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## Molecular Crystals and Liquid Crystals

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## Liquid Crystal Orientation on Various Surfaces

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# Liquid Crystal Orientation on Various Surfaces

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The liquid crystal orientation on various surfaces were investigated by using the "Liquid Crystal Chromatography," which is useful for observing the true orientation by eliminating impurities in the liquid crystal materials. As the result, it was found that all nematic liquid crystals of Schiff base-, biphenyl-, ester-, PCH-, and azoxy-compounds aligned parallel to the surfaces of  $\text{In}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ , soda-lime glass,  $\text{SiO}_2$ , etc. On the other hand, some of these liquid crystals such as MBBA, 5CB have been known to have a tendency to align perpendicular to the surface like  $\text{In}_2\text{O}_3$ . Then, the reason of this discrepancy was also investigated and it was found that impurities produced by hydrolysis of these liquid crystals had amphiphilic characteristic which induced a perpendicular alignment of these liquid crystals.

## 1 INTRODUCTION

Molecular alignment of nematic liquid crystals on substrate surfaces is practically important and many experimental results have been reported.<sup>1-3</sup> On the other hand, there are several reports which tried to give reasonable explanation for the experimental results from either macroscopic or microscopic point of view.<sup>4-6</sup> The authors have also been investigating the molecular alignment on substrate surfaces regarding dipole moments in the liquid crystal molecule.<sup>7</sup> But in spite of these investigations, any mechanisms which can apply to all of the experimental results have not been found yet. This may be due to the lack of consideration about the effect of impurities contained in the liquid crystal material which affect the liquid crystal molecular alignment.

In this report, a method to remove impurities in the liquid crystal materials by applying the technique of adsorption chromatography is proposed, and true alignment of typical liquid crystals on various clean substrate surfaces are investigated.

In our paper of the Digest of the Eighth International Liquid Crystal Conference, it was misunderstood that cyanobiphenyl liquid crystals aligned perpendicular on some substrates. But, we confirmed lately that these liquid crystals aligned parallel to the substrates. These cyanobiphenyl and some Schiff base liquid crystals have generally been known to have a tendency to align perpendicular on a particular substrate. Then, the cause of these tendencies are also investigated.

## 2 EXPERIMENT

### 2.1 Elimination of impurities applying the principle of adsorption chromatography

Adsorption chromatography is generally used to separate solute from the solvent according to the adsorbing ability of the solute.<sup>8</sup> This phenomenon can be applied to the elimination of impurities in liquid crystal material by using the cell shown in Figure 1. Liquid crystal materials are introduced by capillary action from one side of it. We will call this method liquid crystal chromatography (abbreviated as LC-chromatography). This LC-chromatography is compared with the well-known thin layer chromatography (abbreviated as TL-chromatography) in Table I. Both of these chromatographic systems consist of adsorbent, solute and solvent; the stronger the polarity of the adsorbent and the solute become, the less the solute can be along the drawn flow of the solvent. In the TL-chromatography, the characteristics of the solute are known to be described by the ratio  $R$  of migration length of the solute to that of the solvent, i.e.  $R = b/a$  as shown in Figure 2(a). The same idea can be applied to the LC-chromatographic system as is shown in Figure 2(b). In this case,  $R$  is also defined by  $R = b/a$  but  $a$  is the length between the induction hole and the

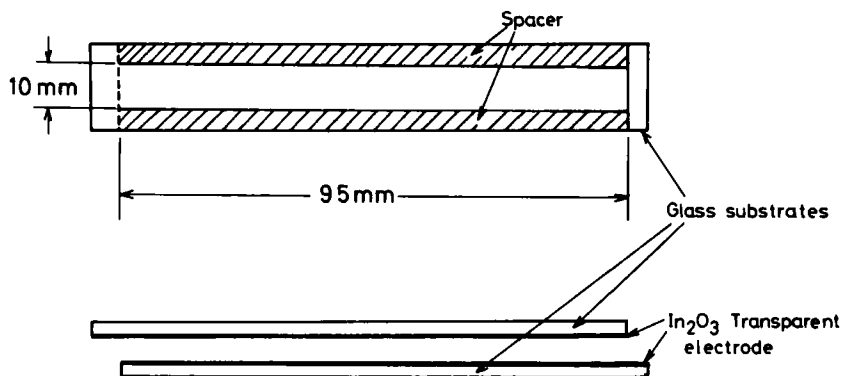


FIGURE 1 Structure of a LC-chromatographic cell.

