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## Liquid Crystals

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# Thermotropic liquid crystalline $\alpha$ -[bis(2-hydroxyethyl)amino]- $\omega$ -(4-methoxybiphenyl-4-oxy)alkane hydrochlorides

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Liquid crystalline  $\alpha$ -[bis(2-hydroxyethyl)amino]- $\omega$ -(4'-methoxybiphenyl-4-oxy)alkane hydrochlorides with different spacer lengths (6, 8, 10 methylene units) have been synthesized and characterized by NMR, DSC, polarizing optical microscopy and X-ray diffraction. The melting temperatures of the hydrochlorides decrease with increasing number of methylene units in the spacer. Highly ordered and very viscous liquid crystalline (LC<sup>1</sup>) smectic phases are formed on melting. Upon further heating these phases are transformed into a less viscous smectic C phase (LC<sup>2</sup>). The temperature of the LC<sup>1</sup>–LC<sup>2</sup> transition decreases and the temperature of the LC<sup>2</sup> to isotropic phase transition (LC<sup>2</sup>–I transition) increases with increasing number of methylene units in the spacer.

## 1. Introduction

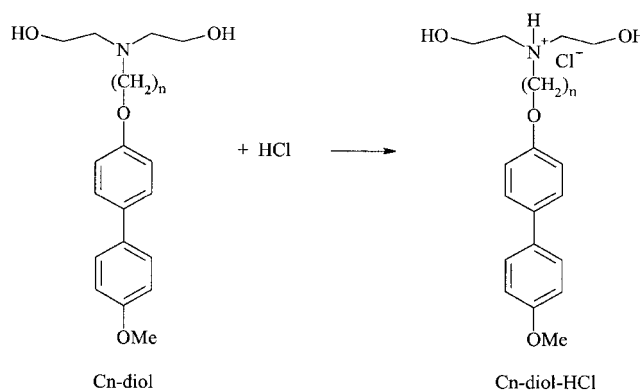
Liquid crystalline (LC) amines have been used to prepare liquid crystalline polymers [1, 2] and complexes with monomeric [3–5] or polymeric organic acids [6]. Many LC quaternary ammonium salts have also been prepared [7–11]. LC amine hydrochlorides, which are the focus of this work, have not been the subject of intensive research work. So far, mainly amine hydrochlorides with long aliphatic chains have been synthesized. However, only a few of them were liquid crystalline [12–14]. LC amine hydrochlorides having a mesogenic biphenyl group have not yet been reported.

$\alpha$ -[Bis(2-hydroxyethyl)amino]- $\omega$ -(4-methoxybiphenyl-4-oxy)alkanes with different spacer lengths ( $C^n$ -diol,  $n$  = number of methylene units in the spacer), as well as their analogues have been used as diols in the synthesis of liquid crystalline polyurethanes [15–19] and several polyesters which mostly do not exhibit LC properties [20–22]. When we synthesized polyesters from diacid dichlorides and  $C^n$ -diols in the presence of tertiary amine as an acid acceptor, it was observed that the thermal properties of the polyesters greatly depended on the method of isolation of the product. That led to the conclusion that HCl, the side product of polyesterification, was not bound to the tertiary amine but to the nitrogen atom of the  $C^n$ -diol in the polyester. The preparation of  $C^n$ -diol hydrochlorides ( $C^n$ -diol-HCl) and their liquid crystalline properties are the subject of the present work.

## 2. Experimental

The synthesis of the  $C^n$ -diols ( $n$  = 6, 8, 10) is described elsewhere [15].  $C^n$ -diol-HCl were prepared by dissolving the  $C^n$ -diol (1 g) in distilled tetrahydrofuran (10 g). Concentrated hydrochloric acid was added in an excess of 20 mol %.  $C^n$ -diol-HCl precipitated immediately and the reaction mixture was left at room temperature for one hour. After filtration, the product was dried for 24 h over P<sub>2</sub>O<sub>5</sub> in a vacuum at 50 °C. The reaction is depicted in the scheme. The yields of  $C^n$ -diol-HCls, which precipitated from THF as a white powder, were 80%.

