

CHAPTER I

INTRODUCTION

Many organic compounds with molecules having strong shape anisotropy go over from the crystalline phase to the isotropic liquid phase through one or more intermediate phases. These intermediate phases are called mesophases or liquid crystalline phases. They possess some properties of crystals like optical, dielectric and diamagnetic anisotropies and some properties of liquids like the ability to flow or to form a meniscus. The liquid crystals obtained by thermal effects, i.e., either by heating a crystal or by cooling a liquid are called thermotropic liquid crystals. There is another class of liquid crystals obtained by dissolving certain organic compounds in suitable solvents. These are called lyotropic liquid crystals. In this thesis, we are exclusively concerned with thermotropic liquid crystals. For general reviews of liquid crystals we refer to Chandrasekhar (1977), de Gennes (1974) and Priestley et al (1974).

Only those compounds whose molecules possess geometrical anisotropy can exhibit liquid crystalline phases. The molecules have to be either rod-shaped or disc-shaped. Mesophases formed by the latter type of molecules have been discovered only recently (Chandrasekhar et al 1977). All our

studies have been made ON liquid crystals whose molecules are rod-shaped.

1.1 Classification of liquid crystals

Thermotropic liquid crystals of rod-shaped molecules are classified into three groups (Friedel 1922), viz., nematic, cholesteric and smectic.

Nematic liquid crystals. In nematic liquid crystals, the molecules tend to align parallel to a common axis, (Figure 1.1a) denoted by a dimensionless unit vector $\vec{n}$, called the 'director'. This phase is characterised by a high degree of orientational order of the rod-like molecules and no long range translational order. Experiments show that $\vec{n}$ and $-\vec{n}$ are equivalent. A nematic liquid crystal is optically uniaxial, (the optic axis being *along* $\vec{n}$) positive and strongly birefringent.

Cholesteric liquid crystals. These consist of optically active molecules. The cholesteric is similar to the nematic with the essential difference that its structure has a screw axis normal to the director (figure 1.1b). The spiral structure of the cholesterics gives rise to some unique optical properties like selective reflection of circularly polarised light at normal incidence and abnormally high optical rotatory power.

Smectic liquid crystals. In smectic liquid crystals, the molecules are arranged in layers. Depending upon the molecular arrangement within the layers, smectics have been classified

