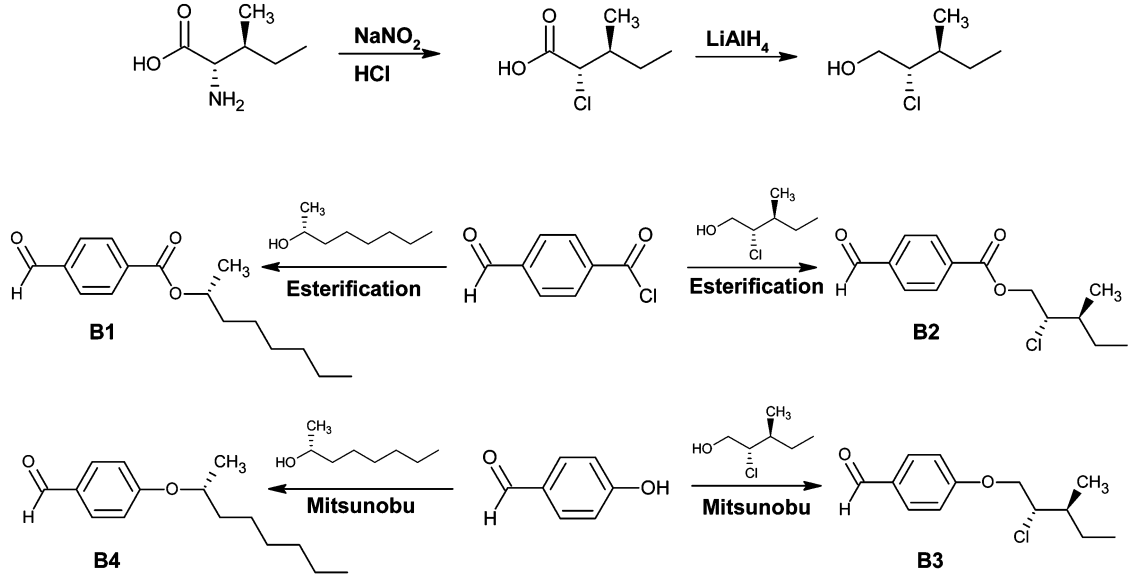
of the acid group with $LiAlH_4$ [9–11]. Compounds **B1** and **B2** were obtained by reaction of 4-formylbenzoic acid chloride with (*R*)-2-octanol and (2*S*, 3*S*)-2-chloro-methylpentanol respectively [5, 6]. The synthesis of the chiral 4-alkoxybenzaldehydes (**B3** and **B4**) was achieved by the Mitsunobu reaction [12] starting from 4-hydroxybenzaldehyde and the corresponding chiral alcohols. The aminothiadiazole derivative and Schiff bases (**SB1**–**SB4**) were synthesized according to methods previously described [5–8].

