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Method and Means for Disclination-Free Orienting
of Liquid Crystals

10 The invention is concerned with a method for the disclination-free planar orientation of chiral liquid crystalline side-chain polymers, with layers of disclination-free oriented chiral liquid crystalline side-chain polymers and with filters and retard films manufactured using these layers.

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For applications of chiral liquid crystalline side-chain polymers in optical components, especially e.g. in optical filters or retarders for so-called STN cells, it is necessary to give a liquid crystalline polymer layer a uniform planar orientation. "Planar" as used in this description
20 means either arrangements of molecules parallel to the layer surfaces or arrangements in which the molecules have a so-called "tilt angle" of up to 30°.

During the manufacture of such layers focal-conical disclination
25 lines occur, e.g. as a result of flow orientation or cooling of the liquid crystalline side-chain polymers from the isotropic phase. As a result of these disclination lines, incident light is scattered. The scattered light is also depolarized and thus reduces the optical properties of the filters, retarders etc. which are manufactured using the chiral liquid
30 crystalline side-chain polymers (see Jpn. J. Appl. Phys., Vol. 29, No.10, 1990, pages 1974-1984, especially page 1975).

The object of the invention is to avoid such interfering disclinations in layers of chiral liquid crystalline side-chain polymers.

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This is achieved in accordance with the invention by subjecting a layer of chiral liquid crystalline side-chain polymer having negative dielectric anisotropy in its cholesteric phase to an electric field directed

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at right angles to the layer and fixing the orientation induced by the electric field by cooling the polymer to below its glass temperature.

5 The method according to the invention is surprisingly successful in giving a uniform planar orientation over a large area to layers of chiral liquid crystalline side-chain polymers having a small pitch, such that practically no disclination lines occur over the entire orientated region.

10 Furthermore, it has been found that this is also possible when the layer of chiral liquid crystalline side-chain polymers is arranged between plates or boundary walls of which only one or none has a wall orientation directed in a preferred direction. Above all, it is also possible to perform this orientation on layers which generally are arranged not
15 between two plates, but only on one substrate. This is particularly surprising, because such orientation operations on the two sides of the layer have hitherto been considered to be unavoidable.

An embodiment of the invention will now be described with
20 reference to the accompanying drawing.

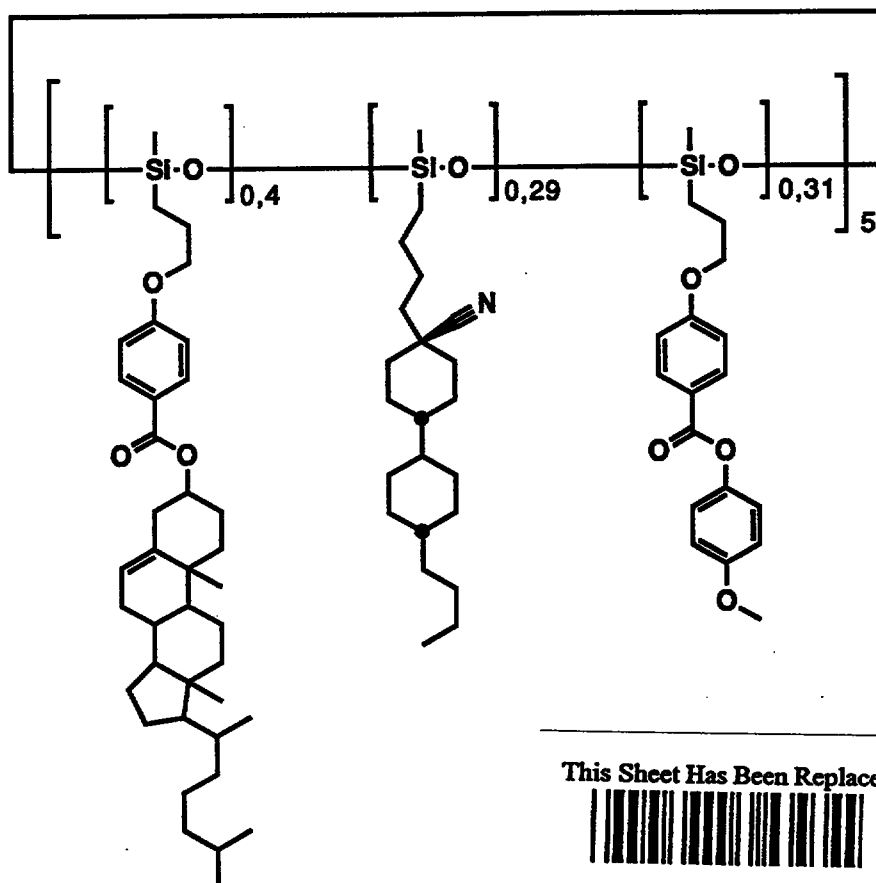
The single drawing shows a schematic representation in section of a chiral liquid crystalline side-chain polymer layer between two plates. Such arrangements are known, for example, from the article
25 mentioned above. In the present embodiment a layer of chiral liquid crystalline side-chain polymer 3 having a small pitch p is situated between two plane-parallel glass plates 1, 2. The glass plates have, on their surfaces facing the layer, ITO coatings 4, 5 which are known from liquid crystal displays. In the present simplest example these ITO
30 coatings are not segmented as is usual in liquid crystal displays. However, for special applications they can also be segmented. The separation of the glass plates is adjusted to about $8\text{ }\mu\text{m}$ using so-called spacers, although the plate separation is not critical. The chiral LC side-chain polymers which are orientatable in accordance with the
35 present invention are characterized in that their liquid crystalline side-chain(s) induce(s) the following functions: 1. chirality, 2. a cholesteric phase above the glass temperature of the polymer and 3. a negative dielectric anisotropy. These three properties can be combined together in a single side-chain monomer type or can be divided into different side-



