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## Liquid Crystals

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## Liquid crystalline side chain polymers consisting of acrylate and 2(S)-[2(S)-methylbutoxy]propionate

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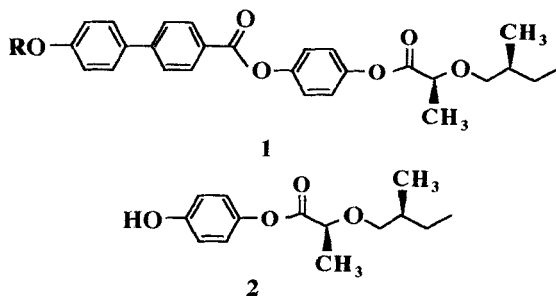
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Besides the usual acrylates, 4-[4-(*n*-acryloxyalkoxy)phenyl]benzoic acids (**4a-4c**), new acrylates containing two units of acrylic acid, 4-{4-[*n*-(3-acryloxypropionyloxy)alkoxy]phenyl}benzoic acid (**5a-5c**), are formed by the esterification of acrylic acid and 4-[4-(*n*-hydroxyalkoxy)phenyl]benzoic acid (**3**). All the mesogenic monomers which were synthesized from the corresponding acrylates (**4a-5c**) and 4-hydroxyphenyl 2(S)-[2(S)-methylbutoxy]propionate (**2**) exhibit the enantiotropic  $S_C^*$  and  $S_A$  phases. The  $S_C^*$  phase temperature range is up to 44°C. The range of spontaneous polarization and response time for those monomers is between 34 and 85 nC cm<sup>-2</sup>, and 55 and 460 μs, respectively. Some liquid crystal side chain polymers containing **2** are switchable in the presence of an electric field. A spontaneous polarization of 72 nC cm<sup>-2</sup> and a response time of 2.6 ms were the best results obtained for those polyacrylates.

### 1. Introduction

The invention of the surface stabilized ferroelectric liquid crystal (SSFLC) light valve in 1980 [1] opened the application epoch of FLCs, which were first described in 1975 [2]. The combination of the properties of liquid crystals with those of polymers is an important progression not only for liquid crystal science but for polymer science. The success of preparing liquid crystal side chain polymers with a smectic C phase [3] is a milestone for realizing FLC side chain polymers. The first FLC side chain polymer was synthesized and characterized by Shibaev *et al.* [4]. The basic and applied research in this field has grown substantially over the last decade [5].

A chiral smectic C ( $S_C^*$ ) unit is the key molecular part of FLC side chain polymers. We have recently synthesized new FLCs **1** in which two centres of chirality were incorporated [6]. Some of them exhibit enantiotropic  $S_C^*$  and  $S_A$  phases. The  $S_C^*$  phase temperature range is broad, from *c.* 75°C to 140°C. The results indicate that the new derivative of the lactate **2** is useful in the formation of the  $S_C^*$  phase. In order to study the



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influence of the chiral molecule **2**, in a polymer, on the formation of liquid crystal phases, some new ferroelectric liquid crystal side chain polymers using polyacrylate as the backbone polymer were prepared and characterized.