Differential scanning calorimetry (DSC) analysis was performed on a Perkin-Elmer Pyris 1 calorimeter. The samples were heated twice and subsequently cooled in



Scheme. The quaternization of a  $C^n$ -diol ( $n$  = 6, 8, 10) with hydrochloric acid.

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the temperature range from 20 to 220 °C. Heating and cooling rates were 10 K min<sup>-1</sup>. After the heating or cooling cycle, the samples were maintained for 2 min at 220 or 20 °C, respectively. To detect the onset of decomposition, DSC measurements were run up to 300 °C with a heating rate of 10 K min<sup>-1</sup>. <sup>1</sup>H NMR spectra were recorded at 25 °C on a Varian VXR-300 spectrometer using DMSO-d<sub>6</sub> as the solvent and TMS as the internal standard. Optical textures were obtained using a Carl Zeiss polarizing optical microscope, Stemi SV6, equipped with an MC 80 microscope camera and a Mettler Toledo FP82 hot stage. X-ray diffraction (XRD) patterns were taken on a Siemens D-5000 diffractometer using Cu K<sub>α</sub> radiation ( $\lambda = 1.54 \text{ \AA}$ ) in 0.04° steps from 5° to 35° (in  $2\theta$ ) with 5 s per step.

### 3. Results and discussion

#### 3.1. NMR spectroscopy

The structures of the *C<sup>n</sup>*-diols and *C<sup>n</sup>*-diol-HCl were confirmed by <sup>1</sup>H NMR and <sup>1</sup>H NMR COSY spectroscopy. The signals in the <sup>1</sup>H NMR spectra of the *C<sup>n</sup>*-diols are assigned as: 7.5 and 7.0 ppm (m, 4H<sup>+</sup> 4H, biphenyl), ≈ 4.3 ppm (t, 2H, OH), 4.0 ppm (t, 2H, biphenyl-O-CH<sub>2</sub>), 3.8 ppm (s, 3H, O-CH<sub>3</sub>), 3.4 ppm (q, 4H, CH<sub>2</sub>OH), 2.5 ppm (overlapping with DMSO-d<sub>6</sub>, N(CH<sub>2</sub>CH<sub>2</sub>OH)<sub>2</sub>), 2.4 ppm (t, 2H, N-CH<sub>2</sub>-spacer), 1.7 ppm (m, 2H, O-CH<sub>2</sub>-CH<sub>2</sub>), 1.2–1.5 ppm (m, 8H–14H, inner CH<sub>2</sub> groups of aliphatic spacer). The NMR spectra of the *C<sup>n</sup>*-diols differ only in the intensity of the latter signal. The NMR spectra of the *C<sup>n</sup>*-diol-HCl exhibit significant changes only in the vicinity of the quaternary nitrogen atom. The signals are assigned as follows: ≈ 10 ppm (broad s, 1H, H<sup>-</sup>N<sup>+</sup>), 7.5 and 7.0 ppm (m, 4H<sup>+</sup> 4H, biphenyl), ≈ 5.3 ppm (broad s, 2H, OH), 4.0 ppm (t, 2H, biphenyl-O-CH<sub>2</sub>), 3.8 ppm (m, 7H, O-CH<sub>3</sub> and CH<sub>2</sub>OH), 3.25 ppm (m, 4H, N<sup>+</sup>(CH<sub>2</sub>CH<sub>2</sub>OH)<sub>2</sub>), 3.15 ppm (m, 2H, N<sup>+</sup>CH<sub>2</sub>-spacer) 1.7 ppm (m, 4H, O-CH<sub>2</sub>-CH<sub>2</sub> and N<sup>+</sup>-CH<sub>2</sub>-CH<sub>2</sub>-spacer), 1.2–1.5 ppm (m, 4H–12H, inner CH<sub>2</sub> groups of aliphatic spacer).