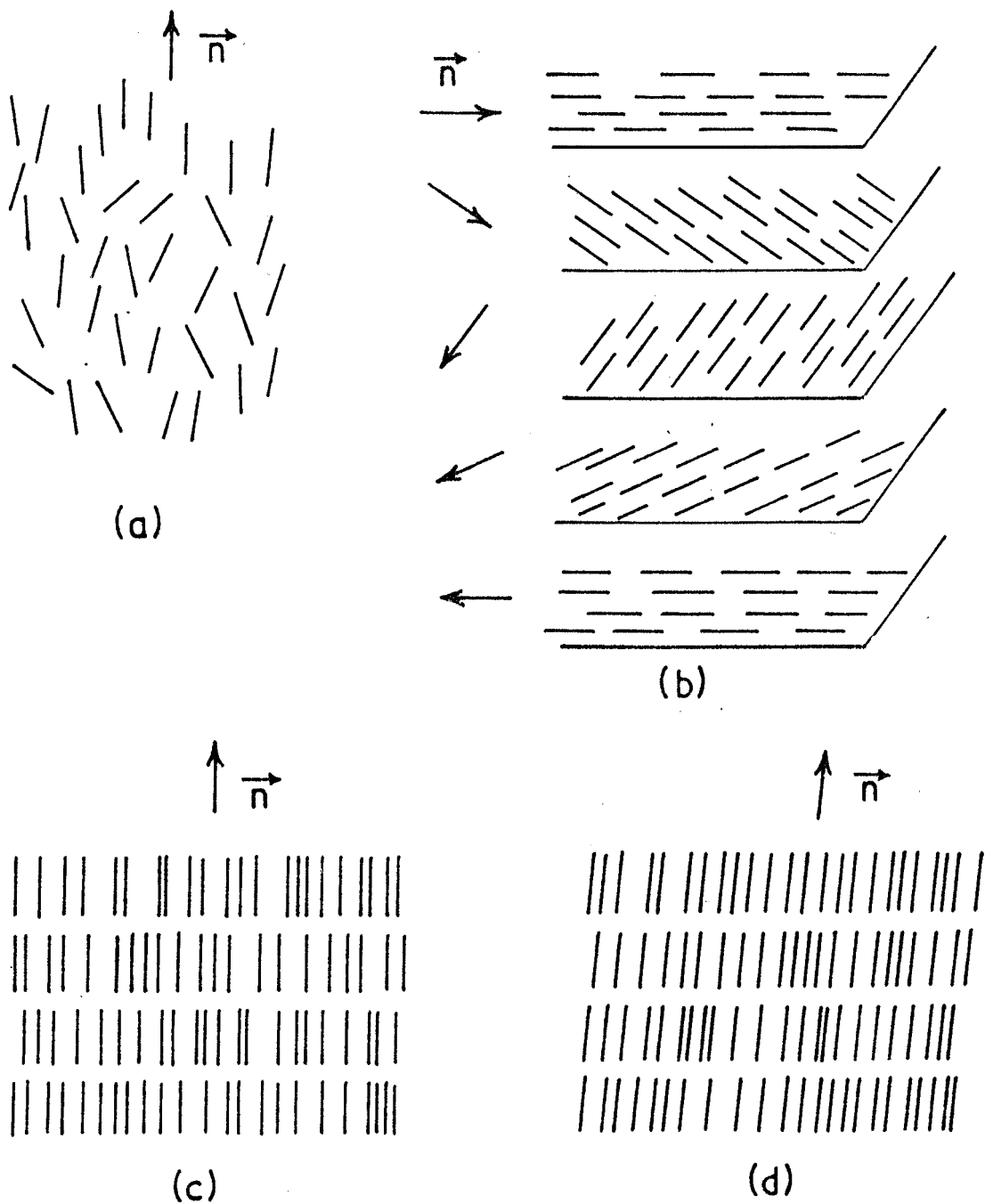


FIGURE 1.1

Schematic diagram showing the molecular arrangement in different types of liquid crystals, viz., (a) nematic, (b) cholesteric, (c) smectic A, and (d) smectic C.

into several types. Among these only two types - smectic A and smectic C - will be of interest to us in the present thesis.

In the smectic A phase (figure 1.1e) there is long range orientational order but no long range positional order within each layer and the director is parallel to the layer normal. Smectic A liquid crystals are optically uniaxial.

Smectic C can be considered as a tilted form of smectic A. In this phase, the molecules in each layer are tilted with respect to the layer normal (figure 1.1d). Smectic C liquid crystals are optically biaxial.

All the characteristic properties of nematic liquid crystals owe their existence to the orientational ordering of the molecules. Hence it is of considerable interest to study how the orientational order parameter of the medium varies with temperature. There are several methods of determining the orientational order parameter. In our work we have used three different methods, viz., (1) by measuring the birefringence of liquid crystals, (2) by infrared (IR) dichroism studies, and (3) by the nuclear magnetic resonance (NMR) technique.

As will be described in a later section liquid crystals possess curvature elasticity (Oseen 1925, Frank 1958). The curvature elastic constants depend strongly on the degree of the long-range orientational order of the liquid crystal. In addition, they throw considerable light on the degree of any

short-range order that may be present in such liquid crystals. We have measured the elastic constants of a few cyano compounds and binary mixtures that show some interesting short-range order effects.

1.2 Alignment of liquid crystals.

In an unaligned nematic sample, $\vec{n}$ varies from point to point. For measuring the anisotropic properties one needs a monodomain sample of the liquid crystal in which $\vec{n}$ is in the same direction throughout the sample. Usually either a homogeneous or a homeotropic alignment is used. A homogeneously aligned sample is one in which the director $\vec{n}$ is unidirectional and parallel to the surfaces enclosing the sample. On the other hand, in a homeotropically aligned sample the director is perpendicular to the enclosing surfaces. Homogeneous alignment is obtained by the unidirectional rubbing of the glass plates (Chatelain 1955) or by vacuum deposition of materials like silicon monoxide at some oblique angle on the substrates (Janning 1972). The homeotropic alignment is usually obtained by treating the glass plates with suitable surfactants like lecithin or cetyl trimethyl ammonium bromide.

1.3 Order parameter

In the case of uniaxial liquid crystals with molecules having the shape of cylindrical rods, the degree of orientational order is specified by that parameter s defined as (Zwetkoff 1942),

$$s = \frac{1}{2}(\overline{3\cos^2 \theta} - 1) \quad (1.1)$$

Here θ is the angle between the long axis of the molecule and the director $\vec{n}$; the bar represents a statistical average over the whole ensemble.

