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Trifluorinated Liquid Crystals for TFT Displays

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TRIFLUORINATED LIQUID CRYSTALS FOR TFT DISPLAYS

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ABSTRACT 3,4,5-tri-fluorobenzene derivatives with different core structures have been synthesized and their physical properties have been determined. Most of the compounds are nematic with broad existence regions. The materials have large dielectric anisotropy, low viscosity, low optical anisotropy and very good voltage holding ratio and high chemical stability and are valuable components for mixtures to be used in TFT displays.
The good voltage holding ratio of the compounds is related to their high electrical resistivity. In order to reproduce the dependence of the resistivity on the dielectric constant, we derived a general formula. Its derivation is based on the interaction of the impurity ions with the induced and permanent dipoles of the liquid crystal molecules. We are able to show, that ion/ion interactions in highly purified liquid crystals may be neglected.

INTRODUCTION

For use in TFT displays the nematic liquid crystals should have high electrical resistivity (high voltage holding ratio), large dielectric anisotropy, low birefringence, low viscosity and very good chemical and thermal stability[1].
In continuation of former work of the Chisso Research

Part of a plenary lecture at the 15th International Liquid Crystal Conference, Budapest 1994.

Center on 3,4-di-fluorobenzene derivatives[2], the 3,4,5-trifluorobenzene group was used as new moiety and first results were already mentioned by Yamamoto et al.[3]. Here we report in more detail about this substance class, including the materials from Reference 3 as well as additional derivatives and discuss their physical characteristics.

Because of the importance of the electrical resistivity for the voltage holding ratio, by considering the intermolecular interactions of ionic impurities with the dipolar liquid crystal molecules, we derived a formula describing the dependence of the resistivity on the dielectric constant of a material. The comparison with experimental data shows that the formula can reproduce the results. Additionally we are able to show, that in liquid crystals of high electrical resistivity the ion/ion interactions may be neglected.

SYNTHESIS

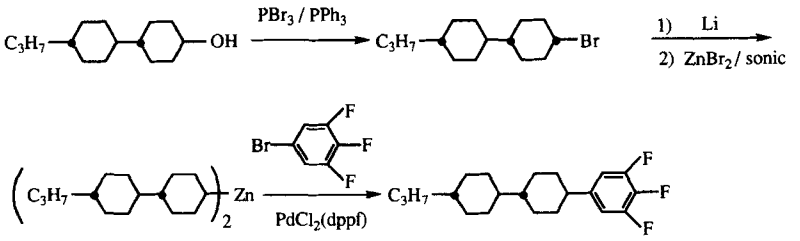
Confirmation of the structures of the products was obtained by ^{1}H-NMR spectroscopy (JEOL EX-90). Transition temperatures were measured using a Mettler FP5 hotstage and control unit in conjunction with a Nikon OPTIPHOT polarizing microscope. The purities of each compound shown in Tables 1.1, 1.2 and 1.3 were checked by GLC analysis (Shimadzu GC-14APF fitted with a 40m CBP1-M50-025 column) and were found to be 99.9% unless stated otherwise.

All the compounds listed in Tables 1.1, 1.2 and 1.3 were synthesized by applying the previously reported catalytic C-C bond formation reactions[4,5,6].

We have described the synthetic procedures for two typical 3,4,5-tri-fluorinated compounds shown in Tables 1.1 and 1.2. The other compounds may be prepared by analogous instructions.

Synthesis of compound:3cF3

1-(trans-4-(trans-4-propylcyclohexyl)cyclohexyl)-3,4,5-trifluorobenzene



cis-4-(trans-4-Propylcyclohexyl)cyclohexylbromide (10g,0.035mol) and zinc bromide (3.92g,0.0175mol) in 20ml of a mixture toluene:tetrahydorofurane (5:1) were placed in the reaction flask under an argon atomsphere. The lithium wire (0.486g,0.07mol) was placed in the flask under the ultrasonic probe. The flask was cooled in an ice bath below 5°C. Stirring with a magnetic bar ensured a homogenous temperature.

The energy level of the sonication was adjusted to the minimum giving the cavitation noise, and a black color was immediately developed. After 30 min irradiation a solution of 1-bromo-3,4,5-trifluorobenzene (3.7g, 17.5mmol) and dichloro-(1,1'-bisdiphenylphosphinoferrocene)palladium (II) (70mg) and 15ml tetrahydorofuran was added dropwise within a few minutes.

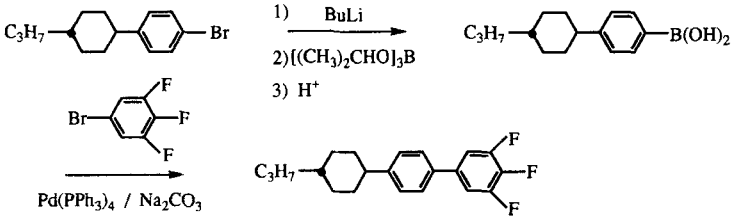
The reaction mixture was stirred at room temperature for 18h. The mixture was poured into saturated aqueous NH_4Cl and the product was extracted with toluene (twice). The combined extracts were washed with water and dried ($MgSO_4$). The solvent was removed in vacuo and the residue was purified by column chromatography [silicagel: hexane:ethyl acetate 10:1] to yield a colorless solid which was recrystallized from ethanol yielding colorless crystals.