#### 3.2. Differential scanning calorimetry

The DSC curves of all three hydrochlorides taken during the first heating scan are shown in figure 1. They all exhibit a melting peak (Cr–LC<sub>1</sub>) followed by a liquid

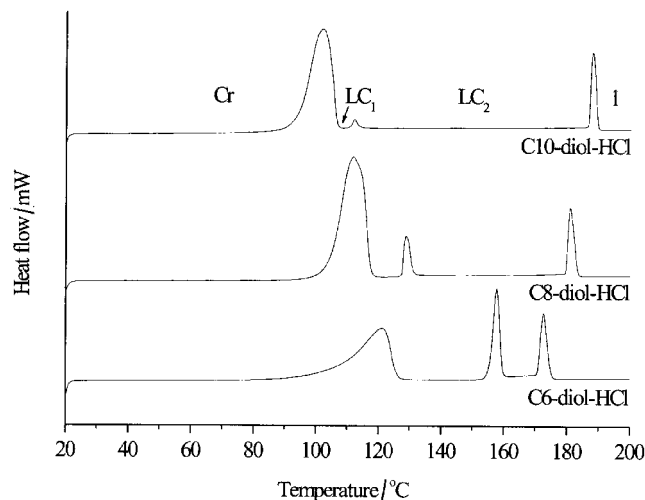


Figure 1. DSC thermograms of the *C<sup>n</sup>*-diol-HCl taken during the first heating (endo—up).

crystal transition (LC<sub>1</sub>–LC<sub>2</sub>) and isotropization (LC<sub>2</sub>–I). The melting temperature decreases with increasing number of methylene units in the spacer, as similarly observed for *n*-alkylammonium chlorides [7]. The temperature of the LC<sub>1</sub>–LC<sub>2</sub> transition also decreases, and the temperature of transition from LC<sub>2</sub> to isotropic (LC<sub>2</sub>–I) melt increases with increasing number of methylene units in the spacer. The melting temperatures and enthalpies are summarized in table 1. The isotropization temperatures of the *C<sup>n</sup>*-diol-HCl are 70–80 °C higher than the isotropization temperatures of the corresponding *C<sup>n</sup>*-diols [15].

All three *C<sup>n</sup>*-diol-HCl exhibited I–LC<sub>2</sub> and LC<sub>2</sub>–LC<sub>1</sub> transitions on cooling. Crystallization (LC<sub>1</sub>–Cr) was only observed for the C10-diol-HCl at 40 °C. There was no crystallization observed for the C8-diol-HCl and only a very small transition at 77 °C for the C6-diol-HCl. This behaviour is ascribed to relaxation and is explained below.

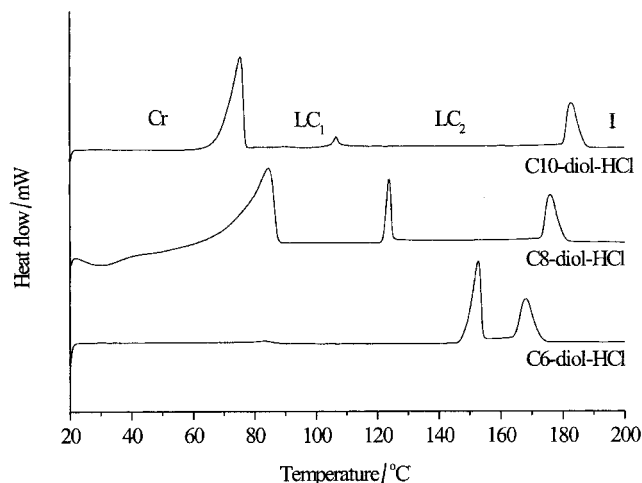
The second heating scans of the *C<sup>n</sup>*-diol-HCl are shown in figure 2 and details concerning the thermal properties are presented in table 2. C6-diol-HCl exhibits a small endothermic peak at 81 °C, as well as the LC<sub>1</sub>–LC<sub>2</sub> and LC<sub>2</sub>–I transitions. C8-diol-HCl exhibits a small cold crystallization peak at 30 °C, followed by a

Table 1. Thermal properties of *C<sup>n</sup>*-diol-HCl taken during the first heating.

