Unique effect of the electric field on a new liquid crystalline lactic acid derivative

Vladimíra Novotná, Milada Glogarová, Miroslav Kašpar, Věra Hamplová, Lubor Lejček and Damian Pociecha

a Institute of Physics, The Czech Academy of Sciences, Na Slovance 2, CZ-182 21 Prague 8, Czech Republic
b Laboratory of Dielectrics and Magnetics, Chemistry Department, Warsaw University, Al. Zwirki i Wigury 101, 02-089 Warsaw, Poland

Synthesis of intermediates

Synthesis of protected 4-(methoxycarbonyloxy)benzoyl chloride (1)
4-Hydroxybenzoic acid (69 g, 0.5 mol) was added to a solution of 1.5 mol sodium hydroxide in 1.5 l water/ice mixture with stirring. Then methyl chloroformate (80 g, 0.85 mol) was slowly added to the solution which was maintained at 0°C. The reaction mixture was stirred for further 4 hrs and then acidified to pH = 4-5 with concentrated HCl. The precipitate was filtered off and recrystallized from ethanol. The yield of white crystals (m.p. 85°C) was 90 g (92 %).
Thionyl chloride (80 ml) was mixed with a 4-methoxycarbonyloxybenzoic acid (40 g, 0.21 mol) and with 1 ml of N,N-dimethylformamide. The solution was refluxed for 2 hrs and then the thionyl chloride was distilled off. The yield of crude acyl chloride (1) was 41 g.

\[^1\text{H}-\text{NMR of 1 (CDCl}_3\text{): 3.95 (3H, s, OCH}_3\text{), 7.37 (2H, d, J=8.9 Hz, H-Ar meta to –COCl), 8.18 (2H, d, J= 8.9 Hz, H-Ar ortho to –COCl).}\]

Synthesis of (S)-n-pentyl 2-(p-hydroxybenzoyloxy)propanoate (2)
The solution of 41 g (0.19 mol) of chloride 1 in 100 ml of CH2Cl2 was slowly added to a solution of 37 g (0.23 mol) of (S)-n-pentyl lactate in 100 ml of dichloromethane and 80 ml of pyridine. The reaction mixture was stirred for one day at a room temperature. The resulting mixture was washed with aqueous solution of HCl (200 ml, 10 %), evaporated to dryness and 100 ml of ethanol and 100 ml of aqueous ammonia (27 %) were added. After the stirring for 1 hour the reaction mixture was poured into water (200 ml), acidified with concentrated acetic acid and extracted by diethyl ether. Organic layer was washed with water, dried over anhydrous sodium sulphate and after evaporation of ether the product was distilled under reduced pressure (b.p. 190 – 195 °C/1 torr). Specific rotation was \([\alpha]_D^{25} = +23.5^\circ\) (c = 0.2, EtOH). The yield was 54 g (69%).

\[^1\text{H}-\text{NMR of 2 (CDCl}_3\text{): 0.88 (3H, t, J= 6 Hz, CH}_3\text{CH}_2\text{), 1.2 -1.3 (4H, m, CH}_2\text{), 1.6 -1.7 (5 H, wide doublet CH}_3\text{C}*\text{H, J=7 Hz, and OCH}_2\text{CH}_2\text{), 4.18 (2H, td, J= 6.6 Hz, J= 3.3 Hz, OCH}_2\text{), 5.29 (1H, q, J=7 Hz, C*H), 6.80 (2H, d, J=8.7 Hz, H-Ar ortho to OH), 7.12 (1H, br, OH), 7.89 (2H, d, J=8.7 Hz, H-Ar meta to OH).}\]
**Textures**

Fig. S1
Planar commercial cell (5 μm) of PHB(S) at T=27°C after the electric field is switched off. The filaments appeared only in the vicinity of the electrode edge (bottom of the figure is not cover by the electrode). The width of every figure is about 250 μm.

Fig. S2
Home-made cell without any surface treatment (5 μm) for PHB(S) at temperature T=28°C, a) under applied electric field of 15 V/μm (left side is without electrode and shows the virgin texture, b) under applied electric field of 5 V/μm and c) after the field is switched off. The width of every figure is about 150 μm.
Fig. S3
Planar texture of PHB(rac) in a commercial cell (5 μm) at the phase transition from the isotropic to the SmA phase on cooling. Nuclei of the SmA phase are in a typical form of “batonnets”. The width of the figure is about 200 μm.

Fig. S4
Planar texture of the binary mixture of PHB(rac) and PHB(S), Mix 1:1, in a commercial cell 5 μm (a) under the applied electric field of 15 V/μm and (b) when the field is switched off. The upper right corner shows the area without electrode and it corresponds to the virgin texture. Temperature is about 31°C and the width of every figure is about 200 μm.
Fig. S5
Contact-probe of PHB(S) on the right side and PHB(rac) on the left side at a) 32°C and b) 30°C. The width of every figure is about 180 μm.