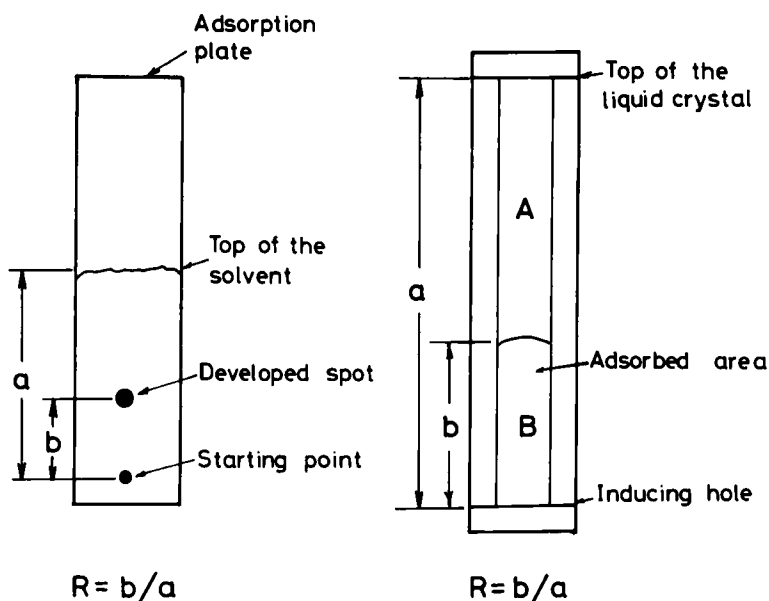
TABLE I

Comparison between TL-chromatography and LC-chromatography

	TL-chromatography	LC-chromatography
Adsorbent	powder of aluminum oxide or silica gel	substrate surface
Solute	composite material	impurities
Solvent	organic solvent	pure LC

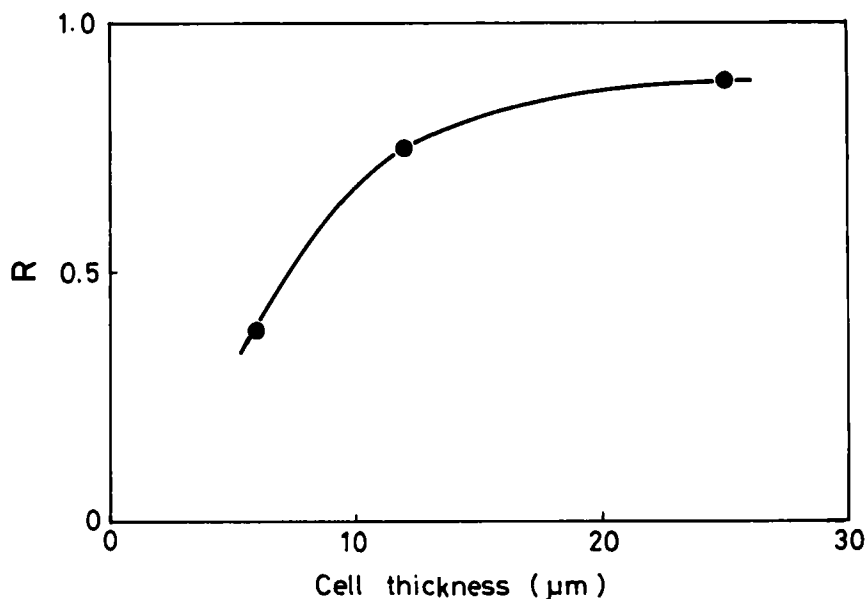
top of the liquid crystal and  $b$  is the length between the induction hole and the top of the adsorbed area B where the molecular alignment is different from that of area A.

In LC-chromatography, the adsorbent is only the flat surface of the substrate and solute i.e. impurity is the supplied continuously. Therefore,  $R$  depends also on the cell thickness and the impurity concentration of the liquid crystal material. Cell thickness dependence of  $R$  is shown in Figure 3. The solute is  $7.0 \times 10^{-2}$  mol% of *n*-octadecylamine (abbreviated as ODA) pro-



(a) TL-chromatography (b) LC-chromatography

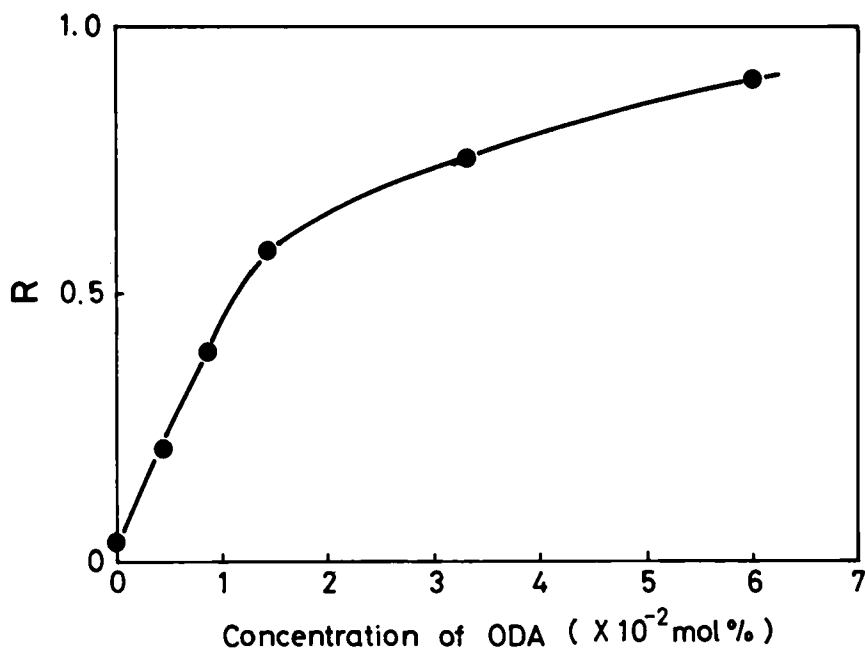
FIGURE 2 Comparison between the TL-chromatography and the LC-chromatography.