Sample	Cr–LC <sub>1</sub>		LC <sub>1</sub> –LC <sub>2</sub>		LC <sub>2</sub> –I	
	<i>T<sub>m</sub></i> /°C	$\Delta H_m$ /J g <sup>-1</sup>	<i>T</i> /°C	$\Delta H$ /J g <sup>-1</sup>	<i>T</i> /°C	$\Delta H$ /J g <sup>-1</sup>
C6-diol-HCl	121.1	62.0	157.8	19.7	172.6	13.4
C8-diol-HCl	112.1	103.8	129.1	9.0	180.7	14.7
C10-diol-HCl	101.8	92.1	112.0	2.1	188.0	15.2

Table 2. Thermal properties of C<sup>n</sup>-diol-HCl taken during the second heating.

Sample	Cr-LC <sup>1</sup>		LC <sup>1</sup> -LC <sup>2</sup>		LC <sup>2</sup> -I	
	T <sub>m</sub> /°C	ΔH <sub>m</sub> /J g <sup>-1</sup>	T/°C	ΔH/J g <sup>-1</sup>	T/°C	ΔH/J g <sup>-1</sup>
C6-diol-HCl	/	/	152.9	18.0	168.4	13.3
C8-diol-HCl	84.3	65.4	123.8	8.4	176.1	14.5
C10-diol-HCl	75.1	39.5	106.6	1.7	182.6	15.0

Figure 2. DSC thermograms of the C<sup>n</sup>-diol-HCl taken during the second heating (endo—up).

melting peak at 84.6°C. The melting temperature on the second scan is 37°C lower than on the first scan. The melting enthalpy is 64.5 J g<sup>-1</sup>, which is less than that obtained in the first scan, but much more than could be expected from the cold crystallization peak. In fact, the crystallization of C8-diol-HCl had already started when the sample was held at 20°C. This was confirmed by running the experiment from -20 to 220°C. A small crystallization peak appeared on cooling in the temperature range 0–30°C with the peak temperature appearing at 20.2°C. Nevertheless, cold crystallization was observed again in the second heating scan. In this experiment, the melting temperature was 88.0°C and the melting enthalpy was 69.5 J g<sup>-1</sup>, which is, as expected, higher than in the previous experiment. Melting was followed by the LC<sup>1</sup>-LC<sup>2</sup> and LC<sup>2</sup>-I transitions. C10-diol-HCl also exhibits a much lower melting temperature and melting enthalpy in the second heating scan, followed by LC<sup>1</sup>-LC<sup>2</sup> and LC<sup>2</sup>-I transitions.

The third scan was performed after the samples had been held at room temperature for 12 days. The DSC curves of C6-diol-HCl and C8-diol-HCl were the same as in the second scan, while the melting point of C10-diol-HCl was higher than in the second scan. Actually, there was a broad endotherm with two peaks at 88°C and 92°C. The enthalpy was 72.5 J g<sup>-1</sup>, which

is almost double the value obtained in the second scan. This means that the crystallization of C10-diol-HCl proceeds slowly at room temperature, which is not the case with C6-diol-HCl and C8-diol-HCl.

DSC curves showed no significant deviations on heating to 220°C, indicating no decomposition. The mass of the samples after two heating cycles also remained the same. Decomposition was observed on DSC curves at temperatures above 230°C. The weight loss was 7.0% for C6-diol-HCl, 8.3% for C8-diol-HCl and 11.3% for C10-diol-HCl.

### 3.3. Optical microscopy

While heating the C8-diol-HCl and C10-diol-HCl, melting was observed at 112°C and 100°C, respectively. The melts were relatively viscous and the small droplets formed did not coalesce. Therefore, it was not possible to determine the texture and the nature of the LC<sup>1</sup> phase. Upon heating the samples above the LC<sup>1</sup>-LC<sup>2</sup> transition, the melts became more fluid, and small droplets coalesced and formed bigger droplets with a sanded texture. On the other hand, C6-diol-HCl showed no signs of melting below 159°C, which is just above the LC<sup>1</sup>-LC<sup>2</sup> transition. It seems that the melt of C6-diol-HCl in the LC<sup>1</sup> state was so viscous that droplets could not be formed. We assume that the high viscosity of the LC<sup>1</sup> state also prevents rearrangement of the molecules on cooling and crystallization. Therefore, the LC<sup>1</sup> phase freezes on cooling to room temperature. The molecules of C8-diol-HCl and C10-diol-HCl seem to be more mobile. Therefore, the viscosity of their LC<sup>1</sup> phases is not as high. Crystallization is possible although supercooling is necessary.