3. Results and discussion

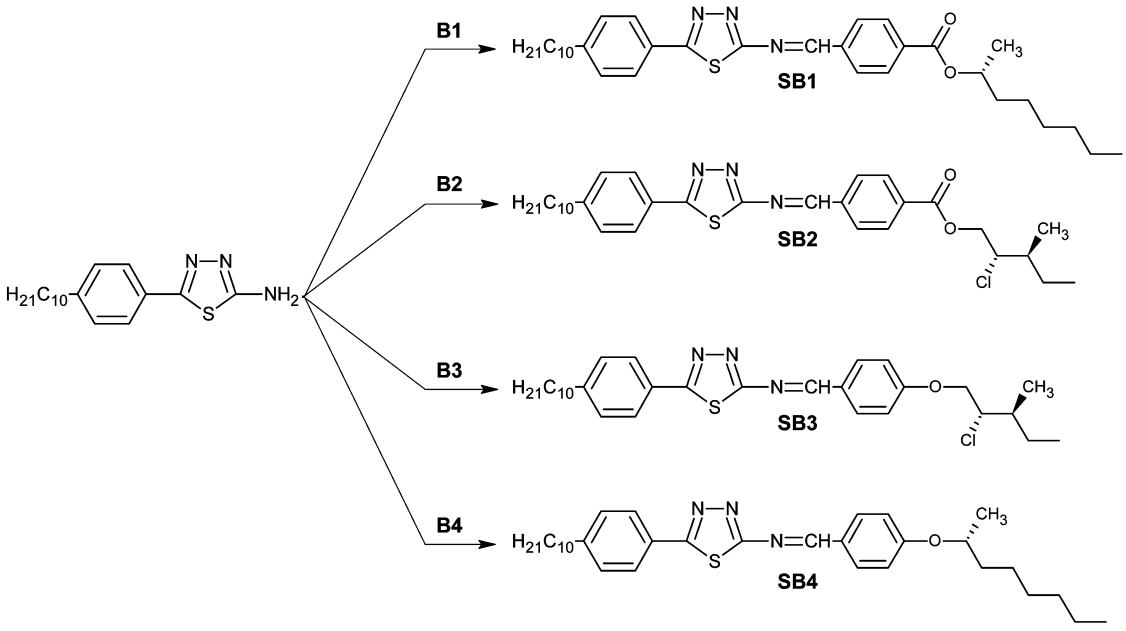
3.1. *Mesomorphic properties*

The transition temperatures and phase behaviour of the new materials are given in table 1; a graphical representation of the mesomorphic behaviour is presented in figure 2.

A part from the Schiff base containing an ester group derived from (*S*)-isoleucine (**SB2**), all the compounds display mesomorphic properties. Enantiotropic phase sequences are observed for compounds **SB1** (Cr–SmC*–I), **SB3** (Cr–SmC*–I) and **SB4** (Cr–SmC*–N*–I). Compound **SB2** does not show mesomorphism, exhibiting only a Cr–I transition. Compounds **SB1**–**SB4** have the same central rigid core and the same achiral alkoxy chain. The difference between them is in the terminal chiral chain. Compounds containing a chiral ester chain (**SB1**, **SB2**) have higher melting points than those containing a chiral alkoxy chain (**SB3**, **SB4**). Schiff bases **SB3** and **SB4** have higher mesomorphic temperature ranges (48 and 31°C, respectively) than compound **SB1**. Thus mesomorphic behaviour strongly depends on the nature of the chiral chain in the molecule. The chiral ester derivatives (**SB1**, **SB2**) show much poorer mesomorphism.



Scheme 1. Synthetic route for chiral precursors **B1**–**B4**.



Scheme 2. Synthetic route for chiral Schiff bases **SB1**–**SB4**.

It is noticeable that the effect of the branch in the stereogenic centre is quite different for ester chains than for alkoxy chains. Lateral interactions between the polarizable nuclei must be much more hindered in the ester derivatives (**SB1**, **SB2**), especially in compund **SB2**. In contrast, the stereogenic centre appears to be less harmful to the lateral interactions of molecules within the mesophase in derivatives with a chiral alkoxy chains. It is also worth noting that a short range chiral nematic phase appears in the compound **SB4** (approximately 3°C). These results show that the chiral alkoxy chain favours the SmC* phase, indicating that the lateral dipolar interactions associated with terminal chiral chain dipoles dominate phase structure in a SmC* liquid crystal [13, 14].

As mentioned above, previously we have reported FLCs with a thiadiazole ring in the mesogenic core [8]. These compounds had the same central rigid core and the same achiral alkoxy chain (decyloxy) as those of the compounds **SB1**–**SB4**. The main difference is in the chiral terminal chains. The former have a chiral chain at the end of the rigid core derived from chiral ethyl lactate, whereas the latter have a shorter chiral chain derived from chiral isoleucine and chiral 2-octanol. Those compounds reported earlier [8] have higher SmC* temperature ranges than the Schiff bases reported here. From these results we conclude that the SmC* phase is more favoured with a chiral chain derived from (*S*)-ethyl lactate.

3.2. *Textures observed by polarizing optical microscopy*

The SmC* and N* phases were determined from textural observations by thermal microscopy with a polarizing microscope under heating and cooling conditions. Phase transition temperatures observed through thermal microscopy were found to be in reasonable agreement with the corresponding DSC thermograms.

The chiral nematic phase of compound **SB4** was characterized by formation of the oily-streaks texture characteristic of the cholesteric phase [15]. The chiral smectic C phase showed a characteristic broken focal-conic texture with dechiralization lines [16, 17].