FIGURE 3 Cell thickness dependence of  $R$ .

duced by Tokyo Kasei Kogyo Co., Ltd. and the solvent i.e. liquid crystal is pentyloxy-cyanobiphenyl purchased from Chisso Corporation. Substrate is soda-lime glass with  $\text{In}_2\text{O}_3$  transparent electrode produced by Matsuzaki Shinkuu Himaku Kogyo Co., Ltd. This figure shows that the thinner cell is preferred in removing impurity in the liquid crystal. Figure 4 shows the dependence of  $R$  on the concentration of ODA, where the cell thickness is  $6\ \mu\text{m}$ . The fact that  $R$  is 0.05 without the addition of ODA indicates the existence of some impurities in the liquid crystal material. It is seen that  $R$  increases with the increase of the concentration of ODA. Therefore, relative concentration of an impurity can be known from the value  $R$ . In the experiments, molecular alignments on various substrates are investigated by using the  $6\ \mu\text{m}$  thick LC-chromatographic cell.

## 2.2 Liquid crystal alignment on various substrates

Table II shows molecular structures of liquid crystals used in the experiment. Schiff base liquid crystals are synthesized from *para* derivatives of benzaldehydes and anilines. These are purified by vacuum distillation and recrystallization. The other liquid crystals were purchased from companies referred to in the table. Table III shows substrates on which molecular alignments are investigated. Aluminum, gold and silicon oxide are evaporated from resistance-heated source at a pressure of about  $5 \times 10^{-6}$  Torr.  $\text{In}_2\text{O}_3$  coated glass is the

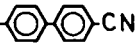
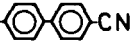
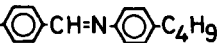
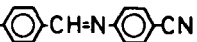
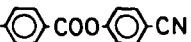
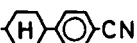
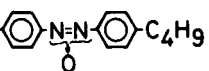
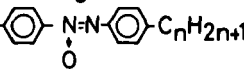
FIGURE 4 ODA concentration dependence of  $R$ .

same one as described in a previous section. The  $\text{SiO}_2$  film is prepared as follows. First, the compound, silicon dioxide (Si-85000 of Tokyo Ohka Kogyo Co., Ltd.), is deposited by dipping a substrate in the 50 vol% ethanol solution. Then, the substrate is heat treated at  $150^\circ\text{C}$  for 15 minutes followed by  $550^\circ\text{C}$  for 15 minutes. All these substrates used in the experiments are cleaned by argon ion bombardment. Table IV shows molecular alignments of heptylcyanobiphenyl (I-7) on the various substrates mentioned above. In this table, "Molecular alignment" for the "LC-chromatography" indicates alignments observed at the upper part of the LC-chromatography (region A in Figure 2(b)), where the true alignment independent of impurities can be obtained, because adsorptive impurities are removed at the lower part (region B in Figure 2(b)). "Molecular alignment" for the "Usual method" indicates alignments observed by the following method. A drop of liquid crystal material is placed on the bottom substrate and then it is covered by the top substrate with a spacer of  $12\ \mu\text{m}$  thick Mylar film.

Polarity of the substrates can be also analyzed by using the LC-chromatographic cell as follows: A monolayer of amphiphilic compound is previously adsorbed at a small area near the entrance hole. Then a pure liquid crystal is introduced into the cell by which the adsorbed region of the amphiphilic compound is shifted. The shift length is closely related to the polarity of the sub-

TABLE II

Liquid crystals used in the experiment

Type	Molecular structure	Symbol Type-(n)	Source
Biphenyl	$C_nH_{2n+1}$ -  -CN	I-5,7	C
Biphenyl	$C_nH_{2n+1}O$ -  -CN	II-5	C
Shiff base	$C_nH_{2n+1}O$ -  -C <sub>4</sub> H <sub>9</sub>	III-1	L
Shiff base	$C_nH_{2n+1}O$ -  -CN	IV-2,4	L
Ester	$C_nH_{2n+1}$ -  -CN	V-5,7	R
PCH	$C_nH_{2n+1}$ -  -CN	VI-5	C
Azoxy	$C_nH_{2n+1}O$ -  -C <sub>4</sub> H <sub>9</sub>	VII-1	M
Azoxy	$C_nH_{2n+1}$ -  -C <sub>n</sub> H <sub>2n+1</sub>	VIII-4,6	F

Source: C Chisso Corp.

L Synthesized in our laboratory

R F. Hoffman-La Roche Co., Ltd.

M E. Merck

F Fuji Color Co., Ltd.