Yield :6.12g,(52%).

^{1}H-NMR ($CDCl_3$) δ 0.5-2.1 (26H,m), 2.37(1H,m), 6.79(2H,m)

Synthesis of compound:3eF3

4'-(trans-4-propylcyclohexyl)-3,4,5-trifluorobiphenyl



Butyl-lithium (1.5 mol/l in hexane; 24ml) was added dropwise to a stirred, cooled(-78° C) solution of 1-bromo-4-(trans-4-propylcyclohexyl)benzene (10g, 0.036mol) in dry tetrahydorofurane (30ml) under dry nitrogen. The reaction mixture was maintained under this condition for 3h and then a previously cooled solution of triisopropyl borate (13.6g, 0.072mol) in dry tetrahydorofurane (30ml) was added dropwise at -78° C. The reaction mixture was allowed to warm to room temperature over night and then stirred for 1h in 10% hydrochloric acid(35%). The product was extracted with ether(twice) and the combined ether extracts were washed with water and dried ($MgSO_4$). The solvent was removed in vacuo to yield colorless crystals of 4-(trans-4-propylcyclo hexyl)phenylboronic acid (7.9g, 89%).

A solution of 4-(trans-4-propylcyclohexyl)phenylboronic acid (7.9g, 0.032mol) in ethanol (20ml) was added to a stirred mixture 1-bromo-3,4,5-trifluorobenzene (6.75g, 0.032mol), tetrakistriphenylphosphinepalladium(0) (1.22g, 1.06mmol) in benzene (20ml) and aqueous sodium carbonate (2mol/l, 64ml) at room temperature under dry nitrogen. The stirred mixture was heated under reflux for 20h. The product was extracted with ether (twice) and the combined ether extracts were washed with water and dried ($MgSO_4$). The solvent was removed in vacuo and the residue was purified by column chromatography [silicagel: petroleum fraction (b.p. 40-60°C):dichloromethane 5:1] to yield a color-

less solid which was recrystallized from ethanol yielding colorless crystals.
Yield : 6.82g, (64%).
^{1}H-NMR ($CDCl_3$)δ 0.82-2.0(16H,m), 2.51(1H,m), 7.15(2H,m), 7.27 (2H,d,J=8.4Hz), 7.42 (2H,d,J=8.4Hz)

PROPERTIES

In Tables 1.1 - 1.3 3,4,5-tri-fluorobenzene derivatives with different core structures are presented.
The two ring compounds do not possess nematic phases, but all three ring compounds are nematic, partially in very broad temperature intervals. The comparison with mono- or di-fluorinated compounds (Figure 1) shows that the length-to-breadth ratio of the trifluorinated species is slightly reduced, which explains the reduced clearing temperatures (Table 2).

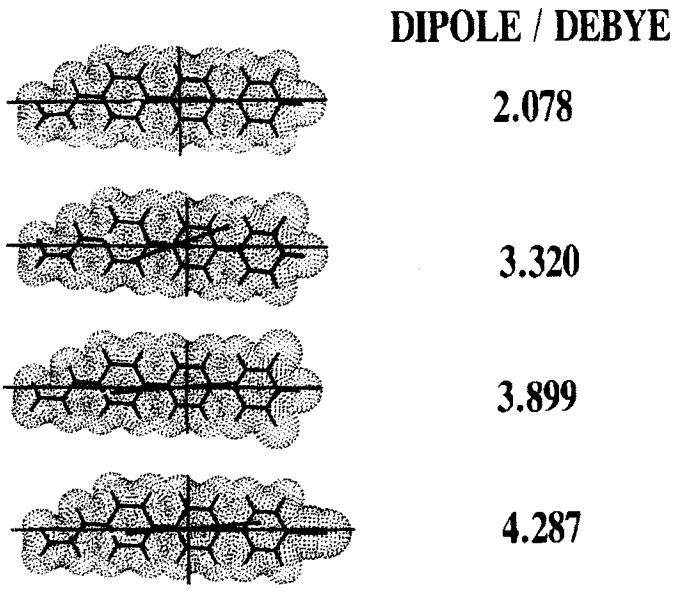


FIGURE 1 . Substituted (trans-4-(trans-4-propylcyclohexyl) cyclohexyl)benzenes. Substituents are from top to bottom: mono-fluoro, di-fluoro, tri-fluoro, cyano. See Color Plate I.