## 2. Synthesis

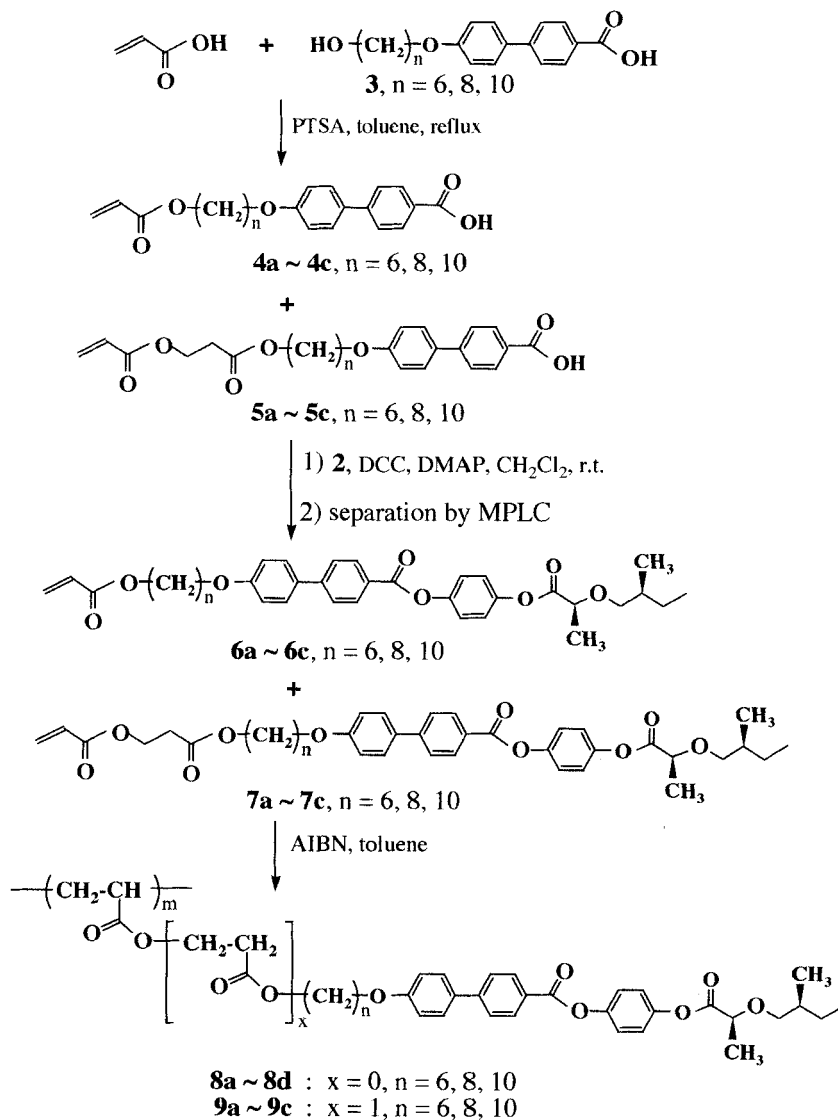
According to our original synthetic plan the monomers **6a–6c** containing the 2(*S*)-[2(*S*)-methylbutoxy]propionyloxy group are the synthetic targets. During the preparation of **4a–4c** from acrylic acid and 4-[4-(*n*-hydroxyalkyloxy)phenyl]benzoic acid (**3**), new acrylates **5a–5c** with the addition of one more acrylic acid were found. The evidence for these addition products can be confirmed from the chemical shift of  $^1\text{H}$  NMR spectra at 2.72 ppm (triplet) and 4.39 ppm (triplet). The amount of **5a–5c** to that of the corresponding **4a–4c** is about one to two, evaluated from the  $^1\text{H}$  NMR spectra. The separation of **4a–4c** from the corresponding **5a–5c** was not attempted due to anticipated difficulty. The successful separation of these two types of acrylates was achieved at the next esterification step. A known esterification condition [7] was used to couple each of the acrylates **4a–5c** and the chiral compound **2** whose detailed synthesis is reported in [6]. The pure acrylates **6a–6c** and the corresponding materials **7a–7c** were isolated by chromatography without any problem. Their structures were identified and confirmed by spectroscopic data. The initiator AIBN was used to polymerize those monomers. The synthetic route is shown in the scheme.

The reaction to prepare **4a–4c** from the acrylic acid and 4-[4-(*n*-hydroxyalkyloxy)-phenyl]benzoic acid (**3**) is a well-known and frequently used chemical procedure [8–14]. However, formation on the acrylates containing two molecules of acrylic acid had never been reported. This is the first time that **5a–5c** have been prepared and identified. The reason for the formation of **5a–5c** is not clear. There are two possibilities: (i) because the purchased acrylic acid contained the dimer adduct in about a one-twelfth amount, the dimerization of the acrylic acid can go further before the esterification in the acidic reaction conditions; (ii) the addition of a second acrylic acid molecule to the corresponding **4a–4c** in these reaction conditions.

## 3. Physical properties

The liquid crystal phases and phase transition temperatures for the monomers **6a–6c** and **7a–7c** were identified by texture observations and differential scanning calorimetry. All the materials exhibit enantiotropic  $S_C^*$  and  $S_A$  phases. The phase sequence  $C-S_C^*-S_A-I$  for these FLCs is identical to that of the FLCs **1** [6]. Even the addition of two molecules of acrylic acid in the side chains has no influence on the liquid crystal phase formation or sequence. However, the  $S_C^*$  phase temperature range becomes gradually narrower, from **1** to **6a–6c**, as well as from **6a–6c** to **7a–7c**. The thermal stability of the  $S_A$  phase is also higher for **6a–6c** than for **7a–7c**.