When the isotropic melt of C10-diol-HCl was cooled to 180°C, small bright particles were formed which immediately disintegrated into a much smaller, fog-like texture. Only a few of the particles remained that were big enough to observe their focal-conic texture. The texture remained the same even after the LC<sup>2</sup>-LC<sup>1</sup> transition at 105°C. At 40°C the texture changed from fog-like into a colourful focal-conic fan texture. Similar observations were found on cooling the C8-diol-HCl, except that the focal-conic fan texture was formed at 120°C, i.e. at the LC<sup>2</sup>-LC<sup>1</sup> transition.

On cooling the isotropic melt of C6-diol-HCl, a fog-like texture with a few focal-conics was also observed. The focal-conic fan texture appeared at 155°C, which is  $\approx 5^\circ\text{C}$  above the LC<sub>2</sub>–LC<sub>1</sub> transition as determined by DSC. There was no change observed on further cooling.

The structure of the fog-like texture was only clearly visible in the uncovered part of the C6-diol-HCl and is shown in figure 3. A similar texture has also been obtained in the uncovered part of 4,4'-di-*n*-dodecyloxyazobenzene at 105°C and was assigned as a smectic C phase [23].

### 3.4. X-ray diffraction

The diffractograms of C6-diol-HCl, C8-diol-HCl and C10-diol-HCl were recorded at different temperatures and are shown in figures 4–6, respectively. Multiple reflections of the crystal phases can be observed at room temperature in the diffractograms 1.

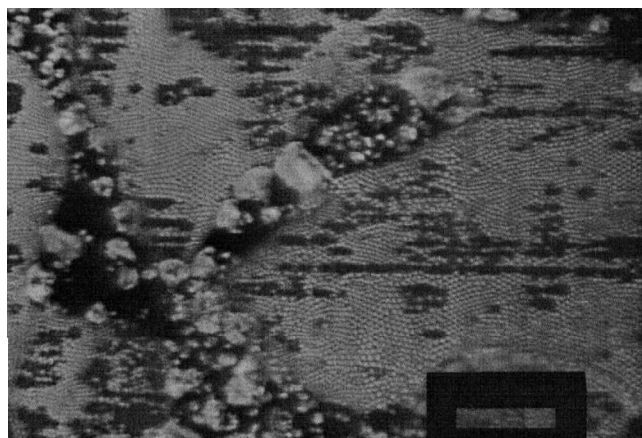


Figure 3. The fog-like texture observed in the uncovered part of C6-diol-HCl. (Magnification 125 $\times$ ).

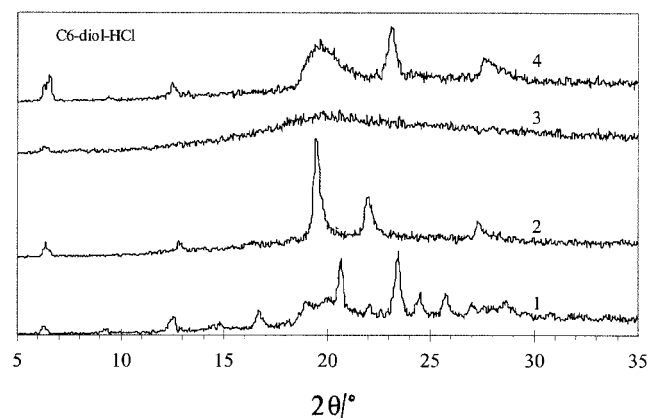


Figure 4. X-ray diffractograms of C6-diol-HCl: 1—room temperature before heating; 2— $T = 140^\circ\text{C}$ ; 3— $T = 160^\circ\text{C}$ ; 4—room temperature after cooling from LC<sub>2</sub> phase.