$s = 1$ for a perfectly ordered system and $s = 0$ for the isotropic liquid. In liquid crystals the value of s usually lies between 0.3 and 0.9, the actual value being dependent on the material and its temperature.

When the temperature of a nematic liquid crystal is raised s decreases monotonically and drops to zero discontinuously at the nematic-isotropic transition point. The abrupt drop in the value of s at the nematic-isotropic transition temperature is a consequence of the first order nature of this transition.

The variation of s at the smectic-nematic transition point depends upon the nature of this transition. If it is a first order transition, s shows a discontinuous change at the transition point. On the other hand, if the transition is almost a second order transition, s varies continuously, but it may show a discontinuity in slope (McMillan 1971). When the smectic phase goes over directly to the isotropic phase, a discontinuous change in s is observed at the transition.

1.4 Elastic constants of nematic liquid crystals

In a uniformly oriented monodomain sample of a nematic

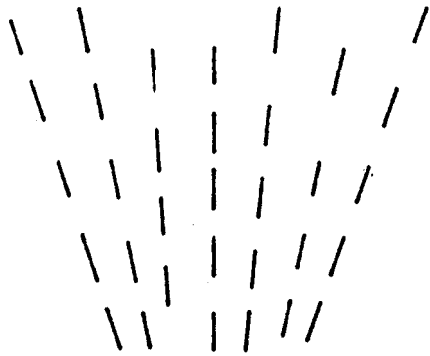
liquid crystal, the director $\vec{n}$ is in the same direction throughout the sample. Such a medium can be easily deformed by the application of an external torque such as a magnetic or an electric torque. In the distorted sample $\vec{n} = \vec{n}(\mathbf{r})$. When the sample is distorted, elastic torques come into play within the sample and tend to restore $\vec{n}$ to the original undistorted state.

Three independent types of elastic deformations are possible in nematic liquid crystals as illustrated in figure 1.2. These deformations called splay, twist and bend are characterised by the elastic constants k_{11} , k_{22} and k_{33} respectively.

The elastic properties are described in terms of a continuum theory which disregards the details of the structure on a molecular scale. Such a theory was developed by Oseen (1925) and was reformulated by Frank (1958). The theory is in fact an analogue of the continuum theory of elasticity of solids and is based on the assumptions that the intermolecular forces are of short range nature and the density is the same throughout the sample. Let us consider a coordinate system with its z -axis along $\vec{n}$ at the origin. For weak distortions $n_z \approx 1$ in the first approximation.

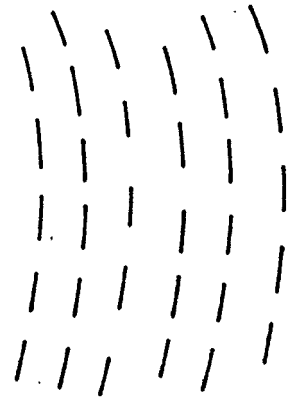
At any point (x, y, z) in the distorted medium

$$n_x = \frac{\partial n_x}{\partial x} x + \frac{\partial n_x}{\partial y} y + \frac{\partial n_x}{\partial z} z = a_1 x + a_2 y + a_3 z.$$



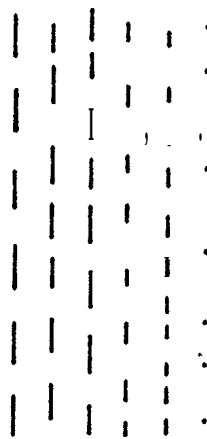
(a)

SPLAY



(b)

BEND



(c)

TWIST

FIGURE 1.2

The three types of basic distortions in a nematic liquid crystal.

$$n_y = \frac{\partial n_y}{\partial x} x + \frac{\partial n_y}{\partial y} y + \frac{\partial n_y}{\partial z} z = a_4 x + a_5 y + a_6 z.$$

$$n_z = 1.$$

The elastic free energy per unit volume can be expressed in the form

$$W_e = k_1 a_1 + \frac{1}{2} k_{ij} a_i a_j \quad i, j = 1, 2, \dots, 6 \text{ and } k_{ij} = k_{ji} \quad (1.2)$$

k_1 has the dimensions of force/distance and k_{ij} , of force.

k_1 and k_{ij} are the elastic constants of the medium.

In a medium where there is no intrinsic deformation,

$k_1 = 0$. $k_1 \neq 0$, if there is an intrinsic deformation as, for example, in a cholesteric liquid crystal in which there is an intrinsic twist.