The spontaneous polarization and the response time were measured at 10°C below the  $S_C^*-S_A$  transition by the triangular wave method [15] and the electric reversal method [16], respectively. A cell, having a 2  $\mu\text{m}$  gap and 0.16  $\text{cm}^2$  active area, was used for the monomers **6a–6c** and **7a–7c**. An electric field of 30 V (peak) and 50 Hz frequency was applied to the cell. Among the monomers **6a–6c**, **6c** has the largest  $P_s$  value of 80  $\text{nC cm}^{-2}$ , and **6b** has the shortest response time of 55  $\mu\text{s}$ . For the monomers **7a–7c**, **7a** and **7b** have the better physical properties: while **7a** has the largest  $P_s$  value of 85  $\text{nC cm}^{-2}$ , **7b** has the shortest response time of 135  $\mu\text{s}$ . The physical property data are summarized in tables 1 and 2 for **6a–6c** and **7a–7c**, respectively. No regular dependence of the physical properties on the alkyl spacer length could be found.



The initiator AIBN was used to polymerize the monomers. The molecular weight of the polymers **8a–8c** and **9a–9c** was determined by the internal standard polystyrene. The  $T_g$ s of **9a–9c** are lower than those of **8a–8c**. The DSC data show that all polymers, except **9a**, have a  $T_m$ . The  $T_m$ s of **9b** and **9c** are also lower than those of **8a–8c**.

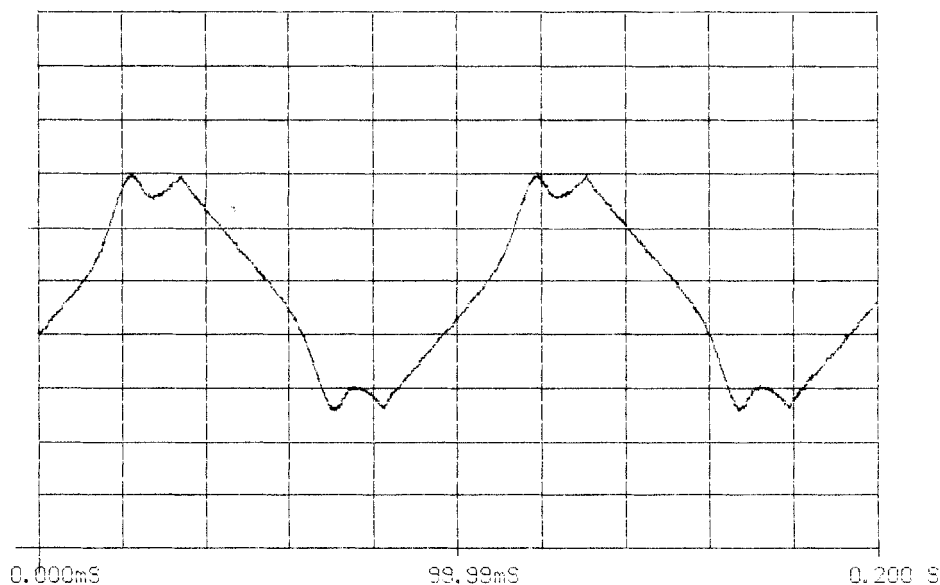
To see whether the polymers could be switched by the electric field, methods adapted from the literature [15, 16] were used. The spontaneous polarizations and response times of the switchable polymers were determined. They were measured at  $T_c - T = 10^\circ\text{C}$ . A cell having a  $25\ \mu\text{m}$  gap and a  $0.16\ \text{cm}^2$  active area was used. An electric field of 30 V (peak) and 10 Hz frequency was applied. While only **8c** of **8a–8c** could be switched by the electric field, **9a–9c** were all switchable by the application of the electric field. The addition of one more molecule of acrylic acid to the polyacrylates **8a–8c** gives the corresponding polyacrylates **9a–9c** a longer and more flexible spacer, thus increasing the tendency to form the  $S_C^*$  phase. A typical switching signal of polymer

Table 1. Phases, transition temperatures, spontaneous polarization  $P_s$ , and response times  $\tau$  of **6a–6c**.

$n$	Phase transition temperatures/ $^{\circ}\text{C}$					$P_s/n\text{C cm}^{-2}$	$\tau/\mu\text{s}$		
	C	$S_C^*$	$S_A$	I					
6	●	57	●	101	●	135	●	39	460
8	●	76	●	105	●	128	●	75	55
10	●	72	●	99	●	127	●	80	235

Table 2. Phases, transition temperatures, spontaneous polarization  $P_s$ , and response times  $\tau$  of **7a–7c**.