chain monomer types. In the latter case, this is a liquid crystalline side-chain copolymer.

As monomer components there generally come into consideration
5 two types, namely either those which are bonded terminally with the polymerizable function or those in which the central part is linked approximately in the middle with the polymerizable function.

A compound of formula I is an especially suitable example of a
10 chiral liquid crystalline side-chain polymer:



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This compound has been prepared as follows:

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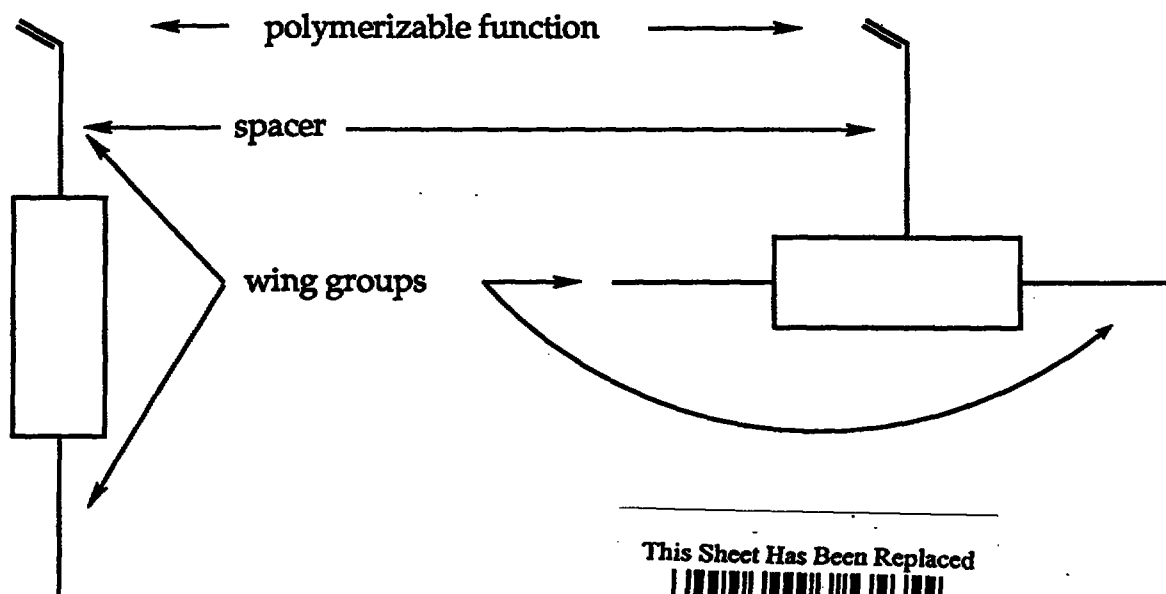
1.19 g (2.18 mmol) of 4-allyloxybenzoic acid cholesterol ester, 0.48 g (1.69 mmol) of 4-allyloxybenzoic acid (4-methoxy)phenyl ester, 0.5 g (1.58 mmol) of 1-(3-butenyl)-4 α -(trans-4-pentylcyclohexyl)-1 β -cyclohexanecarbonitrile and 0.33 g (1.09 mmol) of cyclic siloxane with 5 OSi(CH₃)H
20 units were placed in 6 ml of dry toluene under an argon atmosphere and heated to 60°C. After the addition of 0.03 ml of platinum dicyclo-

pentadiene dichloride (1 wt.% in CH_2Cl_2) as the catalyst the mixture was heated to 100°C for 2 hours. For the working-up, the solution was evaporated, the residue was taken up in THF and the catalyst was separated by filtration over a silica gel column ($l = 4 \text{ cm}$, $\phi = 3 \text{ cm}$).