Table 1.1. 3,4,5-F3 Liquid Crystal Compounds

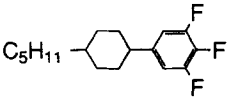
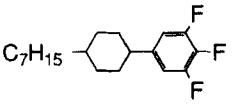
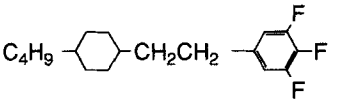
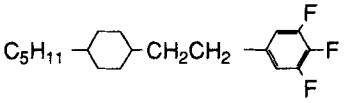
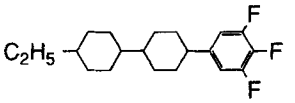
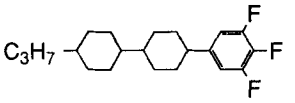
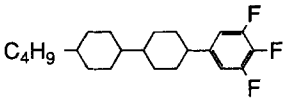
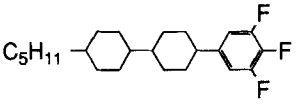
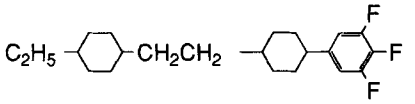
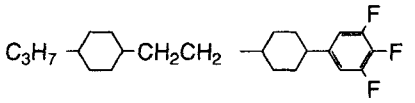
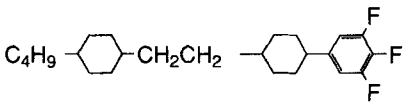
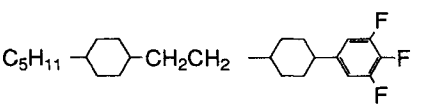
Abbreviation	Structual Formula	Mesophase(℃)	Δ ε	Δn	η [mPa · s]
5aF3		C 16.7 I	—	—	—
7aF3		C 25.6 I	6.8	0.034	—
4bF3		C 8.4 I	5.8	0.024	—
5bF3		C -0.6 I	6.8	0.029	—
2cF3		C 73.0 (N 48.9) I	8.3	0.068	27.1
3cF3		C 64.7 N 93.7 I	8.3	0.073	25.1
4cF3		C 66.7 N 91.3 I	8.3	0.079	26.6
5cF3		C 87.3 N 101.2 I	7.8	0.078	26.6
2dF3		C 64.5 (N 50.0) I	6.8	0.065	31.6
3dF3		C 50.7 N 83.4 I	7.8	0.069	28.6
4dF3		C 61.5 N 83.2 I	8.8	0.064	25.1
5dF3		C 46.5 N 91.1 I	6.3	0.070	30.1

Table 1.2. 3,4,5-F3 Liquid Crystal Compounds

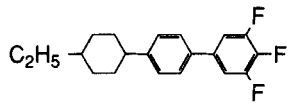
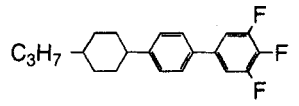
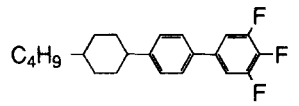
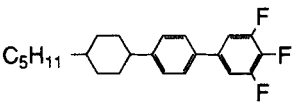
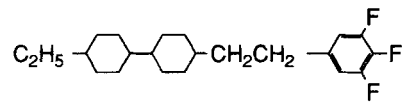
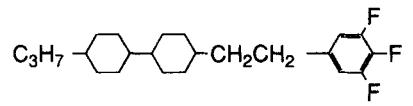
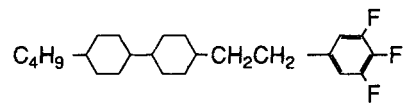
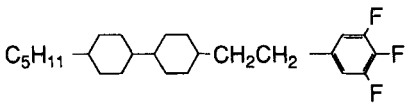
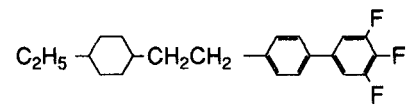
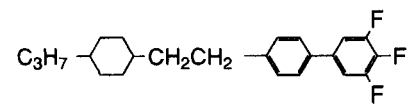
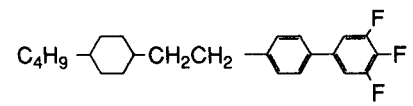
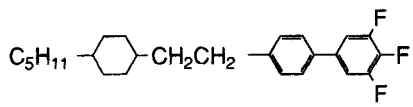
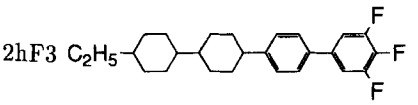
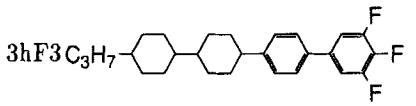
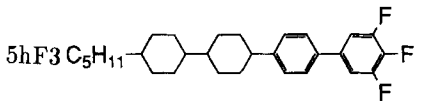
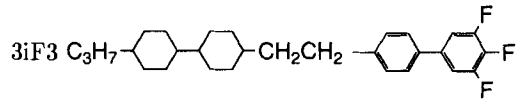
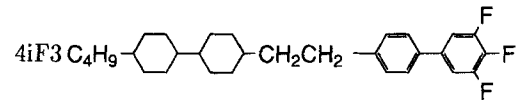
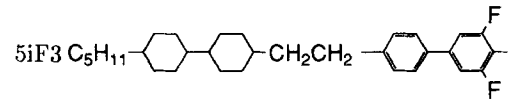
Abbreviation	Structual Formula	Mesophase(℃)	Δ ε	Δn	η [mPa · s]
2eF3		C 46.2 I	12.8	0.124	21.6
3eF3		C 40.7 (N 33.2) I	12.8	0.137	18.6
4eF3		C 37.2 (N 33.2) I	12.3	0.129	21.1
5eF3		C 30.4 N 58.0 I	11.3	0.134	32.1
2fF3		C 65.1 N 65.8 I	8.3	0.069	23.1
3fF3		C 41.8 N 98.3 I	7.3	0.074	31.6
4fF3		C 57.6 N 96.5 I	7.8	0.074	26.6
5fF3		C 54.6 S_B 57.0 N 103.9 I	6.3	0.074	40.1
2gF3		C 43.0 (N37.4) I	—	—	—
3gF3		C 48.0 N 51.8 I	11.8	0.129	16.1
4gF3		C 50.9 (N 50.7) I	11.8	0.124	18.1
5gF3		C 40.2 N 65.2 I	10.8	0.124	18.6