$n$	Phase transition temperatures/ $^{\circ}\text{C}$					$P_s/n\text{C cm}^{-2}$	$\tau/\mu\text{s}$		
	C	$S_C^*$	$S_A$	I					
6	●	37	●	68	●	100	●	85	330
8	●	50	●	74	●	100	●	34	135
10	●	53	●	76	●	97	●	60	370

Figure 1. Electrical response of polyacrylate **8c** at  $166^{\circ}\text{C}$ .

**8c** is shown in figure 1. The spontaneous polarization and response times for those switchable polymers are thus obtained. The  $P_s$  values are between 50 and  $72\text{ nC cm}^{-2}$ . The response times for the polymers are short, around 3 ms.

The textures of two different liquid crystal phases of **8c** are shown in figure 2. The phase possessing a fan texture, which could not be switched by the electric field, is a  $S_A$  phase. On the other hand, the phase having a broken fan texture, which could be switched by the electric field, is assigned as a  $S_C^*$  phase. Apparently the  $S_A$  phase thermal stabilities of **9a–9c** are lower than those of **8a–8c**. All the physical property data of **8a–8c** and **9a–9c** are summarized in tables 3 and 4, respectively.



(a)



(b)

Figure 2. (a)  $S_A$  phase texture of **8c** at 173°C, (b)  $S_C^*$  phase texture of **8c** at 165°C.

#### 4. Experimental

Commercially available chemical reagents and solvents were used directly without further purification. Lobar column (Lichroprep Si 60) from Merck was used for middle pressure liquid chromatography (MPLC). Infrared and NMR spectra were recorded on a Perkin-Elmer infrared spectrophotometer 882 and a Varian Gemini 200 spectrometer, respectively. NMR chemical shifts are given in parts per million (ppm) of the total applied magnetic field relative to tetramethylsilane (TMS). Mass spectra were recorded on a JMS-D100 mass spectrometer. Values of specific rotation were measured on a JASCO DIP 370 polarimeter. Liquid crystal phase transition temperatures and textures were determined with a Seiko SSC 5000 DSC differential scanning calorimeter and a Nikon Microphot-FXA polarizing microscope in conjunction with a Mettler FP 52 hot stage, respectively. Gel permeation chromatograms were recorded on a Water GPC instrument and separation columns (Lichrogel PS 4 and 4000), from Merck, were used. Spontaneous polarization and response times were measured by the triangular wave method [15] and the field-reversal method [16], respectively. Cells having a  $2\ \mu\text{m}$  or  $25\ \mu\text{m}$  gap and a  $0.16\ \text{cm}^2$  active area were used.

Table 3. Phases, transition temperatures, spontaneous polarization  $P_s$ , response times  $\tau$ , and molecular weights of polymers **8a–8c**.

Polymers	Phase transition temperature/ $^{\circ}\text{C}$										Physical properties			Molecular weight		
	$T_g$	$T_m$	$S_{\gamma}$	$S_{\zeta}^*$	$S_A$	I	$P_s/\text{nC cm}^{-2}$	$\tau/\text{ms}$	$M_w$	$M_n$	$D$					
<b>8a</b>	57	89	•	187	—	•	203	•	—	—	—	11300	8500	1.30		
<b>8b</b>	50	74	•	188	—	•	201	•	—	—	—	12000	9500	1.27		
<b>8c</b>	47	73	—	—	176	•	184	•	62	3.0	—	6900	5400	1.29		

Table 4. Phases, transition temperatures, spontaneous polarization  $P_s$ , response times  $\tau$ , and molecular weights of polymers **9a–9c**.

Polymers	Phase transition temperature/ $^{\circ}\text{C}$										Physical properties			Molecular weight		
	$T_g$	$T_m$	$S_{\gamma}$	$S_{\zeta}^*$	$S_A$	I	$P_s/\text{nC cm}^{-2}$	$\tau/\text{ms}$	$M_w$	$M_n$	$D$					
<b>9a</b>	34	—	—	—	162	•	178	•	72	3.3	—	11000	9000	1.20		
<b>9b</b>	35	55	—	—	175	•	180	•	50	2.6	—	12100	10000	1.22		
<b>9c</b>	45	68	•	79	149	•	166	•	61	3.0	—	8900	7300	1.22		