TABLE III

Substrates used in the experiment

1. In<sub>2</sub>O<sub>3</sub> film
2. Evaporated Al film
- 3a. Heat-treated soda-lime glass (400°C, 30 min)
- 3b. Untreated soda-lime glass
- 3c. Soda-lime glass treated by acid (20% HNO<sub>3</sub>, 80°C, 2 hour)
4. Pyrex glass
5. SiO<sub>2</sub> film
6. Evaporated SiO film
7. Evaporated Au film

TABLE IV

Molecular alignment of hexylcyanobiphenyl (I-7)

Substrate	Molecular alignment		Polarity
	LC-chromatography	Usual method	
1		⊥	strong
2		⊥	strong
3a		⊥	strong
3b		●	strong ~ medium
3c			medium
4			medium
5			medium
6			medium
7			weak

⊥: Perpendicular alignment

●: Undistinguished alignment

||: Parallel alignment

strate. By this method, using stearyltrimethylammonium chloride (produced by Tokyo Kasei Kogyo Co., Ltd.) as an amphiphilic compound, and pentyloxycyanobiphenyl as a liquid crystal, polarity of the substrate was estimated and classified into three groups. Substrates of No. 1, 2 and 3a have strong polarity, substrates of No. 3c, 4, 5 and 6 have medium polarity and substrate No. 7 has fairly weak polarity as shown in Table IV. It is seen that the polarity of the substrate of soda-lime glass changes with the treatments. On the other hand, we have measured the sodium ion density of the soda-lime glass by the atomic absorption method. The result was that the sodium ion density at the surface of the glass without special treatment was rather low in comparison with that in the bulk. As for the glass treated by acid, the ion density is still lower than that of the untreated one. But, when these glasses were heat treated, sodium ion migrated from the bulk to the surface and it made the surface polar. Comparing these results with the strength of polarity as shown in Table IV, it can be considered that the sodium ion increases the polarity of the surface.

It is recognized from Table IV that the perpendicular alignment in the "Usual method" can be seen for the surface with the strong polarity.

Next, molecular alignments of various liquid crystals on  $\text{In}_2\text{O}_3$  with strong polarity are investigated. The results are shown in Table V. Some liquid crystals align perpendicular in the "Usual method," while no liquid crystals align perpendicular in the "LC-chromatography." Therefore, it can be concluded that true alignments of these liquid crystals are parallel to the clean inorganic flat surfaces.

The liquid crystals which align perpendicular in the "Usual method" are alkylcyanobiphenyl, alkyloxycyanobiphenyl and some of Schiff base com-



### Liquid crystal alignments on $\text{In}_2\text{O}_3$ surface

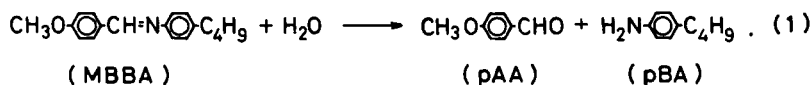
Liquid crystal	LC-Chromatography	Usual method
I-5		⊥
I-7		⊥
II-5		⊥
III-1		⊥
IV-2		(  )
IV-4		⊥
V-5		
V-7		(⊥)
VI-5		
VII-1		
VIII-4		
VIII-6		

⊥: Perpendicular alignment  
(⊥): Nearly perpendicular alignment  
||: Parallel alignment  
(||): Nearly parallel alignment

pounds. These liquid crystals have been known to have a tendency to align perpendicular,<sup>1,9</sup> the reason for which is discussed in the succeeding section.

### 2.3 Degradation of Schiff base liquid crystal and its effect on molecular alignment

It has been reported that *p*-methoxybenzylidene-*p*-*n*-butylaniline (MBBA) aligns perpendicularly on the surface of carefully cleaned SnO<sub>2</sub> or In<sub>2</sub>O<sub>3</sub>.<sup>1</sup> This characteristic of MBBA has been attracting the interests of many researchers, and a lot of arguments have been encountered, while unfortunately the effect of impurities on molecular alignment was not considered. As is described previously, perfectly pure liquid crystal can hardly be obtained by usual purification method. In addition, MBBA is easily dissolved into *p*-anisaldehyde (pAA) and *p*-*n*-butylaniline (pBA) by water as follows:<sup>10</sup>



### Scheme 1

The homeotropic alignment of MBBA mentioned above is considered to be induced by products formed by hydrolysis. It is known that there is the linear relationship between the concentration of these products and the nematic to isotropic transition temperature  $T_c$ ,<sup>11</sup> so that  $T_c$  can be used as a measure of the

purity of the liquid crystal. Then, samples with various  $T_c$  are prepared by exposing the vacuum distilled MBBA to high humidity atmosphere. These samples are introduced into the LC-chromatographic cell and the molecular alignments are observed. The results are shown in Figure 5. It is seen that a sample with low  $T_c$  has three regions i.e. the upper and lower parallel aligned regions, ① and ③, and the middle perpendicular aligned region, ②. The sample with  $T_c = 30^\circ\text{C}$  was analyzed by the TL-chromatography and was found to contain pBA and pAA by comparing their  $R$  values with those of pure compounds. It is considered from comparison of the LC- and the TL-chromatography that the alignment in the region ① in Figure 5 is due to MBBA itself and those in the region ② and ③ are induced by pBA and pAA, respectively. To confirm the effect of these materials on the molecular alignment, pBA and pAA are added to di-butyl-azoxybenzene (BAB) produced by Fuji Color Co., Ltd. which aligns parallel to the  $\text{In}_2\text{O}_3$  surface by itself. As a result, it is confirmed that BAB doped with 1 wt% of pAA aligns parallel but the one with 1 wt% of pBA aligns perpendicular to the surface of  $\text{In}_2\text{O}_3$ .