Table 1. 3. 3,4,5-F3 Liquid Crystal Compounds

Abbreviation	Structual Formula	Mesophase(℃)	Δ ε	Δn	η [mPa · s]
2hF3		C 109.3 N 234.0 I	12.8	0.149	56.1
3hF3		C 105.8 N > 250 I	12.6	0.154	57.6
5hF3		C 87.8 N > 250 I	11.3	0.144	51.1
3iF3		C 79.2 N 216.0 I	11.3	0.144	46.6
4iF3		C 83.1 N 210.6 I	10.8	0.139	47.1
5iF3		C 86.1 N 212.5 I	10.8	0.139	45.1

Δε, Δn, η: extrapolated value. Measurement performed using 20wt% solution of 3,4,5-F3 in
* FB-01 (2cF2 : 3cF2 : 5cF2=1 : 1 : 1 mixture, NI 112.8 °C, Δε 4.8, Δn 0.079, η 25.6).
As an exception, 5cF3 measurement was performed using 10wt% solution in FB-01.
Temperature of measurement : Δε 20°C, Δn 25°C, η 20°C.

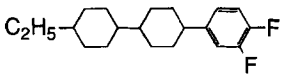
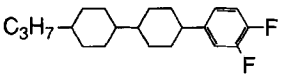
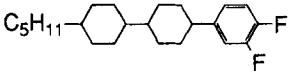
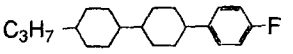
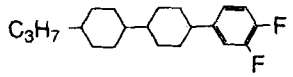
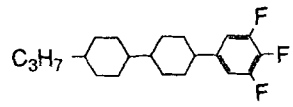
* FB-01 {  33.3mol%
 33.3mol%
 33.3mol%

Table 2. Relationship between transition temperatures and length to-breadth ratio

Abbreviation	Compound	transition temperatures (°C)	length-to-breadth ratio •
3eF		C 87.6 N 155.2 I	3.93
3eF2		C 45.6 N 123.8 I	3.76
3eF3		C 64.7 N 93.7 I	3.62

• length to-breadth ratio was calculated by MOPAC ver.6

All new compounds possess high dielectric anisotropy, low optical birefringence and low viscosity. The discussion of these data will be performed by use of selected examples and comparison with analogous derivatives of the practically important substance classes derived from 3,4-di-fluorobenzene[2] and 4-cyanobenzene[7].

As to be expected, the dielectric anisotropy of the trifluorinated compounds is intermediate between those of the CN-substituted and those of the di-fluorinated compounds (Figure 2). This can be explained by the increased longitudinal component of the dipole in comparison to the mono- and di-fluorinated compounds (Figure 1). The temperature dependence of $\Delta\varepsilon$ is about the same in the substance classes displayed in Figure 2. Compounds with large conjugated core systems, this is biphenyl derivatives (Table 1), have distinctly larger $\Delta\varepsilon$ than benzene derivatives.

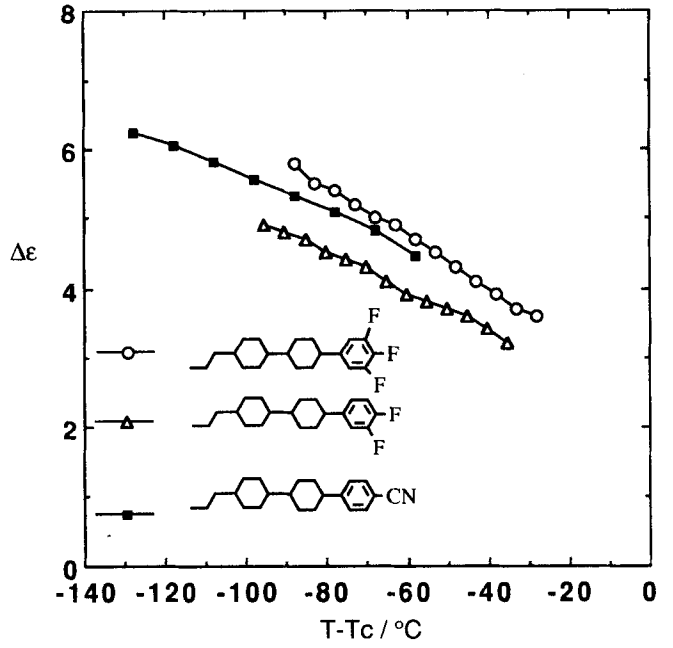


Figure 2 Temperature dependence of dielectric anisotropy(Δε)