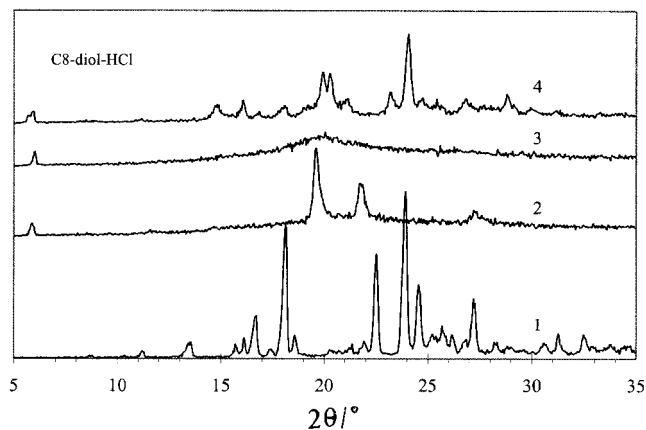


Figure 5. X-ray diffractograms of C8-diol-HCl: 1—room temperature before heating; 2— $T = 120^\circ\text{C}$ ; 3— $T = 135^\circ\text{C}$ ; 4—room temperature after cooling from LC<sub>2</sub> phase.

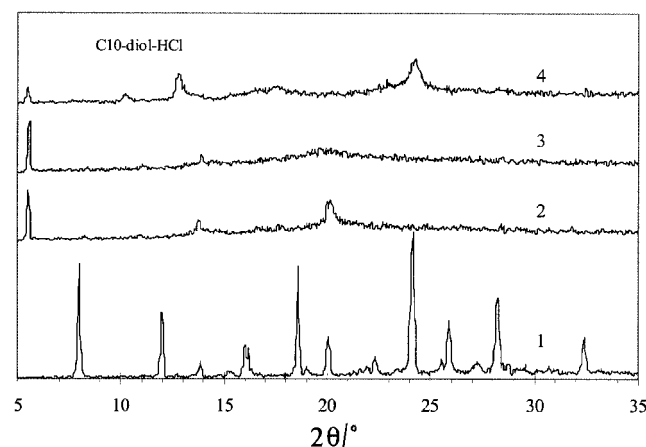


Figure 6. X-ray diffractograms of C10-diol-HCl: 1—room temperature before heating; 2— $T = 108^\circ\text{C}$ ; 3— $T = 120^\circ\text{C}$ ; 4—room temperature after cooling from LC<sub>2</sub> phase.

Diffractograms 2 were recorded in the temperature region of the LC<sub>1</sub> phase. In diffractogram 2 of C6-diol-HCl recorded at 140°C, there are two Bragg reflections of the second and fourth order at  $2\theta = 6.36^\circ$  and  $12.8^\circ$  corresponding to a layer distance of 2.78 nm. This spacing is 1.21 times the length of the molecule (2.30 nm) calculated in its most extended conformation. Three reflections in the wide angle region ( $2\theta = 19.48^\circ$ ,  $22.08^\circ$ , and  $27.48^\circ$ ) indicate that a highly ordered smectic phase was formed. In diffractogram 2 of C8-diol-HCl recorded at 120°C, there is only one Bragg reflection in the middle angle region at  $2\theta = 5.88^\circ$ . Assuming that this reflection is also of the second order, it corresponds to a layer distance of 3.01 nm. This spacing is 1.18 times the length of the molecule (2.55 nm). Three reflections in the wide angle region ( $2\theta = 19.6^\circ$ ,  $21.72^\circ$  and  $27.20^\circ$ ) indicate that the same type of highly ordered smectic phase was formed as in the C6-diol-HCl. In diffractogram 2 of

C10-diol-HCl recorded at 102°C, there are four Bragg reflections of second, third, fourth and fifth order at  $2\theta = 5.52^\circ$ ,  $8.28^\circ$ ,  $10.96^\circ$  and  $13.76^\circ$ , which correspond to a layer thickness of 3.2 nm. This spacing is 1.14 times the length of the molecule (2.80 nm). There is only one reflection in the wide angle region at  $2\theta = 20.16^\circ$ , indicating that the smectic phase formed is not of the same type as for the other hydrochlorides.