This pBA is a kind of amphiphilic compound and has polar substituent amine in one end of the molecule, and non-polar substituent aromatic ring and  $n$ -alkyl chain in the other. Generally, the amphiphilic compound adsorbs perpendicularly to the polar surface because of relatively strong interaction between polar group of the compound and the polar surface. Therefore, when

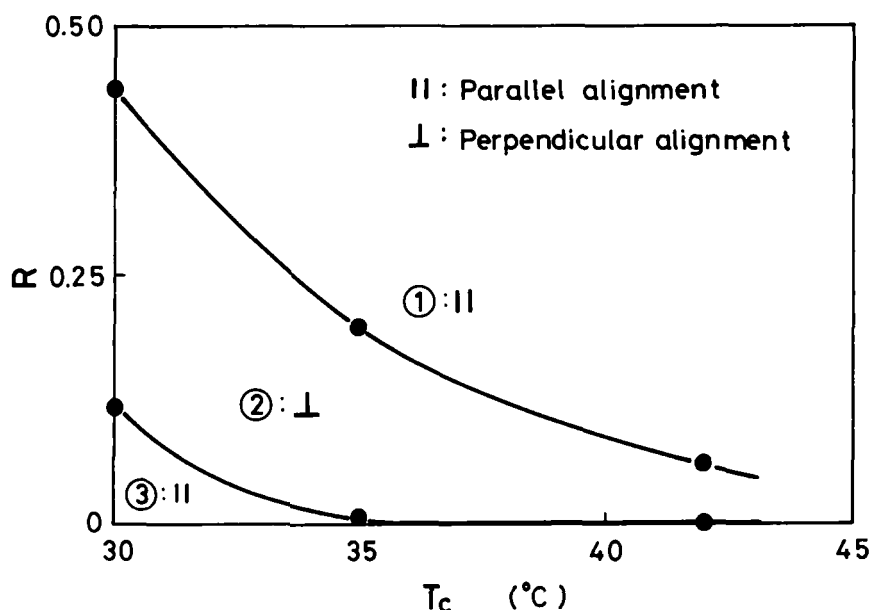
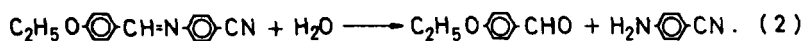


FIGURE 5  $T_c$  dependence of  $R$  in the case of degraded MBBA (III-1).

this kind of material is contained in liquid crystal material, perpendicular alignment is induced.

On the other hand, there are some Schiff base compounds such as IV-2 does not align perpendicular even after degradation. The reason can be explained as follows. This liquid crystal is dissolved according to the following reaction:

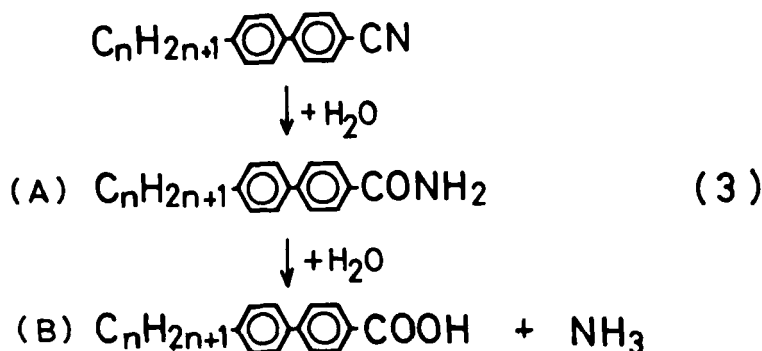


Scheme 2

But in this case, both products are not amphiphilic materials.

#### 2.4 Degradation of cyanobiphenyl liquid crystal and its effect on molecular alignment

It has been known that cyanobiphenyl liquid crystals are stable to moisture contrary to the previously mentioned Schiff base liquid crystals, while they are unstable to ultraviolet light.<sup>12</sup> Authors have also confirmed these characteristics by the following experiment. Heptylcyanobiphenyl in a small bottle of Pyrex glass was exposed to the light of an ultraviolet fluorescent lamp (FL15BLB of Sankyo Denki Co., Ltd., 15 watt, the emission peak wavelength is 355 nm) for several hours. Then it was analyzed by the LC-chromatography. The result is shown in Figure 6 by solid line. It is seen that *R* increases with increase of irradiation time. On the other hand, the same liquid crystal kept in the dark place did not change as is shown by dashed line. These results indicate that ultraviolet light (abbreviated as UV—light) is necessary for the degradation. The degradation of cyanobiphenyl liquid crystals is considered as follows:



Scheme 3

First reaction produces impurity (A) by hydrolysis. The succeeding reaction produces impurity (B) by hydrolysis also. Next, a new heptylcyanobiphenyl (I-7) was introduced into the LC-chromatographic cell, followed by exposing

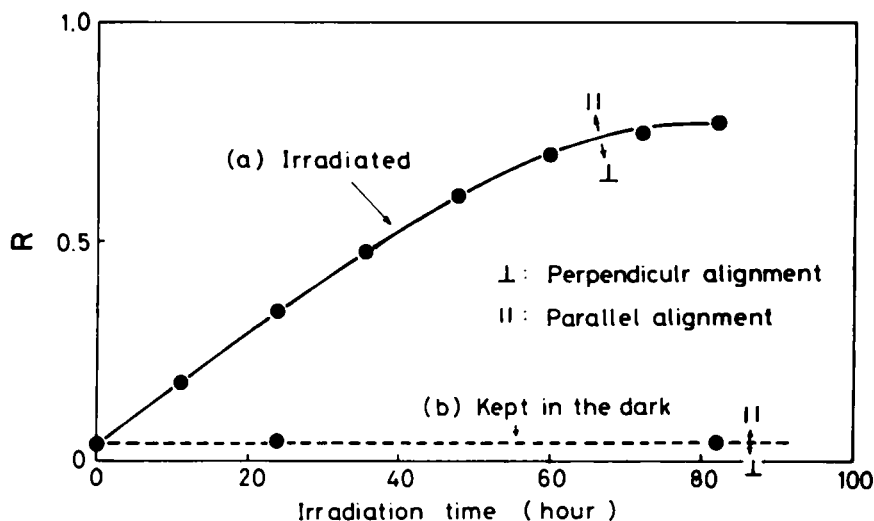


FIGURE 6 Dependence of  $R$  in a case of hexylcyanobiphenyl (I-7) on the irradiation time of UV-light.

to UV-light. It was found that the molecular alignment gradually changed to perpendicular and  $R$  increased as shown in Figure 7. But, it saturated after 10 hours irradiation. This fact indicates that the degradation is not occurred by only UV-light. It is considered that the perpendicular alignment in this cell is due to the cooperation of water adsorbed on the surface and UV-light.

To confirm the degradation reaction and to analyze the impurities, two samples were prepared. One of them was heptylcyanobiphenyl (sample I) and the other was the one doped with water and exposed to UV-light for about 100 hours (sample II). These samples were analyzed by LC—chromatography and it was found that  $R$  of the LC-chromatography for samples I and II were respectively 0.04 and 0.82, by which the sample II was confirmed to be degraded. This sample II appeared pale yellow, while sample I was white. By using TL-chromatography, we found three materials are contained in sample II. Developed spots are denoted by (A), (B) and (C) from the top of the plate. Table VI shows corresponding  $R$  values, fluorescent color and brightness of each spot under irradiation of Black Light (SBL-100B of Stanley Electric Co., Ltd.). The existence of carboxyl group of product (B) was checked by spraying with the basic solution of bromochresole-green (produced by Tokyo Kasei Kogyo Co., Ltd.). The result is also shown at the right hand side of the Table VI. These results are summarized as follows:

- (1) Spot (A) of both samples corresponds to a liquid crystal, because pure liquid crystal shows only spot (A) and  $R$  values and fluorescent colors of both of the samples agree with each other.

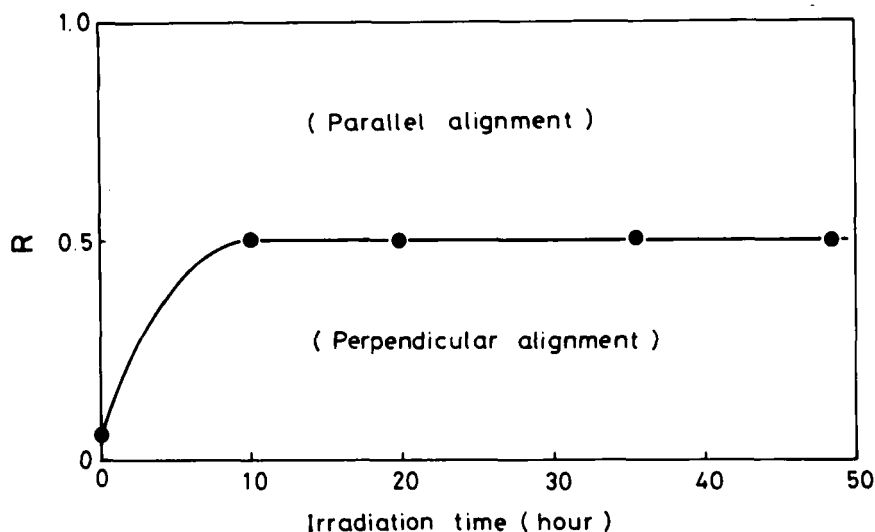


FIGURE 7 Dependence of  $R$  on irradiation time of UV-light, where the cell was irradiated after introducing liquid crystal.