3.3. *X-ray diffraction*

The mesomorphic Schiff bases (**SB1**, **SB3** and **SB4**) were studied by X-ray diffraction (XRD) in their liquid crystal phases. In the three cases, the patterns were characteristic of smectic mesophases: a sharp, strong maximum at small angles, characteristic of the layer periodicity, and a diffuse halo at large angles, characteristic of short range correlations within the layers. Applying Bragg's law, the layer spacing is deduced from the small angle maximum (table 2), whereas the large angle halo allows an estimation of the mean intermolecular distance. This distance is about 4.5 Å, a value typical in calamitic mesophases. The broadness and diffuseness of this maximum exclude the existence of long range order within the layers, which is consistent

Table 1. Transition temperatures and enthalpies of the chiral Schiff bases **SB1**–**SB4**. Cr=crystal, SmC*=chiral smectic C, N*=chiral nematic, I=isotropic.

Compound	Transition	Temperature/°C	ΔH/J g^{-1}
SB1	Cr–SmC*	100.1	33.9
	SmC*–I	124.6	4.2
SB2	Cr–I	105.2	40.1
SB3	Cr–SmC*	92.5	39.1
	SmC*–I	140.0	5.3
SB4	Cr–SmC*	81.5	35.8
	SmC*–N*	108.9	3.5
	N*–I	112.0[a]	—

[a]Optical microscopy data.

with a SmC mesophase and allows us to rule out other more organized smectic mesophases. At first sight this kind of pattern would also be consistent with a SmA mesophase; however, the SmC nature of the mesophase is confirmed on the basis of the microscope textures and the ferroelectric properties (see later). Moreover the experimentally measured layer spacings (d) are in all cases significantly smaller than the molecular lengths in the most extended conformation estimated using Dreiding stereomodels (L, table 2), suggesting that the molecules are tilted. As expected, d decreases as L decreases, the d/L ratio being about 0.7. No conclusions about the tilt angle can be drawn from this ratio because the conformational disorder of the hydrocarbon chains, and not only the tilt angle, contributes to decrease in d.

For **SB4**, variable-temperature measurements were performed, and the d-layer spacing was found to be independent of temperature within the experimental error (table 2). At the same time, these experiments confirmed the nematic nature of the high temperature mesophase of this compound. In this mesophase, the small angle reflection that is strong and sharp in the SmC mesophase transforms into a diffuse maximum located approximately at the same position. This kind of small angle diffuse peak is characteristic of a nematic mesophase and corresponds to a short range density wave arising from smectic fluctuations, the length of which (d for the nematic mesophase of **SB4** in table 2) is roughly equivalent to the d-layer spacing of the smectic mesophase.

3.4. *Ferroelectric properties*

The mesomorphic Schiff bases (**SB1**, **SB3** and **SB4**) were observed under electric fields in 5 μm ITO/