We have measured the elastic constants of nematic liquid crystals in which there is no intrinsic deformation. Hence we can ignore the first term on the right hand side of equation (1.2).

Taking into account the cylindrical symmetry of a nematic liquid crystal about $\vec{n}$ and the equivalence of $\vec{n}$ and $-\vec{n}$, the elastic free energy density can be written in the form

$$W_e = \frac{1}{2} k_{11} (\nabla \cdot \vec{n})^2 + \frac{1}{2} k_{22} (\vec{n} \cdot \nabla \times \vec{n})^2 + \frac{1}{2} k_{33} [\vec{n} \times (\nabla \times \vec{n})]^2 \\ - \frac{1}{2} (k_{22} + k_{24}) [(\nabla \cdot \vec{n})^2 + (\vec{n} \cdot \nabla \times \vec{n})^2 - \nabla n : \nabla n]$$

.. (1.3)

where $\nabla n : \nabla n = \sum a_n^2$.

Nehring and Saupe (1971) have argued that the second derivatives of $\vec{n}$ make contributions to the elastic free energy comparable to the square of the first derivative. When this is taken into account the expression for the free energy density takes the form

$$W_e = \frac{1}{2} k'_{11} (\nabla \cdot \vec{n})^2 + \frac{1}{2} k_{22} (\vec{n} \cdot \nabla \times \vec{n})^2 + \frac{1}{2} k'_{33} [\vec{n} \times (\nabla \times \vec{n})]^2 \\ + k_{13} \nabla \cdot (\vec{n} \nabla \cdot \vec{n}) - \frac{1}{2} (k_{22} + k_{24}) [(\nabla \cdot \vec{n})^2 + (\vec{n} \cdot \nabla \times \vec{n})^2 \\ - \nabla \cdot \nabla \cdot \vec{n}]$$

where $k'_{11} = k_{11} - 2k_{13}$ and $k'_{33} = k_{33} + 2k_{13}$.

The experimentally determined values of splay and bend constants are actually values of k'_{11} and k'_{33} . There is no method of determining k_{13} separately. For the sake of simplicity we will take $k'_{11} = k_{11}$ and $k'_{33} = k_{33}$ and will assume that equation 1.3 is valid.

Ericksen (1962) has shown that $[(\nabla \cdot \vec{n})^2 + (\vec{n} \cdot \nabla \times \vec{n})^2 - \nabla \cdot \nabla \cdot \vec{n}]$ identically satisfies the Euler-Lagrange equation and hence k_{24} does not contribute anything to the equations of equilibrium. k_{24} involves surface forces and torques which need not be considered when the director is strongly anchored to the surfaces. Then, we get,

$$W_e = \frac{1}{2} k_{11} (\nabla \cdot \vec{n})^2 + \frac{1}{2} k_{22} (\vec{n} \cdot \nabla \times \vec{n})^2 \\ + \frac{1}{2} k_{33} [\vec{n} \times (\nabla \times \vec{n})]^2 \quad (1.4)$$

1.5 Birefringence and order parameter of nematic liquid crystals

A uniformly aligned nematic sample exhibits strong, positive birefringence. If n_o is the refractive index of the ordinary beam having its electric vibrations perpendicular to the director and n_e is the refractive index of the extraordinary beam with the electric vibrations parallel to the director, n_e is always greater than n_o . The birefringence ($n_e - n_o$) is a measure of the order parameter of the liquid crystal as can be seen from the following discussion.

If α_o and α_e are the polarisabilities of the medium for the ordinary and the extraordinary beams

$$\begin{aligned}\alpha_e &= \bar{\alpha} + \frac{2}{3} \Delta\alpha \\ \alpha_o &= \bar{\alpha} - \frac{1}{3} \Delta\alpha\end{aligned}$$

where $\Delta\alpha$ is the polarisability anisotropy of the molecule and $\bar{\alpha}$ is the mean polarisability (Chatelain 1955).