- (2) Spots (B) and (C) corresponds to impurities, which do not have a carboxyl group.

For obtaining further information about impurities, infrared absorption was measured by an infrared spectrophotometer (Model 285 made by Hitachi Ltd.). Each sample sandwiched between 0.5 mm thick Si-wafers. Thickness of the sample was controlled to 50  $\mu\text{m}$  by Mylar spacer. Transmission spectra of sample I and II are compared in Figure 8. Absorbing bands corresponding to substituents in the impurities (A) and (B) are also shown in the figure for comparison. If these materials are contained in sample II, transmission of this region should be lower in comparison with the sample I. It is clear that sample II has relatively strong absorption at  $1600 \sim 1750 \text{ cm}^{-1}$  compared with the sample I, which correspond to absorption band of the  $-\text{CONH}_2$  and/or

TABLE VI  
Results of TL-chromatography (developed by trichloroethylene)

Sample	R	Fluorescence		Existence of carboxyl group
		Color	Brightness	
I	(A) 0.66	purple	dark	no
II	(A) 0.66	purple	dark	no
	(B) 0.32	blue	bright	no
	(C) $\sim 0$	blue	bright	no

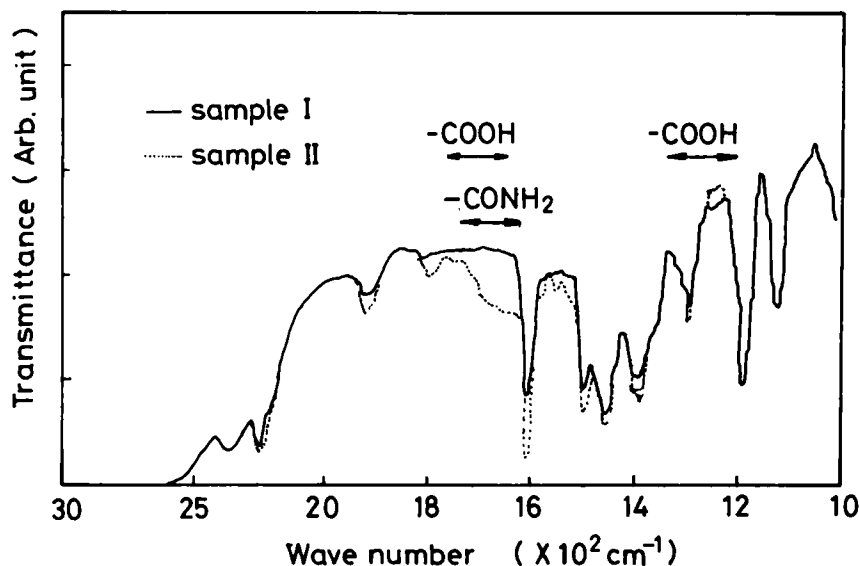


FIGURE 8 Comparison of infrared spectra between sample I and II.

—COOH. Taking into account the result (2) mentioned above, this absorption is considered to correspond to —CONH<sub>2</sub>. The other compounds could not be confirmed.

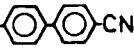
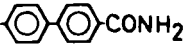

Throughout these experimental results, the detected impurity is considered to be the compound (A) in Eq. 3, while the results of the TL-chromatography indicates also existence of the other impurity without carboxyl group, which is not identified yet.

Finally, effect of impurities on the molecular alignment of liquid crystals on the In<sub>2</sub>O<sub>3</sub> surface is investigated as follows. *n*-octadecanonitrile (ODN), *n*-octadecylamine (ODA) and stearic acid (SA) shown in Table VII, whose structures respectively resemble the alkylcyanobiphenyl and its degraded products (A) and (B), were respectively added to di-butylazoxybenzene which itself aligns parallel to the surface. Then the alignment of the liquid crystal on the surface of In<sub>2</sub>O<sub>3</sub> were examined. We found that ODA with the amino group and SA with the carboxyl group have the ability to align the liquid crystal perpendicularly. On the other hand ODN with the cyano group, which corresponds to the alkylcyanobiphenyl liquid crystal, has no such abilities.

By the way, phenylcyclohexane (PCH) aligns parallel to the In<sub>2</sub>O<sub>3</sub> surface as is shown in Table V though it resembles alkylcyanobiphenyl in structure. Therefore, PCH was exposed to the UV-light under the same condition as that of the hexylcyanobiphenyl mentioned above. Then it was introduced to the LC-chromatographic cell. The dependence of *R* on the exposed time is shown in Figure 9, which indicates that this liquid crystal is rather stable to UV-light

TABLE VII

Aligning effect of additives corresponding to liquid crystal and impurities (A) and (B)