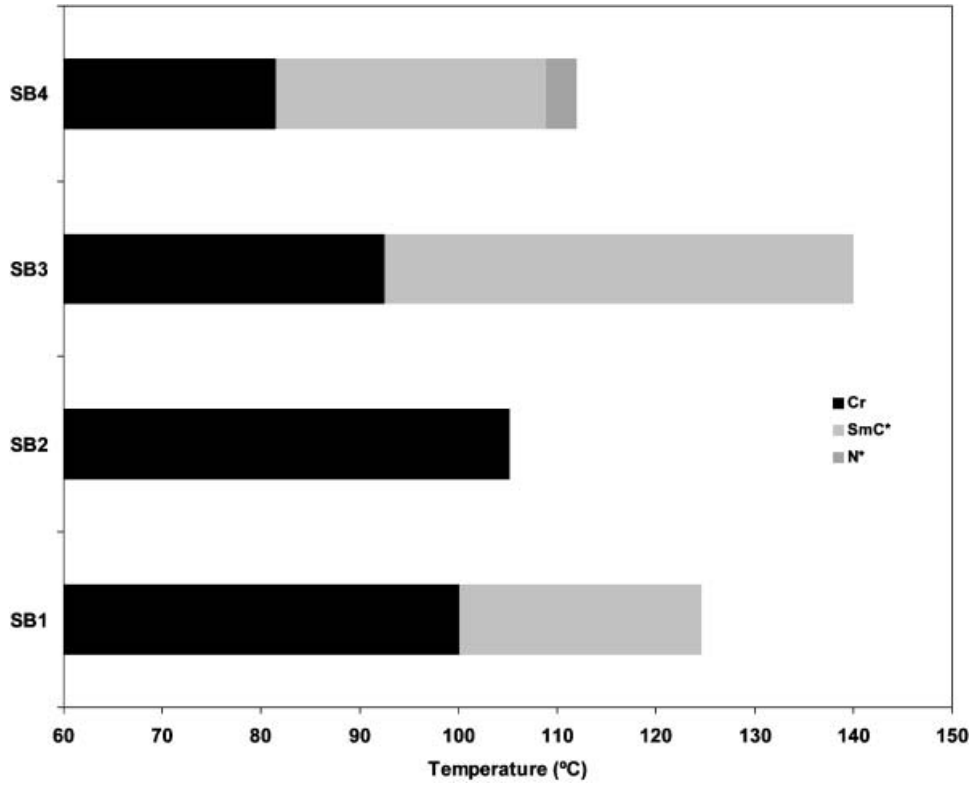


Figure 2. Plots of the mesomorphic behaviour of compounds **SB1**–**SB4**.

Table 2. Results of XRD on the mesophases of compounds **SB1**, **SB3** and **SB4**. Parameter *d* represents the layer spacing in the SmC mesophase or the wavelength of the smectic fluctuations in the nematic mesophase (see discussion in the text). Parameter *L* is the molecular length estimated from Dreiding stereomodels for the most extended conformation.

Compound	Temperature/°C	Phase	*d*/Å	*L*/Å
SB1	108	SmC	31.8	41
SB3	108	SmC	25.7	37
SB4	83	SmC	26.4	39.5
	105.5	SmC	26.4	
	108	SmC	26.4	
	109.5	N	~27[a]	
	111	N	~27[a]	

[a]Diffuse maximum.

polyimide-coated cells (Linkam), in which aligned samples were obtained. Ferroelectric switching was detected for all the mesomorphic compounds, when a voltage of 70 V_{pp} was applied. Unfortunately, these compounds decomposed on the application of electric fields, probably due to the presence of the imine group. The appearance of a current peak due to inversion of the spontaneous polarization was short-lived, so accurate measurement of $\mathbf{P}_s$ values was not carried out. Similar results were obtained for the FLCs reported earlier [8].

4. Experimental

4.1. *Characterization*

The structures of the compounds were confirmed by ^{1}H NMR, ^{13}C NMR (Bruker AC-250P) and FTIR (Nicolet 550) spectra. The purity of final products was evaluated by thin layer chromatography.

Transition temperatures and textures of mesophases were determined by optical microscopy using an Ortholux Pol BK-11 polarizing microscope equipped with a Mettler FP 800 hot stage. Transition temperatures and enthalpies were investigated by differential scanning calorimetry using a Rheometric DSC-V calorimeter. Samples were encapsulated in aluminium pans and observed at a scanning rate of 5°C min^{-1} during heating and cooling. The instrument was calibrated using an indium standard (156.6°C, 28.44 J g^{-1}).

XRD patterns were obtained with a pinhole camera (Antoon–Paar) operating with a point-focused Ni-filtered Cu-K_α beam. The samples were held in Lindemann glass capillaries (1 mm diameter) and heated with a variable temperature oven. The patterns were collected on flat photographic films. The capillary axis and the film were perpendicular to the X-ray beam; spacings were obtained via Bragg's law.

The cells used for ferroelectric measurements were coated with polyimide and carried indium tin oxide (ITO) electrodes.

4.2. *Synthesis of intermediates and products*

(*S*)-isoleucine and (*R*)-2-octanol were purchased from Merck. The organic solvents were of analytical grade quality and all were dried by traditional methods. Analitycal thin layer chromatography (TLC) was conducted on Merck aluminum plates with 0.2 mm of silica gel 60 F-254.

The chiral alcohol (2*S*, 3*S*)-2-chloro-3-methylpentanol was synthesized using (*S*)-isoleucine, according to the previously described method [9–11]. The 5-(4-*n*-decyloxy)phenyl-2-amino-1,3,4-thiadiazole was synthesized as in [5, 8].