Therefore

$$s = \frac{\alpha_e - \alpha_o}{\Delta\alpha} \quad (1.5)$$

The exact relationship between the principal polarisabilities α_e and α_o and the refractive indices n_e and n_o is not known in strongly anisotropic media like liquid crystals. Two approximate relationships, viz., Neugebauer's (1950) formula and Vuks' (1966) formula have been generally used to relate α_e and α_o with n_e and n_o (Saupe and Maier 1961, Chandrasekhar

and Madhusudana 1969). The Neugebauer's formula uses an anisotropic internal field which is more realistic in a medium like a liquid crystal. On the other hand, Vuks' formula makes use of an isotropic internal field and tends to over-estimate the anisotropy of polarisability. However, this discrepancy exists both in the nematic and the crystalline phases and consequently the order parameter which is the ratio of the anisotropy in the two phases is practically the same in both the Neugebauer's and Vuks' methods (Madhusudana 1980, Subramanyam and Krishnamurti 1973). Further, Vuks' formula is the simpler of the two. Moreover, we should have independent data on temperature variation of density to use the Neugebauer's formula whereas Vuks' formula can itself be used to derive this data from the values of n_x and n_o . Hence we have used Vuks' formula in our calculations.

Vuks' formula. Vuks found that the Lorenz-Lorentz formula works fairly well for the average polarisability of strongly anisotropic organic molecules, i.e.,

$$\frac{\overline{n^2} - 1}{\overline{n^2} + 2} = \frac{4\pi}{3} \nu \bar{\alpha} \quad (1.6)$$

where ν is the number of molecules/cc and

$$\overline{n^2} = \frac{n_x^2 + n_y^2 + n_z^2}{3} \quad \text{and} \quad \bar{\alpha} = \frac{\alpha_x + \alpha_y + \alpha_z}{3} .$$

n_x , n_y and n_z are the principal refractive indices of the crystal and α_x , α_y and α_z are the principal polarisabilities

of the molecule.

In the isotropic medium, the polarisation $\vec{p}$ is given by

$$\vec{p} = \frac{n^2 - 1}{4\pi} \vec{E} \quad \text{where } \vec{E} \text{ is the applied field.}$$

$$\text{Also } \vec{p} = \alpha \vec{F} = \alpha \frac{(n^2 + 2)}{3} \vec{E}$$

$$\text{where } \vec{F} = \frac{(n^2 + 2)}{3} \vec{E} \text{ is the internal field.}$$

For an anisotropic medium,

$$p_i = \frac{(n_{ik}^2 - \delta_{ik}) E_k}{4\pi} \quad i, k = x, y, z.$$

Assuming that the internal field is isotropic we can write

$$p_i = \frac{1}{3} (n^2 + 2) \alpha_{ik} E_k$$

From these equations we get,

$$\frac{n_i^2 - 1}{n^2 + 2} = \frac{4\pi}{3} \alpha_i \quad (1.7)$$

which is the Vuks' formula. Equation (1.6) follows from equation (1.7).

From equations (1.5), (1.6) and (1.7) we get,

$$s = \frac{\bar{\alpha}}{\Delta\alpha} \frac{n_e^2 - n_o^2}{n^2 - 1} \quad (1.8)$$

Equation (1.6) can also be used to evaluate the density of the liquid crystal. If N is the Avogadro number and M is the

molecular weight, $\bar{v} = \frac{N\rho}{M}$ where ρ is the density. Substituting this in equation (1.6) we get.

$$\rho = \frac{3}{4\pi} \frac{M}{N} \frac{1}{\bar{a}} \frac{\overline{n^2} - 1}{\overline{n^2} + 2} \quad (1.9)$$

The re-entrant nematic phase. The usual sequence of phase transitions observed on cooling a mesogen showing smectic A and nematic phases is,

Isotropic — Nematic — Smectic A — Crystal.

It was discovered by Gladis (1975) that binary mixtures of certain mesogenic cyano compounds exhibit a different sequence of phase transitions given below.

Isotropic — Nematic — Smectic A — Nematic — Crystal.

The second nematic phase which occurs at temperatures lower than that of the smectic phase is called the 're-entrant nematic' phase, in analogy with similar re-entrant phases observed in superconductors, Helium-3, etc. More recently, Gladis et al (1977, 1978) observed that some pure cyano-compounds also exhibit the re-entrant nematic phase at elevated pressures.

We observed, for the first time, the re-entrant nematic phase in three pure compounds at atmospheric pressure. Almost simultaneously similar observations were made in some other compounds by Hardouin et al (1979) and Tinh and Gasparoux (1979).

The three compounds we have studied exhibit the re-entrant nematic phase only in the cooling cycle. In other words the re-entrant phase in these compounds is monotropic with respect to the crystalline phase. In chapter II of this thesis we have given the details of these observations. We have studied the isobaric phase diagrams of a few binary mixtures involving these compounds. These have also been described in the same chapter.