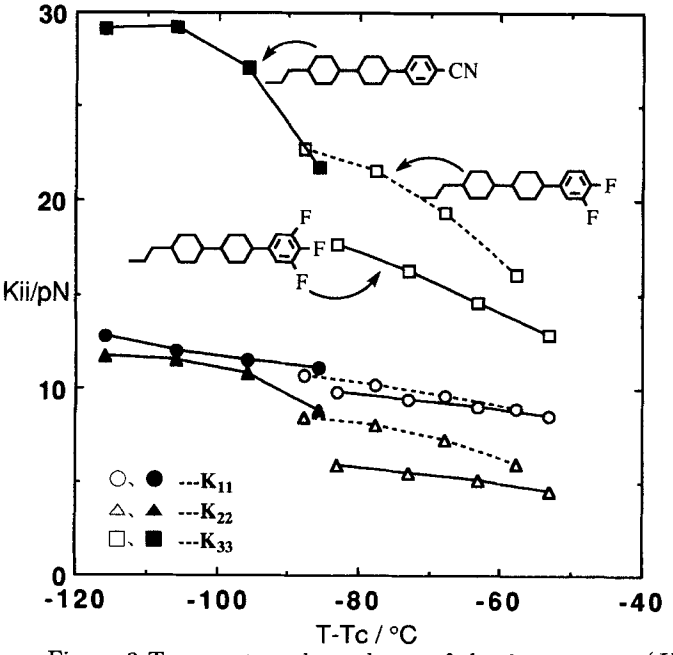


Figure 3 Temperature dependence of elastic constants (K_{ii})

The optical anisotropies of tri-fluorinated compounds are very small and below 0.1, only those of the biphenyl derivatives are slightly higher than 0.1 (Table 1). The order parameters of the tri-fluorinated compounds, because of the diminished length-to- breadth ratio, should be lower than those of the more elongated compounds. This can explain the small optical anisotropies.

The temperature dependence of the elastic constants are shown in figure 3. In all cases there is the relation $K_{33} > K_{11} > K_{22}$. In comparison to the reference compounds, all elastic constants of the trifluorinated compounds are lower. This also holds for $\kappa(\kappa = K_{ii} + (K_{33} - 2K_{22})/4$).

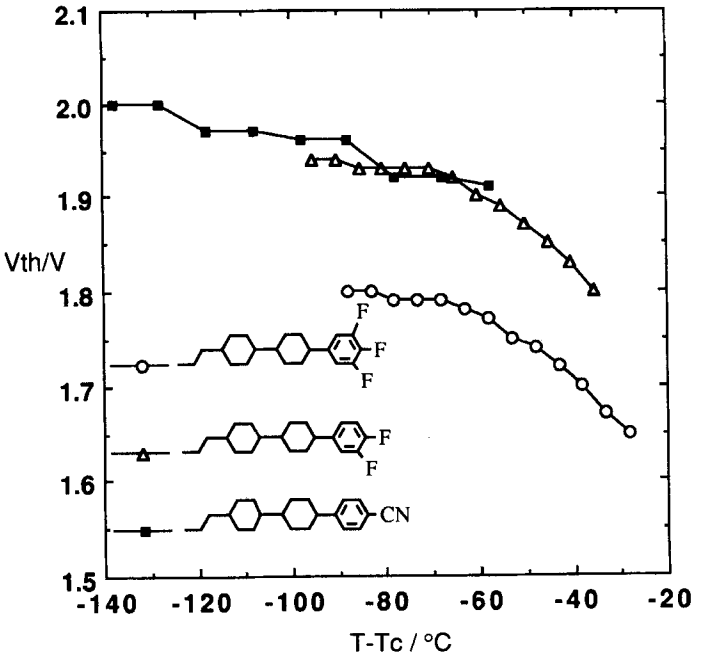


Figure 4 Temperature dependence of threshold voltage(V_{th})

Figure 4 presents the temperature dependence of the threshold voltages, which were measured in conventional TN cells with $\Delta n \cdot d = 0.55\mu m$. The tri-fluorinated compounds show the lowest values. This can be explained by the fact that $\Delta\varepsilon$ of