4.2.1. Optically active (1-methyl)heptyl-4-formylbenzoate (B1) and (2-chloro-3-methyl)pentyl-4-formylbenzoate (B2). These compounds were synthesized using the procedure described previously [7, 8]. Products were obtained in liquid form, and were purified by chromatography using 1/1 *n*-hexane/dichloromethane as eluant. Yields: **B1** (75%), **B2** (54%).

B1: ^{1}H NMR ($CDCl_3$, TMS, 250 MHz): δ ppm = 0.82 (t, J = 6.72 Hz, 3H, CH_3); 1.30 (d, J = 6.20 Hz, 3H of the methyl branch); 1.20–1.70 (m, 10H, 5 CH_2); 5.14 (m, 1H, CH of the chiral chain); 7.89 (d, J = 8.35 Hz, 2H, 2 arom. H); 8.13 (d, J = 8.31 Hz, 2H, 2 arom. H); 10.04 (s, 1H, CHO). ^{13}C NMR ($CDCl_3$, TMS, 62.9 MHz): δ ppm = 14.0 (CH_3); 19.9 (methyl branch); 22.5, 25.3, 29.1, 31.6, 31.9 (aliph. C); 72.6 (CH of the chiral chain); 129.3, 129.4 (arom. C); 138.9, 144.2 (quaternary arom. C); 165.0 (C=O); 191.5 (O=C<u>H</u>). IR (film): cm^{-1} = 2927 (Csp^3–H); 1724 (C=O); 1602 (C=C).

B2: ^{1}H NMR ($CDCl_3$, TMS, 250 MHz): δ ppm = 0.90 (t, J = 7.40 Hz, 3H, CH_3); 1.06 (d, J = 6.70 Hz, 3H of the methyl branch); 1.36–1.50 (m, 2H, CH_2); 2.15 (m, 1H, *C<u>H</u>–CH_3); 4.30 (d, J = 7.03 Hz, 1H, *C<u>H</u>–Cl); 4.50 (m, 2H, OCH_2); 7.21 (d, J = 8.43 Hz, 2H, 2 arom. H); 7.85 (d, J = 8.40 Hz, 2H, 2 arom. H); 9.92 (s, 1H, CHO). ^{13}C NMR ($CDCl_3$, TMS, 62.9 MHz): δ ppm = 10.8 (CH_3); 15.9 (methyl branch); 25.0 (CH_2); 38.9 (*<u>C</u>H–CH_3); 62.3 (*<u>C</u>H–Cl); 65.8 (OCH_2); 121.9, 131.2 (arom. C); 134.3, 154.8 (quaternary arom. C); 167.2 (C=O); 190.7 (O=C<u>H</u>). IR (film): cm^{-1} = 2969 (Csp^3–H); 1730 (C=O); 1596 (C=C).

4.2.2. Optically active 4-(2-chloro-3-methyl)pentyloxybenzaldehyde (B3) and 4-(1-methyl)heptyloxybenzaldehyde (B4). These compounds were synthesized using

the procedure described in [12]. Products were obtained in liquid form, and were purified by chromatography using 1/1 *n*-hexane/dichloromethane as eluant. Yields: **B3** (56%), **B4** (92%).

B3: ^{1}H NMR ($CDCl_3$, TMS, 250 MHz): δ ppm=0.92 (t, J=6.32 Hz, 3H, CH_3); 1.05 (d, J=6.79 Hz, 3H of the methyl branch); 1.36–1.67 (m, 2H, CH_2); 1.96 (m, 1H, *C<u>H</u>CH_3); 4.08 (m, 1H, *C<u>H</u>Cl); 4.20 (m, 2H, OCH_2); 6.99 (d, J=8.76 Hz, 2H, 2 arom. H); 7.82 (d, J=8.80 Hz, 2H, 2 arom. H); 9.84 (s, 1H, CHO). ^{13}C NMR ($CDCl_3$, TMS, 62.9 MHz): δ ppm=11.3 (CH_3); 15.8 (methyl branch); 24.4 (CH_2); 38.0 (*<u>C</u>HCH_3); 64.3 (*<u>C</u>HCl); 69.8 (OCH_2); 114.7; 131.8 (arom. C); 130.2, 163.1 (quaternary arom. C); 190.5 (C=O). IR (film): cm^{-1}=2964 (Csp^3–H); 1694 (C=O); 1597 (C=C).