Additive	Corresponding material	Induced alignment
n-Octadecanonitrile (ODN) $C_{17}H_{35}CN$	Liquid crystal $C_nH_{2n+1}$ -  -CN	
n-Octadecylamine (ODA) $C_{18}H_{37}NH_2$	Impurity (A) $C_nH_{2n+1}$ -  -CONH <sub>2</sub>	⊥
Stearic acid (SA) $C_{17}H_{35}COOH$	Impurity (B) $C_nH_{2n+1}$ -  -COOH	⊥

|| : Parallel alignment  
⊥ : Perpendicular alignment

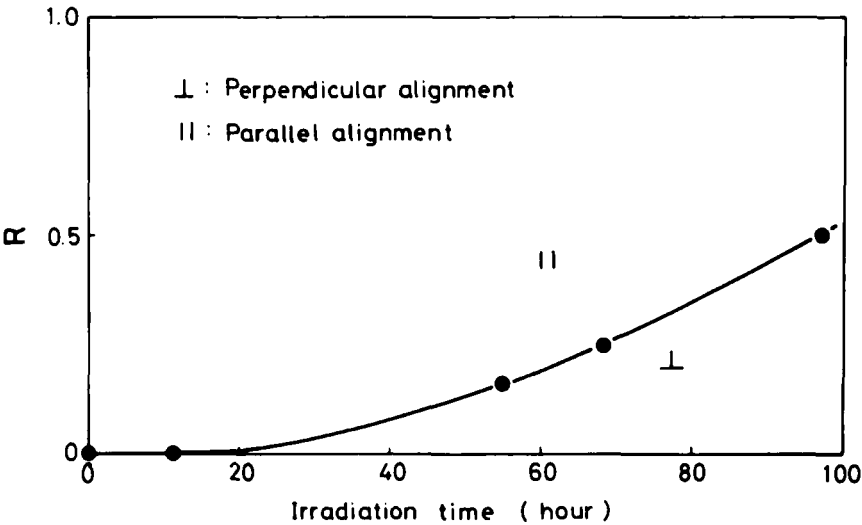


FIGURE 9 Dependence of *R* in the case of PCH(VI-5) on the irradiation time of UV-light.

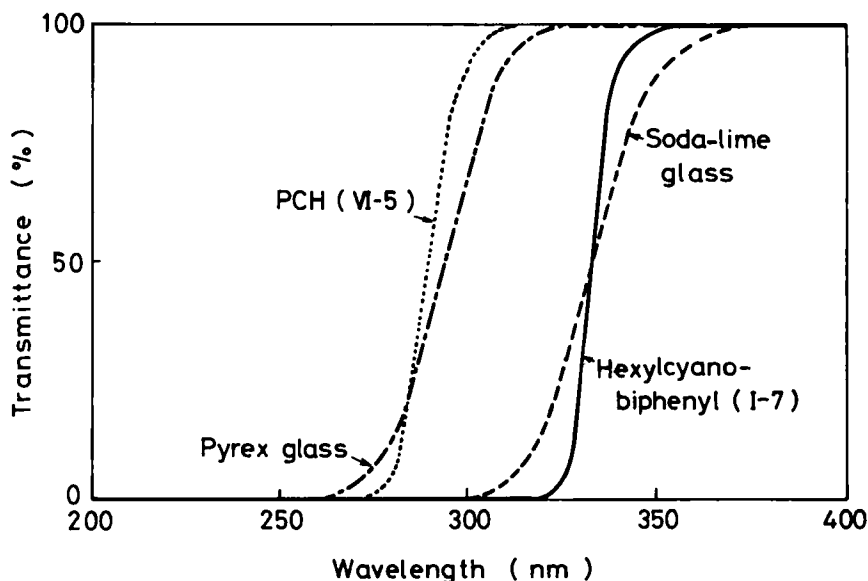


FIGURE 10 UV-transmission spectra of PCH(VI-5), hexylcyanobiphenyl (I-7) and glasses.

in comparison with the hexylcyanobiphenyl shown in Figure 6. This result can be explained by the fact that the absorption edge of PCH is shorter than those of soda-lime glass or Pyrex glass as shown in Figure 10 and hence this liquid crystal is protected from UV-light.

### 3 CONCLUSION

When the liquid crystal molecular alignment is investigated, attention should be placed on the effect of impurities. Concerning it, the LC-chromatography is proposed for elimination of the impurities in the liquid crystal material, and the results by using this method are proposed. We find all the liquid crystals used in the experiment align parallel to the inorganic substrates, though MBBA and cyanobiphenyl liquid crystals have been reported to have the tendency to align perpendicular. The misunderstanding about the molecular alignment of these liquid crystals is also investigated. It is found that the Schiff base liquid crystals are easily dissolved by water and the cyanobiphenyl liquid crystals are dissolved by the combination of water and UV-light. In the case that these products have an amphiphilic characteristic and the surface is strongly polar, they induce the liquid crystal to align perpendicularly.

Creagh *et al.*<sup>4</sup> and Kahn<sup>5</sup> proposed the model to explain the liquid crystal molecular alignment on substrate surface based on the difference between the



surface energy of the liquid crystal and the critical surface energy of the substrate. On the other hand, experimental results which conflict with these ideas are also reported.<sup>13</sup> But as is shown in this report, liquid crystal alignment on the substrate surface is strongly affected by impurities in the liquid crystal material, so that it will be necessary to reconsider the model of molecular alignments based on the true liquid crystal alignments.

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