4.1. Synthesis of 4-[4-(*n*-acryloxyalkoxy)phenyl]benzoic acids **4a–4c** and 4-{4-[*n*-(3-acryloxypropionyloxy)alkoxy]phenyl}benzoic acids **5a–5c**

To a solution of 4-[4-(*n*-hydroxyalkoxy)phenyl]benzoic acid (**3**) (10 mmol) and 7 ml of acrylic acid in 20 ml of toluene were added 0.4 g of *p*-toluenesulphonic acid and 0.4 g of hydroquinone, consecutively. The mixture was refluxed for 4 h in a Dean–Stark apparatus until the calculated amount of water was collected. After cooling, the solution was poured into 100 ml of water. The precipitated product was collected by filtration. Recrystallization of the crude product from 2-propanol gave a mixture of the respective **4a–4c** and the corresponding **5a–5c** in an 80 per cent yield. The ratio of the respective **4a–4c** to the corresponding **5a–5c** was around 2:1, evaluated from <sup>1</sup>H NMR data.

4.2. Synthesis of 4-{2(S)-[2(S)-methylbutoxy]propionyloxy}phenyl 4-[4-(*n*-acryloxyalkoxy)phenyl]benzoates **6a–6c** and 4-{2(S)-[2(S)-methylbutoxy]propionyloxy}phenyl 4-[4-*n*-(3-acryloxypropionyloxy)alkoxy]phenyl}benzoates **7a–7c**

The following esterification procedure, adapted from the literature [7], was used to prepare the compounds **6a–6c** and **7a–7c**. An example procedure is given here:

A solution of a mixture of the acids **4c** and **5c** (10 mmol of both), the substituted chiral phenol **2** (11 mmol) [6], DCC (11 mmol), and DMAP (1 mmol) in 30 ml of THF was stirred at room temperature for 12 h. Filtration and removal of the solvent from the filtrate gave the crude product which was then purified by flash column chromatography (SiO<sub>2</sub>/CHCl<sub>3</sub>/EtOAc/hexane = 1/1/3). The separation of **6c** from **7c** was realized by MPLC (CHCl<sub>3</sub>/EtOAc/hexane = 1/1/3).

**6c**: Yield: 38 per cent. IR (KBr) 2921, 2853, 1771, 1736, 1604, 1503, 1187, 1130, 999, 831 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 8.21 (d, 2 H, *J* = 8.5 Hz), 7.68 (d, 2 H, *J* = 8.5 Hz), 7.59 (d, 2 H, *J* = 8.8 Hz), 7.2 (m, 4 H), 7.0 (d, 2 H, *J* = 8.8 Hz), 6.38 (d, 1 H, *J* = 17.1 Hz), 6.1 (AB, d, 1 H, *J* = 17.1, 10.3 Hz), 5.8 (d, 1 H, *J* = 10.3 Hz), 4.18 (overlapped peaks, 3 H), 4.01 (t, 2 H, *J* = 6.4 Hz), 3.55 (AB, d, 1 H, *J* = 8.7, 6.1 Hz), 3.25 (AB, d, 1 H, *J* = 8.7, 6.7 Hz), 1.9–1.1 (overlapped peaks, 22 H), 1.0–0.85 (overlapped peaks, 6 H); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 171.9, 166.3, 164.9, 159.4, 148.4, 147.8, 146.0, 131.8, 130.7, (2C), 130.5, 128.5, 128.3 (2C), 127.1, 126.5 (2C), 122.6 (2C), 122.2 (2C), 114.9 (2C), 75.8, 75.1, 68.1, 64.6, 35.0, 29.4, 29.3, 29.2 (2C), 29.1, 28.5, 26.1, 26.0, 25.8, 18.6, 16.4, 11.2; MS (*m/z*, relative intensity) 658 (M<sup>+</sup>, 0.66); [α]<sub>D</sub><sup>25</sup> = -29° (CHCl<sub>3</sub>, *c* = 1.0); Elemental analysis for C<sub>40</sub>H<sub>50</sub>O<sub>8</sub>: calculated H 7.65, C 72.92; found: H 7.68, C 72.89 per cent.