The increases in thickness of the lamellae due to elongation of the alkyl chains by two methylene groups are 0.23 and 0.19 nm. This is smaller than the length of one zig-zag in a fully extended paraffin chain (0.25 nm), thus indicating that the chains are tilted. According to the increase in layer thicknesses, the smectic structures are monolayered with molecules laterally arranged head to tail as observed in *N*-alkylpyridinium halides [8]. The exact types of the smectic phases could not be determined on the basis of the X-ray spectra of the unoriented samples.

In diffractograms 3, which were taken in the temperature region of the LC<sub>2</sub> phase, only one Bragg diffraction was observed for C6-diol-HCl and C8-diol-HCl at  $2\theta = 6.28^\circ$  and  $6.00^\circ$ , respectively. In the diffractogram of C10-diol-HCl, two Bragg diffractions of second and fifth order are observed at  $2\theta = 5.6^\circ$  and  $13.92^\circ$ . Assuming that the diffractions of C6-diol-HCl and C8-diol-HCl are also of the second order, the calculated layer spacings are 2.82 nm (C6-diol-HCl), 2.94 nm (C8-diol-HCl) and 3.16 nm (C10-diol-HCl). According to the X-ray diffractograms, as well as polarizing optical microscopy, monolayered smectic C phases are formed.

The diffractograms 4, which were recorded after the samples were cooled from LC<sub>2</sub> phase to room temperature, differ considerably. In diffractogram 4 of C6-diol-HCl, the number of Bragg diffractions is the same as in the LC<sub>1</sub> phase, though they are broader and the positions of the diffractions are slightly changed ( $2\theta = 6.56^\circ$ ,  $12.48^\circ$ ,  $19.68^\circ$ ,  $23.2^\circ$ , and  $27.84^\circ$ ). We ascribe this structure to a highly ordered LC<sub>1</sub> smectic phase that is locked into the glassy phase.

Multiple reflections can be observed in diffractogram 4 of C8-diol-HCl. Some of them correspond to the LC<sub>1</sub> phase and some to the crystalline phase. There are also some diffractions which are not present in any of the diffractograms 1–3. It seems that part of the LC<sub>1</sub> phase is locked in a glassy state as in C6-diol-HCl, and part of it is transformed into a crystal phase that does not seem to be the same as that in the original sample. Possibly one of the crystal smectic phases was formed.

In diffractogram 4 of C10-diol-HCl, the diffraction at  $2\theta = 5.52^\circ$  corresponds to the LC<sub>1</sub> phase, while the others ( $2\theta = 10.24^\circ$ ,  $12.72^\circ$ ,  $17.76^\circ$  and  $24.28^\circ$ ) are not present in diffractograms 1–3. It seems that C10-diol-HCl crystallizes into a crystal smectic phase.

#### 4. Conclusions

Liquid crystalline  $\alpha$ -[bis(2-hydroxyethyl)amino]- $\omega$ -(4'-methoxybiphenyl-4-oxy)alkane hydrochlorides with different spacer lengths (6, 8 or 10 methylene units) have been synthesized and their liquid crystalline properties characterized. The liquid crystalline properties depend on the spacer length. The melting temperature (Cr–LC<sub>1</sub>) of the hydrochlorides decreases with increasing number of methylene units in the spacer. When melted, highly ordered and very viscous smectic phases are formed. According to X-ray spectroscopy, all the phases are monolayered and tilted. Nevertheless, C10-diol-HCl does not form the same type of smectic phase as C6- and C8-diol-HCl. On further heating these phases are transformed into a less viscous smectic C phase. The temperature of the LC<sub>1</sub>–LC<sub>2</sub> transition decreases, while the temperature of the transition from LC<sub>2</sub> to isotropic (LC<sub>2</sub>–I) melt increases, with increasing number of methylene units in the spacer. The isotropization temperatures of the C<sup>*n*</sup>-diol-HCl's are 70–80°C higher than the isotropization temperatures of the corresponding C<sup>*n*</sup>-diols. On cooling from the LC<sub>1</sub> phase to room temperature, the LC<sub>1</sub> phase of C6-diol-HCl is frozen in a glassy state. C8- and C10-diol-HCl's crystallize but not into their original crystal structures. It appears likely that crystal smectics are formed.

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