All the compounds and mixtures in which the re-entrant nematic phase has been observed so far contain molecules having the strongly polar cyano end-group. (A cyano-group has a large dipole moment of 4.05 debye along its axis.) The neighbouring molecules of such compounds have strong anti-parallel correlations (Madhusudana and Chandrasekhar 1973) and this leads to the formation of bilayer structure with inter-digitating molecules. Direct experimental evidence for the existence of bilayer structure has been obtained in x-ray studies (Leadbetter et al 1975, Lydon and Coakley 1975). Subtle changes taking place within the bilayers of such compounds are presumed to be responsible for the occurrence of the re-entrant nematic phase.

In chapter III, we describe the experiments conducted to determine the order parameters of compounds and mixtures exhibiting the re-entrant nematic phase. It is very difficult to get good homogeneously aligned samples in the smectic phase.

Hence a new technique was used to determine s with a homeotropically aligned sample. By this technique the birefringence was measured in the nematic, smectic A and re-entrant nematic phases and order parameters were calculated using Vuks' formula. The order parameters have also been measured by the infrared dichroism method (Kelker et al 1973, Fernandes and Vemugopalan 1976) which again makes use of a homeotropically aligned sample.

In chapter IV we describe the experimental techniques used to determine the splay and bend elastic constants of nematic liquid crystals, along with the necessary theory. The elastic constants were measured by the well known Freedericksz transition technique (Freedericksz and Rejzewska, 1927). In this method a magnetic field is used to distort a well aligned nematic sample. In an appropriate geometry the critical field at which the distortion just sets in is a measure of an elastic constant of the sample. Different geometries have to be used to measure the splay, bend and twist elastic constants.

In the latter half of this chapter we present Sha results of measurements of elastic constants of the compounds exhibiting the re-entrant nematic phase. The bend elastic constant was measured in both the normal and the re-entrant nematic phases. A light scattering experiment was used to study the variations of twist fluctuations in these compounds.

Usually, when two nematogens whose molecules do not

possess the cyano end group are mixed together the mixtures show only the nematic mesophase. This is also true of mixtures both the components of which have molecules containing the cyano end group. However, when only one of the components consists of molecules with the cyano end group, some of the mixtures show the curious phenomenon of induced smectic mesomorphism (Park et al 1975; Oh 1977; Engelen et al, 1978 and others). We have studied the phase diagrams of three such binary mixtures and these are given in chapter V.

As already mentioned mesogens having the cyano end group have bilayer structure. The addition of a nematogen which does not have cyano end-group to this brings about some interactions between the two types of molecules. These interactions favour the formation of layers giving rise to the smectic A phase. It is of interest to study the effects of long-range and short-range order in such mixtures. With this in mind the order parameters and elastic constants of a few of such mixtures were determined. These observations are also presented in chapter V.

In chapter VI, we present the phase diagram of a binary mixture one of whose components consists of molecules with terminal cyano group and the other consists of molecules with lateral cyano-group. Interestingly, these mixtures do not exhibit induced smectic phase. The refractive indices and the replay elastic constants of some of these mixtures have been

measured. These observations are also presented in chapter VI.

It is a well known fact that nematic liquid crystals having negative dielectric anisotropy and positive conductivity anisotropy undergo vigorous electrohydrodynamic motion under the action of a DC or a low frequency AC electric field (Zwetkoff and Mikhailov 1938; Williams 1963). The Carr-Helfrich theory (Carr 1963, Helfrich 1969, Dubois-Violette et al 1971) gives a satisfactory explanation of these electrohydrodynamic instabilities. In principle, a similar electrohydrodynamic motion is possible in nematic liquid crystals having the reverse characteristics (Durand 1976), viz., positive dielectric anisotropy and negative conductivity anisotropy. Some of the mixtures which exhibit induced smectic phase show these characteristics in the nematic phase just above the smectic A-nematic transition point. We have observed electrohydrodynamic patterns in such mixtures. These observations are reported in chapter VII.

Sussmann (1976) found that nematic liquid crystals with high positive dielectric anisotropy also undergo electrohydrodynamic motion under the action of an electric field. Further he observed (Sussmann 1978) that a cell containing such nematogens between asymmetrically treated electrodes acts as a rectifier. During one half of a cycle of alternating voltage it allows the passage of a higher current compared to that

during the other half.

We have observed that in such asymmetric cells, the electrohydrodynamic motion is more vigorous during one half of the AC cycle than during the other half. As a consequence, under certain conditions, there will be less transmission of light during one half of the cycle compared to that during the other half. This interesting electro-optic phenomenon is described in the last chapter (chapter VIII).

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