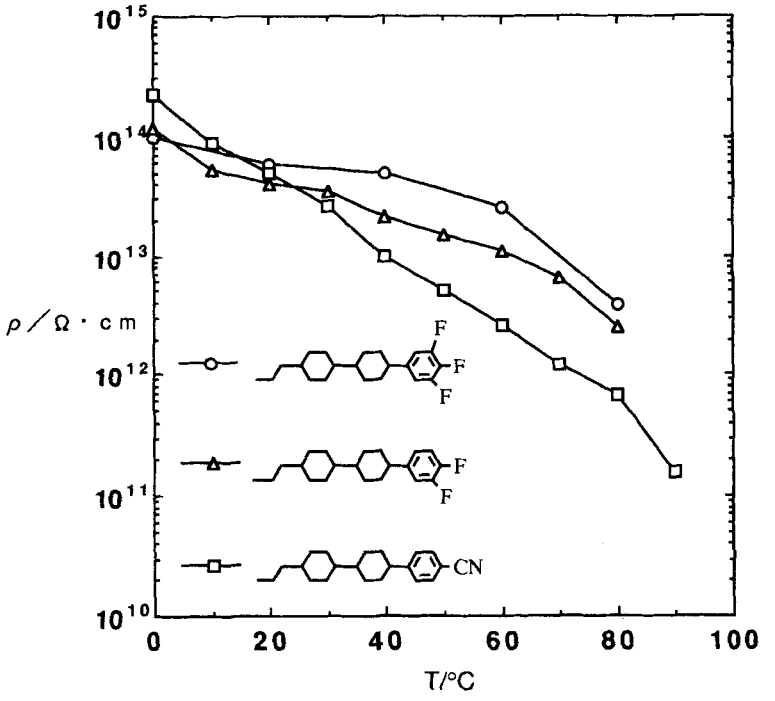


Figure 5 Temperature dependence of specific resistivity(ρ).

them has the largest value, whereas κ is the smallest. These findings suggest that the tri-fluorinated compounds are valuable components for mixtures used in displays driven at low voltage.

The temperature dependence of the specific resistivity is displayed in Figure 5. The fluorinated substances are superior to the cyano substituted compound, which is specially pronounced at elevated temperatures. There is some experimental evidence, that the electrical resistivity in different substance classes is decreasing with increasing dielectric constant, however, we did not find any explanation of this behaviour. In order to understand this effect, in the following section we derive a formula of the dependence of the resistivity on the dielectric constant.

In Figure 6 the temperature dependence of the voltage holding ratios of the three representative compounds is displayed. We see that the data of the fluorinated compounds

are superior to those of the cyano compound, which may be explained by the already discussed differences in the specific resistivities.

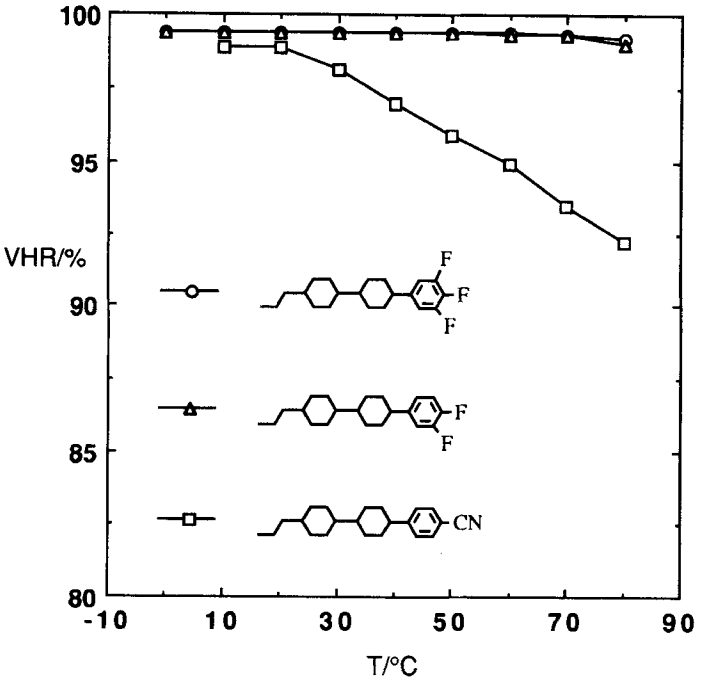


Figure 6 Temperature dependence of voltage holding ratio(VHR)

DEPENDENCE OF THE ELECTRICAL RESISTIVITY ON THE DIELECTRIC CONSTANT

In liquid crystals always certain amounts of impurities exist. They can be of different kind:

1. non-polar, non dissociating (e. g. neighboured homologs of a given compound. Such impurities can be determined by calorimetric investigation or chromatographic methods. They only have minor influence on the electrical properties.

2. polar, completely dissociated. For instance small amounts of inorganic salts, acids or bases. Even small amounts of such impurities strongly influence the electri-

cal resistivity of the material, and therefore easily can be determined by resistivity measurements. Because in many cases they are not soluble in non-polar solvents, they can be removed by recrystallization.

3. polar, partially dissociated

Impurities of this kind may be organic acids, phenols, bases. Their dissociation depends on the dielectric constant (see below). They change the electrical resistivity drastically and can be removed only with large difficulties.

The last of these impurities can already exist in the liquid crystal, but also may be formed in the material due to decomposition in electric fields, thermal or photochemical decomposition.

Weak electrolytes, this is partially dissociated materials, show a dissociation constant which is dependent on the dielectric constant of the solvent.