B4: ^{1}H NMR ($CDCl_3$, TMS, 250 MHz): δ ppm=0.82 (t, J=6.77 Hz, 3H, CH_3); 1.30 (d, J=6.20 Hz, 3H of the methyl branch); 1.27–1.70 (m, 10H, 5 CH_2); 5.12 (m, 1H, C<u>H</u> of the chiral centre); 7.89 (d, J=8.15 Hz, 2H, 2 arom. H); 8.13 (d, J=8.13 Hz, 2H, 2 arom. H); 10.04 (s, 1H, CHO). ^{13}C NMR ($CDCl_3$, TMS, 62.9 MHz): δ ppm=13.9 (CH_3); 19.9 (methyl branch); 22.4, 25.3, 29.0, 31.6, 35.8 (aliph. C); 72.5 (chiral C); 129.3, 130.0 (arom. C); 135.8, 138.9 (quaternary arom. C); 191.5 (C=O). IR (film): cm^{-1}=2928 (Csp^3–H); 1692 (C=O); 1598 (C=C).

4.2.3. Optically active Schiff bases (SB1–SB4). These compounds were synthesized by condensation of 5-(4-*n*-decyloxy)phenyl-2-amino-1,3,4-thiadiazole with the corresponding chiral benzaldehyde (**B1**, **B2**, **B3** or **B4**) using the procedure described in [5, 7, 8]. A mixture of 2-amino-thiadiazole derivative (0.31 mmol, 0.1 g) and 1.85 mmol of the corresponding chiral benzaldehyde was heated in an oil bath at 140°C for 1 h. The residue was cooled and crystallized from ethanol with the following yields: **SB1** (48%); **SB2** (25%); **SB3** (45%); **SB4** (55%).

SB1: ^{1}H NMR ($CDCl_3$, TMS, 250 MHz): δ ppm=0.88 (t, J=6.62 Hz, 6H, 2 CH_3); 1.37 (d, J=6.20 Hz, 3H of the methyl branch); 1.27–1.82 (m, 26H, 13 CH_2); 4.00 (t, J=6.48 Hz, 2H, OCH_2); 5.19 (m, 1H, C<u>H</u> of the chiral chain); 6.98 (d, J=8.82 Hz, 2H, 2 arom. H); 7.91 (d, J=8.69 Hz, 2H, 2 arom. H); 8.07 (d, J=8.85 Hz, 2H, 2 arom. H); 8.09 (d, J=8.64 Hz, 2H, 2 arom. H); 9.00 (s, 1H, N=CH). ^{13}C NMR ($CDCl_3$, TMS, 62.9 MHz): δ ppm=14.3 (2 CH_3); 20.0 (methyl branch); 22.6, 25.3, 25.9, 29.1, 29.3, 29.5, 31.7, 31.8, 35.6 (aliph. C); 68.2 (OCH_2); 72.4 (CH of the chiral chain); 114.9, 129.4, 129.8, 129.9 (arom. C); 122.9, 135.2, 137.7, 161.8, 168.1, 172.4 (quaternary arom. C); 165.2 (N=<u>C</u>H); 192.5 (C=O). IR (KBr disk): cm^{-1}=2925 (Csp^3–H); 1714 (C=O); 1606 (C=C); 1560 (N=C); 1258 (C–O).