- 5 Residues of silica gel were removed by filtration over a micropore filter ($2 \mu\text{m}$). The residual monomer content of the product obtained was about 9%. In order to separate residual monomers - where necessary - the crude product can be precipitated from THF in ethanol. The yield was 2.0 g (80%).

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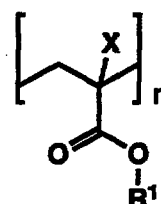
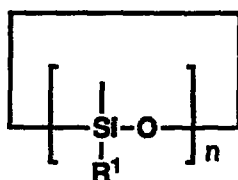
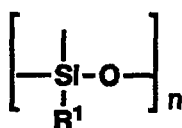
In the monomers used for these LC side-chain polymers the mesogens can be bonded with the polymerizable function or polymer chain either via one of the two wing groups as the spacer or via a separated spacer which is collaterally chemically bonded with the mesogen. Both types of monomer can also be used within a LC copolymer.



II

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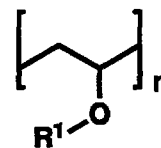
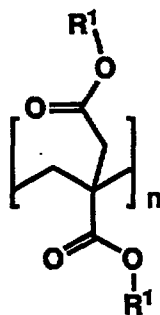
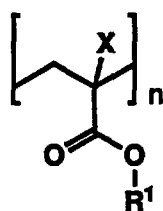
The polymers, copolymers, linear or cyclic oligomers can be synthesized from the following polymer components:



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IV

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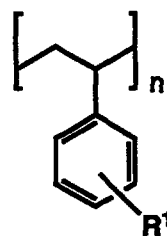
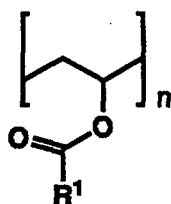


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VI

VII

VIII



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IX

X

wherein X= H (polyacrylate)
 CH₃ (polymethacrylate)
 Cl (poly- α -chloroacrylate)
 R¹ = mesogenic residue
 R² = H or lower alkyl

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These individual monomers can be converted into copolymers, whereby monomers having the same polymerizable functions (copolymers having a "pure" polymethacrylate chain and different side-chains) or different polymerizable functions (e.g. copolymers from styrene derivatives and methacrylates having different side-chains) can be used according to the state of the art in the field of copolymerization of correspondingly substituted monomer components.

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The ITO-coated glass plates 1,2 are used for the cell can have a special orientation layer 6, 7, which aligns the polymer molecule in a preferred direction parallel to the plate surface or at a given tilt angle thereto.

For the orientation, the polymer was heated, as mentioned, to 80°C (glass temperature $T_g = 19^\circ\text{C}$) between two ITO-coated glass plates having parallel orientated layers. By the application of a 300 Hz
5 alternating current of 100 volt to the polymer layer ($d = 8\ \mu\text{m}$) for a few minutes the cholesteric film was given a planar orientation. The planar orientation was fixed by cooling to below the T_g in the electric field. The cholesteric-isotropic phase transition of the side-chain polymer of formula I lies at $T_c = 165^\circ\text{C}$. The cholesteric phase (induced by the side-
10 chain cholesterol ester) is levorotatory. The wavelength of the selective reflection of the side-chain polymer of formula I lies at 503 nm. The three different side-chain monomers have the following functions: 4-allyloxybenzoic acid cholesterol ester induces a levorotatory cholesteric phase; 4-allyloxybenzoic acid (4-methoxy)phenyl ester reinforces the
15 liquid crystalline properties of the LC side-chain copolymer; 1-(3-butenyl)-4 α -(trans-4-pentylcyclohexyl)-1 β -cyclohexanecarbonitrile induces the negative dielectric anisotropy.

Once the orientation has been fixed in the glass state, it remains
20 even after the current has been switched off and is permanently stable.

Instead of providing the plates having ITO coatings and applying a voltage thereto, it is also possible to bring the polymer layer for the required time into an electric field produced away from it, for example
25 in the form of a corona discharge, or to allow it to move through such a field.

As is known, in liquid crystal displays containing highly twisted nematic crystals, the so-called supertwist LCDs, compensation layers
30 are used to avoid the undesired coloration in the switched-off state. Such a compensation layer is usually an additional highly twisted TN layer, which requires a third limiting wall with the corresponding orientation layers, although without electrode coating. This manufacturing technique is a very expensive solution and can be replaced by planar
35 orientated chiral liquid crystalline polymer films which are manufactured according to the invention and which are affixed to the actual display cells.

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