We consider the most simple dissociation equilibrium

$$KA \rightleftharpoons K^+ + A^- \qquad K_{diss} = c_{K^+}\, c_{A^-}/c_{KA} \tag{1}$$

The standard Gibbs energy of this equilibrium is

$$\Delta G_{diss} = \Delta G_o + \Delta G_{solv} \tag{1a}$$

and the equilibrium constants are

$$K_{diss} = K_o K_{solv} \tag{1b}$$

This means, the total change of the standard Gibbs free energy is divided into a part ΔG_o , which would result from dissociation into non-polar particles (which do not produce strong intermolecular interactions), and a contribution ΔG_{solv}, which considers the solvation of the dissociation products (ions) with the solvent (liquid crystal molecules). The molar solvation energy approximately can be

split into an ion/permanent dipole and an ion/induced dipole contribution:

$$\Delta G_{solv} = U(\text{ion/induced dipole}) + U(\text{ion/dipole}) \quad (2)$$

$$U(\text{ion/induced dipole}) = -2\, N_A\, \alpha_{av} e_o^2 p/\{2\,(4\pi\varepsilon_o r^4)\} \quad (3)$$

N_A = Avogadro's number
α_{av} = average polarizability of the liquid crystal molecule
e_o = electrical elementary charge
ε_o = dielectric constant of vacuum
r = distance of the particles
p = number of nearest neighbours

$$U(\text{ion/dipole}) = -\, N_A e_o^2 \mu^2 p/\{4\pi\varepsilon_o r^4 3kT\} \quad (4)$$

μ = permanent electrical dipole of the liquid crystal molecule
k = Boltzmann's constant

In eq. (4) we introduced the statistical factor 1/2 in comparison to eq. (3), because of the direction dependence of the interaction with the highly anisotropic liquid crystal dipole molecules.
Adding (3) and (4) according to eq. (2), we arrive at

$$\Delta G_{solv} = -N_A e_o^2 p/(4\pi\varepsilon_o r^4)\{\alpha + \mu^2/(3kT)\} \quad (5)$$

The Onsager theory of dielectric polarization delivers[8]

$$\varepsilon - 1 = (NhF/\varepsilon_o)\{\alpha + F\mu^2/3kT\} \quad (6)$$

ε = average dielectric constant of the liquid crystal
N = number density
h, F are factors of the Onsager theory describing the reaction field, both are approximately equal to one.

Comparing eqs. (5) and (6), for a given liquid crystal we find

$$\Delta G_{solv} = \text{const.}\ (\varepsilon - 1) \tag{7}$$

Starting from eq. (1b) and using well known general relations[9], we obtain

$$K_{diss} = \exp\{-(\Delta G_o + \Delta G_{solv})/RT\} = K_o \exp(-\Delta G_{solv}/RT) \tag{8}$$

Inserting eq. (7) yields

$$K_{diss} = K_o \exp\{C'(\varepsilon - 1)\} \tag{9}$$

with $C' = -\text{const}/RT = N_A e_o^2/(4\pi r^4 NhFRT)$

The molar conductivity for weak electrolytes is given by:

$$\Lambda_m = \alpha \Lambda'_m \tag{10}$$

α = degree of dissociation
Λ'_m = conductivity of completely dissociated electrolyte (this is $\alpha = 1$)
The specific resistivity (Ωcm) is

$$\rho = 1/\Lambda_m c \tag{11}$$

c = concentration

The dissociation constant can be expressed in terms of Ostwald's law[9]

$$K_{diss} = \alpha^2 c/(1 - \alpha) \tag{12}$$

$$\alpha = 1/2(K_{diss}/c)\{(1 + 4c/K_{diss})^{1/2} - 1\} \tag{13}$$

Now we calculate the specific resistivity from eq. (11),

inserting Λ_m from (10), α from eq. (13) and K_{diss} from eq. (9)

$$\rho = C'' \exp[-C'(\varepsilon - 1)]\{(1 + 4c/K_o \exp[-C'(\varepsilon-1)])^{1/2} - 1\}^{-1} \quad (14)$$

with $C'' = 2/\Lambda'_m K_o$

We calculated data for eq. (14), using the following parameters:

$T = 300$ K

$r = 0.385 \cdot 10^{-9}$ m (average distance of ions and liquid crystal molecules)

$K_o = 4 \cdot 10^{-16}$

$\Lambda'_m = 5 \cdot 10^{-4}$

$\rho' = 5 \cdot 10^{10}$ Ωm = $5 \cdot 10^{12}$ Ωcm (specific resistivity at complete dissociation)

$c = 3 \cdot 10^{-8}$ mol/l = $3 \cdot 10^{-5}$ mol m^{-3}

$C'' = 10^{19}$ Ωcm = 10^{17} Ωm

The calculated data are plotted in comparison to experimental results from many different liquid crystals in Figure 7. Approximately eq. (14) is able to reproduce the experimental data. In ref. 10 experimental data for the resistivity are presented, which show a similar dependence on the mean dielectric constant. The agreement of calculated and experimental data cannot expected to be very good. In eq. (14) the dissociation constant K_{diss} and the molar conductivity, which are material specific constants, are contained. If we consider different liquid crystals, in principle different impurities (with different constants) may be present. The fact that the experimental data accumulate around a common curve points at impurities with similar dissociation and conductivity properties. For synthesis in many cases phenols or carboxylic acids, which can act as dissociating impurities, are used. If their molar mass and the substitution near to the polar group are not too different, similar constants can be expected.