**7c**: Yield: 20 per cent. IR (KBr) 2933, 2854, 1773, 1737, 1636, 1604, 1504, 1180, 1130, 830 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 8.22 (d, 2 H, *J* = 8.5 Hz), 7.68 (d, 2 H, *J* = 8.5 Hz), 7.59 (d, 2 H, *J* = 8.8 Hz), 7.2 (m, 4 H), 7.0 (d, 2 H, *J* = 8.8 Hz), 6.4 (d, 1 H, *J* = 17.2 Hz), 6.1 (AB, d, 1 H, *J* = 17.2, 10.2 Hz), 5.8 (dd, 1 H, *J* = 10.2 Hz), 4.42 (t, 2 H, *J* = 6.4 Hz), 4.15 (overlapped peaks, 3 H), 4.0 (t, 2 H, *J* = 6.4 Hz), 3.55 (AB, d, 1 H, *J* = 8.8, 6.2 Hz), 3.25 (AB, d, 1 H, *J* = 8.8, 6.7 Hz), 2.7 (t, 2 H, *J* = 6.4 Hz), 1.9–1.1 (overlapped peaks, 22 H), 1.0–0.85 (overlapped peaks, 6 H); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 171.9, 170.7, 166.3, 164.9, 159.6, 148.4, 147.9, 146.1, 131.9, 131.1, 130.7 (2C), 128.4 (2C), 128.1, 127.2, 126.6 (2C), 122.7 (2C), 122.3 (2C), 114.9 (2C), 75.9, 75.2, 68.1, 64.9, 60.0, 35.1, 33.9, 29.5 (3C), 29.3, 29.1, 28.5, 26.1, 26.0, 25.8, 18.6, 16.5, 11.3; MS (*m/z*, relative intensity) 729 (M<sup>+</sup> - 1, 13.6); [α]<sub>D</sub><sup>25</sup> = -26° (CHCl<sub>3</sub>, *c* = 1.05). Elemental analysis for C<sub>43</sub>H<sub>54</sub>O<sub>10</sub>: calculated H 7.45, C 70.66 found: H 7.43, C 70.73 per cent.



### 4.3. Preparation of the polymers 8a–8c and 9a–9c

The polymers were prepared by the following general procedure: 0.4 g of a monomer was dissolved in 6 ml of toluene and polymerized in the presence of 1–2 mol% of AIBN in a sealed tube at 70°C for 18 h. The polymer was precipitated in MeOH, filtrated, redissolved in CHCl<sub>3</sub>, and again precipitated in MeOH. The precipitation procedure was repeated until no more monomer was detected on the TLC plate. The yield was around 60 per cent.

**8c**: IR (KBr) 2931, 2855, 1773, 1604, 1503, 1187, 827 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 8.2, 7.8–7.4, 7.2, 7.0, 4.2–3.9, 3.6, 3.2, 2.4, 1.9–1.1, 1.1–0.8; <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 172.47, 165.45, 160.06, 148.94, 148.39, 146.50, 132.37, 131.23, 128.96, 128.87, 127.72, 127.07, 123.19, 122.79, 115.44, 76.40, 75.68, 68.60, 65.3–65.16, 35.61, 30.06–29.13, 26.64, 26.42, 19.17, 17.03, 11.79; [α]<sub>D</sub><sup>25</sup> = -27° (CHCl<sub>3</sub>, c = 1.0) M<sub>n</sub>: 5400, M<sub>w</sub>: 6900, D = 1.29.

**9c**: IR (KBr) 2933, 2854, 1773, 1737, 1604, 1504, 1180, 830 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 8.2, 7.8–7.4, 7.2, 7.0, 4.5–3.9, 3.6, 3.3, 2.7, 2.3, 1.9–1.1, 1.1–0.8; <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 171.9, 164.8, 159.4, 148.3, 147.8, 145.7, 130.6, 128.3, 127.2, 126.4, 122.6, 122.2, 114.8, 75.8, 75.1, 67.9, 64.9, 64.7, 64.6, 35.0, 29.3, 28.6, 28.4, 26.0, 25.9, 18.6, 16.5, 11.2; [α]<sub>D</sub><sup>25</sup> = -28° (CHCl<sub>3</sub>, c = 1.04) M<sub>n</sub>: 7300 M<sub>w</sub>: 8900, D = 1.22.

## 5. Conclusion

The acrylates, containing two acrylic acid moieties, were prepared by the esterification of acrylic acid with the corresponding *n*-hydroxyalkoxyphenylbenzoic acid. These acrylates were used in preparing novel liquid crystal side chain polymers. The synthesis and characterization of the monomers and polymers show that the 2(*S*)-[2(*S*)-methylbutoxy]propionyloxy group is very useful for preparing not only FLCs but their side chain polymers. The response times of these new ferroelectric side chain polyacrylates were as short as 3 ms.

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