SB2: ^{1}H NMR ($CDCl_3$, TMS, 250 MHz): δ ppm=0.82 (t, J=6.10 Hz, 3H, CH_3); 0.90 (t, J=7.57 Hz, 3H, CH_3); 1.07 (d, J=6.69 Hz, 3H of the methyl branch); 1.01–1.27 (m, 16H, 8 CH_2); 1.39–1.72 (m, 2H, CH_2 of the chiral chain); 2.68 (m, 1H, *C<u>H</u>–CH_3); 3.94 (t, 2H, OCH_2); 4.32 (m, 1H, *C<u>H</u>–Cl); 4.45 (m, 2H, OCH_2 of the chiral chain); 6.97 (d, J=8.85 Hz, 2H, 2 arom. H); 7.18 (d, J=8.41 Hz, 2H, 2 arom. H); 7.24 (d, J=8.80 Hz, 2H, 2 arom. H); 7.87 (d, J=8.45 Hz, 2H, 2 arom. H); 8.84 (s, 1H, N=CH). ^{13}C NMR ($CDCl_3$, TMS, 62.9 MHz): δ ppm=10.8 (CH_3); 14.0 (CH_3); 15.9 (methyl branch); 22.6, 24.7, 25.0, 26.0, 29.0, 29.2, 29.3, 29.5, 31.8 (aliph. C); 38.9 (*C<u>H</u>–CH_3); 62.4 (*C<u>H</u>–Cl); 68.2 (OCH_2); 115.0, 122.0, 128.7, 131.2 (arom. C); 154.1, 154.8, 161.7, 165.2, 167.3, 173.3 (quaternary arom. C); 167.7 (N=<u>C</u>H); 172.7 (C=O). IR (KBr disk): cm^{-1}=2925 (Csp^3–H); 1720 (C=O); 1605 (C=C); 1558 (N=C); 1255 (C–O).

SB3: ^{1}H NMR ($CDCl_3$, TMS, 250 MHz): δ ppm=0.92 (t, J=6.10 Hz, 6H, 2 CH_3); 1.09 (d, J=6.79 Hz, 3H of the methyl branch); 1.02–2.70 (m, 19H, 9 CH_2 and *C<u>H</u>–CH_3); 3.96 (t, J=6.50 Hz, 2H, OCH_2), 4.23 (m, 3H, OCH_2 of the chiral chain and *C<u>H</u>–Cl); 6.99 (d. J=8.79 Hz, 2H, 2 arom. H); 7.03 (d, J=8.68 Hz, 2H, 2 arom. H); 7.89 (d, J=8.77 Hz, 2H, 2 arom. H); 7.96 (d, J=8.70 Hz, 2H, 2 arom. H); 8.81 (s, 1H, N=CH). ^{13}C NMR ($CDCl_3$, TMS, 62.9 MHz): δ ppm=10.5, 13.1 (2 CH_3); 15.0 (methyl branch); 22.6, 24.5, 25.9, 29.1, 29.2, 29.3, 29.5, 31.8 (aliph. C); 37.1 (*<u>C</u>HCH_3); 63.5 (*<u>C</u>HCl); 67.2; 68.9 (2 OCH_2); 113.9, 114.1, 128.1, 131.3 (arom. C); 123.0, 131.3, 161.5, 165.7, 166.6, 173.2 (quaternary arom. C); 165.0 (N=CH). IR (film): cm^{-1}=2925 (Csp^3–H); 1608 (C=C); 1562 (N=C); 1250 (C–O).

SB4: ^{1}H NMR ($CDCl_3$, TMS, 250 MHz): δ ppm=0.87 (t, J=6.75 Hz, 6H, 2 CH_3); 1.27 (d, J=6.06 Hz, 3H of the methyl branch); 1.27–1.77 (m, 26H, 13 CH_2); 3.99 (t, J=6.58 Hz, 2H, OCH_2), 4.48 (m, 1H, CH of the chiral chain); 6.97 (d, J=8.22 Hz, 2H, 2 arom. H); 7.01 (d, J=8.20 Hz, 2H, 2 arom. H); 7.89 (d, J=8.85 Hz, 2H, 2 arom. H); 7.93 (d, J=8.84 Hz, 2H, 2 arom. H); 8.81 (s, 1H, N=CH). ^{13}C NMR ($CDCl_3$, TMS, 62.9 MHz): δ ppm=14.0, 14.1 (2 CH_3); 16.9 (methyl branch); 22.5, 22.6, 25.3, 25.9, 29.0, 29.1, 29.2, 29.3, 29.5, 31.7, 31.8, 36.2 (aliph. C); 68.2 (OCH_2); 74.2 (CH of the chiral chain); 114.9, 115.8, 129.0, 132.3 (arom. C); 126.2, 130.4, 134.1, 162.4, 165.3, 170.8 (quaternary arom. C); 165.9 (N=CH). IR (film): cm^{-1}=2924 (Csp^3–H); 1607 (C=C); 1560 (N=C); 1253 (C–O).

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