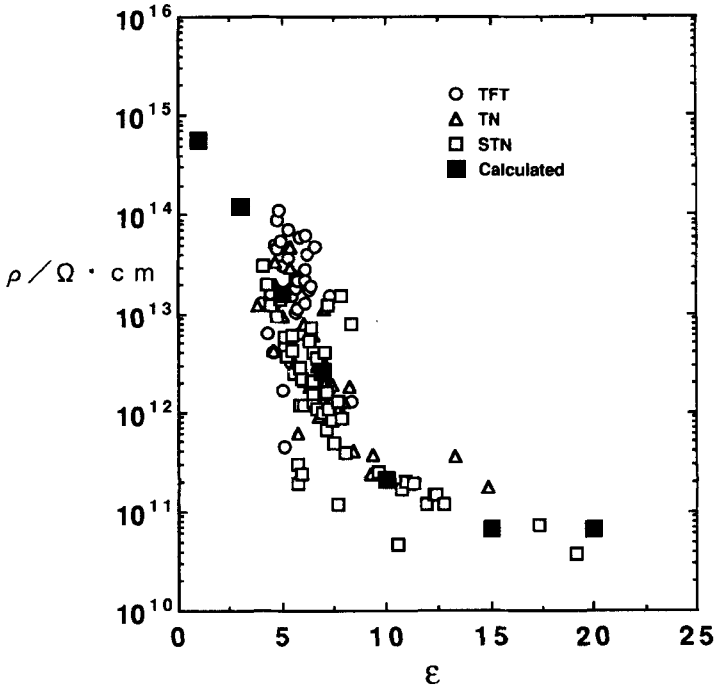


Figure 7 Specfic resistivity vs. average dielectric constant in LC mixture to be used for different display mode.

Coulomb interaction

The dissociation produces ions in the liquid crystalline solution. It is well known that ions cause a long-range interaction, which can be calculated according to Coulomb's law:

$$U(\text{Coulomb}) = N_A z_A z_K e_o^2/(4\pi\varepsilon_o\varepsilon r) \qquad (15)$$

z_A, z_K = charge numbers of the ions

In Reference 11 the following data are given for a typical nematic mixture:

$\rho = 1\cdot10^{12}$ Ωcm , $N = 2\cdot10^{19}$ m^{-3}

From this we calculate $c = 3.3\cdot10^{-8}$ mol/l and the average distance r_{av} of two ions is $r_{av} = 368\cdot10^{-6}$ m

With a typical molecular size of a liquid crystal molecule

Table 3. 3,4,5-F3 LC mixtures for TFT Displays

Mixture	A	B	C	D	E	F	G	Control
SN(°C)	<-40	<-40	<-40	<-40	<-40	<-40	<-40	<-40
NI(°C)	75.9	83.2	81.1	91.3	100.3	109.1	126.4	81.6
η[mPa•s] (20°C)	27.5	28.1	30.4	25.8	26.4	32.2	34.8	25.0
Δn (20°C)	0.0867	0.0807	0.0902	0.0886	0.0802	0.0853	0.0871	0.0813
Δε (20°C)	8.4	7.5	8.4	6.1	6.3	7.0	6.9	4.6
ρ (Ωcm)	$>10^{14}$	$>10^{14}$	$>10^{14}$	$>10^{14}$	$>10^{14}$	$>10^{14}$	$>10^{14}$	$>10^{14}$
V90 (volt)	1.51	1.62	1.62	1.73	1.68	1.85	2.01	1.84
V50 (volt)	1.81	1.96	1.92	2.11	2.07	2.22	2.39	2.17
V10 (volt)	2.22	2.46	2.41	2.58	2.61	2.80	2.99	2.64
Cell width (μm)	5.5	6.3	6.3	5.5	5.6	5.5	5.5	6.3

r_{min} = 2 nm we find, for the relation of the Coulomb interaction with the average neighboured ion and the nearest neighbour, resp.,

$$U_{av}/U_{near} = 5 \cdot 10^{-6} \qquad (16)$$

This means that for small concentrations there is practically no Coulomb interaction between the ions. Only with $\rho < 10^8$ the ratio (16) would exceed 5% .

CONCLUSIONS

All the discussed properties of the tri-fluorinated compounds let expect them to be valuable components for mixtures, specially for use in TFT displays. Table 3 presents some mixtures made from different combinations of tri-fluorinated compounds and additional components, together with a reference mixture based on di-fluorinated compounds. All mixtures have broad nematic existence regions, low viscosities, low optical anisotropies, low threshold voltages and resistivities above 10^{14} Ωcm. Table 3 proves that outstanding mixtures for TFT displays can be formulated using tri-fluorinated compounds.

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