Investigations on ferroelectric liquid crystal by high resolution TEM and solid state $^{13}$C NMR

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\[4-(3)-(S)-methyl-2-(S)-chloropentanoyloxy)]-4'-nonyloxy-biphenyl\] was synthesized as follows.

a. Synthesis of \(4'^-\text{nonyloxy-biphenyl-4-ol}\). An oven-dried three-neck flask was charged with \(4,4'^-\text{Biphenol}\) (18.62 g, 0.1 mol), 1-Bromononane (20.72 g, 0.1 mol) and ethanol (250 ml, 95%). Two necks of the flask were attached to reflux condenser and constant pressure funnel respectively. The compounds were kept heating and stirring until completely dissolved. Dropping the alkaline mixture (0.1 mol KOH and 30 ml, 95% ethanol) when it started to reflux and kept reflux condenser for 8 hours after dripping off. The byproduct was filtered off by hot filtration, and lots of mono-aether separated from the filtrate after cooling. The crude product was recrystallized in benzene for two times, and purified by column chromatography (silica gel, ethyl acetate/petroleum ether 1:8). The product had only one spot when detected with TLC.

b. Synthesis of \([4-(3)-(S)-methyl-2-(S)-chloropentanoyloxy)]-4'-nonyloxy-biphenyl\]. (2S,3S)-3-methyl-2-chloropentanoic acid (2.0 g, 0.0133 mol), \(4'-\text{nonyloxy-biphenyl-4-ol}\) (4.15 g, 0.0133 mol), DCC (2.74 g, 0.0133 mol) and DMAP (0.232 g, 0.0019 mol) were mixed in dichloromethane (24.7 ml) and stirred for about 24 h at room temperature. The precipitate formed was filtered off, and the solution was washed with water and 5% glacial acetic acid for two times, respectively. Then it was evaporated after drying with \(\text{MgSO}_4\) to obtain the crude product. The crude product was decolored with active carbon and then recrystallized in hexane.

c. Characterization of \([4-(3)-(S)-methyl-2-(S)-chloropentanoyloxy)]-4'-nonyloxy-biphenyl\].

\(^1\text{H-NMR (400 MHz, CDCl}_3\): \(\delta=7.547\) (d, \(J=8.8\) Hz, 1H), 7.473 (d, \(J=8.8\) Hz, 1H), 7.153 (d, \(J=8.8\) Hz, 1H), 6.956 (d, \(J=8.8\) Hz, 1H), 4.388 (d, \(J=6.8\) Hz, 1H), 3.987 (t, \(J=13.2\) Hz, 2H), 2.158–2.309 (m, 1H), 1.706–1.860 (m, 2H), 1.390–1.500 (m, 2H), 1.202–1.500 (m, 12H), 1.143 (d, \(J=6.4\) Hz, 3H), 0.990 (t, \(J=14.8\) Hz, 3H), 0.887 (t, \(J=7.0\) Hz, 3H) ppm. \(^1^\text{C-NMR (100 MHz, CDCl}_3\): \(\delta=168.02, 158.88, 149.26, 139.18, 132.47,\)
128.08, 127.15, 121.34, 114.84, 68.11, 62.64, 39.07, 31.87, 29.53, 29.39, 29.27, 26.05, 25.16, 22.66, 15.97, 14.08, 10.87 ppm. MALDI-TOF: m/z (%) = 410 (23), 444 (100), [M^+] 46 (38).