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Phase transition and elastic constants of 4-*n*-octyl-4′-cyanobiphenyl (8CB) obtained using the light-scattering anisotropy of turbidity

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A light-scattering technique was used to study the anisotropy of turbidity and the three elastic constants K_1, K_2 and K_3 of 8CB as a function of temperature and sample thickness. The turbidity was measured in the nematic and schematic A phases at sample thicknesses l of 0·02, 0·04, 0·1 and 0·2 cm. The effect of the smectic-like (cybotactic nematic) order was observed near the smectic A–nematic phase transition. Owing to the surface-enhanced cybotactic order, evaluation of the elastic constants and order parameter was possible only from the turbidity data at l = 0·2 cm. From the divergence of both K_2 and K_3 near T_{S_AN} we estimated an average critical exponent value ν of 0·65, suggesting that S_A–N in 8CB is a second-order phase transition. The magnetic-field quenching of director fluctuations showed observed effects on the order of magnitude of the temperature dependence of the turbidities, elastic constants and order parameter.

1. Introduction

The continuum theory of nematic liquid crystals describes the distortion free energy in terms of three fundamental force constants, which are the macroscopic manifestation of the anisotropy of molecular symmetry and interactions. The knowledge of these moduli, designated as splay K_1, twist K_2 and bend K_3, is important from fundamental and application viewpoints. In monomeric liquid-crystalline systems the elastic constants have been relatively well studied and, within the rigid-rod concept, they are predicted to directly reflect the molecular geometries and interactions [1–7]. Experimentally, these predictions have been verified qualitatively for a number of monomeric nematogens [8, 9]. Generally, the theoretical predictions and the experimental data on the elastic constants of the nematic monomers are in qualitative agreement with each other. They indicate that both the absolute and relative values of K_i can be correlated with the molecular structure and the orientational order in the nematic phase.

The measurement of the elastic constants with the conventional Freedericks distortion technique has been well developed [8], but the application of light-scattering methods is relatively recent in the literature. Among these methods, light scattering from the anisotropy of turbidity had been originally developed to evaluate the three elastic constants K_1, K_2 and K_3 of the nematic phase [10]. This technique has been extended to study the temperature dependence of the elastic constants in nematic homologues and mixtures [11, 12], and has recently been applied to study the turbidities and elastic constants in a polypeptide liquid crystal [13, 14].

In this work we have used the light-scattering approach to study the anisotropy of turbidity in the nematic and smectic A phases of 4-*n*-octyl-4-cyanobiphenyl (8CB), from which we have determined the three elastic moduli K_i (i = 1, 2, 3) and the director order parameter S_d over the whole nematic range. We have also studied the effect of sample thickness on the extent of the cybotatic structure in the nematic phase and estimated the order of the smectic-A–nematic phase transition from the K_2 and K_3 divergence near T_{S_AN}.

The trends K_i and S_d obtained by this method compare well with those measured by other techniques, although a systematic difference in absolute values is apparent [10–12]. This discrepancy is found to originate from the quenching of the nematic director fluctuations by a strong magnetic field used in the present light-scattering studies. Damping of director fluctuations is shown to give a systematic reduction of all three turbidity values, which in turn results in a relative increase in the values of the elastic constants and order parameter. The divergence of K_2 and K_3 near T_{S_AN}, which follows a power law with a characteristic critical exponent for a second-order phase transition, did not seem to be affected by the quenched director fluctuations, because the evaluation of the critical exponent was based on relative changes of K_2 and K_3 near T_{S_AN}.

2. Theory and method

The turbidity of nematic liquid crystals is known to be due to light scattering from long-wavelength orientational fluctuations of the nematic director. The first mathematical description of the turbidity was formulated by de Gennes [15] according to

$$\frac{d\sigma}{d\Omega} = \left(\frac{\varepsilon_a \omega^2 V}{4\pi c^2}\right)^2 \sum_\alpha \langle |\delta n_\alpha(q)|^2\rangle (i_\alpha f_z + i_z f_\alpha)^2, \quad (1)$$

$$\langle |\delta n_\alpha(q)|^2\rangle = \frac{kT}{V}(K_\alpha q_\perp^2 + K_3 q_\parallel^2 + \chi_a B^2)^{-1}, \quad (2)$$

where σ is the scattering cross-section or turbidity; Ω is the solid angle of detection; $\varepsilon_a = (n_e^2 - n_0^2)$ is the dielectric anisotropy in the optical frequency range; n_0 and n_e are the ordinary and extraordinary refractive indices; ω is the frequency of the light beam; V is the sample volume; c is the velocity of light in vacuum; δn_α is the thermally averaged ($\langle\ \rangle$) fluctuation of the local nematic director; $\alpha = 1, 2$ are the two mixing elastic modes; K_1 ($\alpha = 1$), K_2 ($\alpha = 2$) and K_3 are the three Franks elastic constants of splay, twist and bend respectively; $q_\parallel$ and $q_\perp$ are the two orthogonal components of the scattering vector parallel and perpendicular to the selected geometrical direction; χ_a is the anisotropy of the diamagnetic susceptibility; B is the magnetic flux density; and i and f are the unit vectors specifying the polarization directions of the incident and scattered beam respectively.

Equation (1) describes the relation between the turbidity and the elastic constants via the equipartition theorem represented by equation (2). The latter fundamental relation, which correlates the elastic moduli with the orientational fluctuations, is based on the assumption that for classical systems in thermal equilibrium the average energy per degree of freedom is equal to $\frac{1}{2}kT$. A combination of equations (1) and (2) provides the basic formula for light scattering in nematic systems. This formulation is the basis of the present method as well as other light-scattering techniques.

Since equation (1) is based on the premise that the incident and scattered light waves propagate in an isotropic medium, its integration in certain geometries leads to an infinite value of σ. This difficulty has been resolved by Langevin and Bouchiat [10], who made an exact computation of the scattering cross-section for light scattering in an anisotropic medium. They showed that the measurements of turbidities at three selected geometries in a uniaxially oriented nematic phase would allow a direct determination of all three moduli, K_1, K_2 and K_3. The calculation of the scattering cross-section σ was carried out for the three geometries:

$$(1)\ \mathbf{k} \parallel \mathbf{v}_0; \quad (2)\ \mathbf{k} \perp \mathbf{v}_0; \quad \mathbf{i} \perp \mathbf{v}_0; \quad (3)\ \mathbf{k} \perp \mathbf{v}_0,\ \mathbf{i} \parallel \mathbf{v}_0;$$

where $\mathbf{k}$ is the wavevector of the incident beam and $\mathbf{v}_0$ is the direction of the nematic director. The corresponding σ at these geometries are

$$(1) \quad \sigma_1/\sigma_0 = \tfrac{1}{4}[I_0(a_1) + I_0(a_2)], \tag{3 a}$$

$$(2) \quad \sigma_2/\sigma_0 = I_1(a_2) + I_2(a_1) - I_2(a_2), \tag{3 b}$$

$$(3) \quad \sigma_3/\sigma_0 = I_3(a_2) + I_4(a_1) - I_4(a_2) + I_5(a_1) + I_6(a_2) - I_6(a_1), \tag{3 c}$$

where $I_0, \ldots, I_6$ are seven complex integrals (see the appendix of [10]) consist of the reduced quantities:

$$b = n_e/n_0, \tag{4 a}$$

$$a_\alpha = K_\alpha/K_3 \quad (\alpha = 1, 2), \tag{4 b}$$

$$\sigma_0 = kT\omega^2\varepsilon_a^2/4\pi c^2 K_3 n_0^2. \tag{4 c}$$

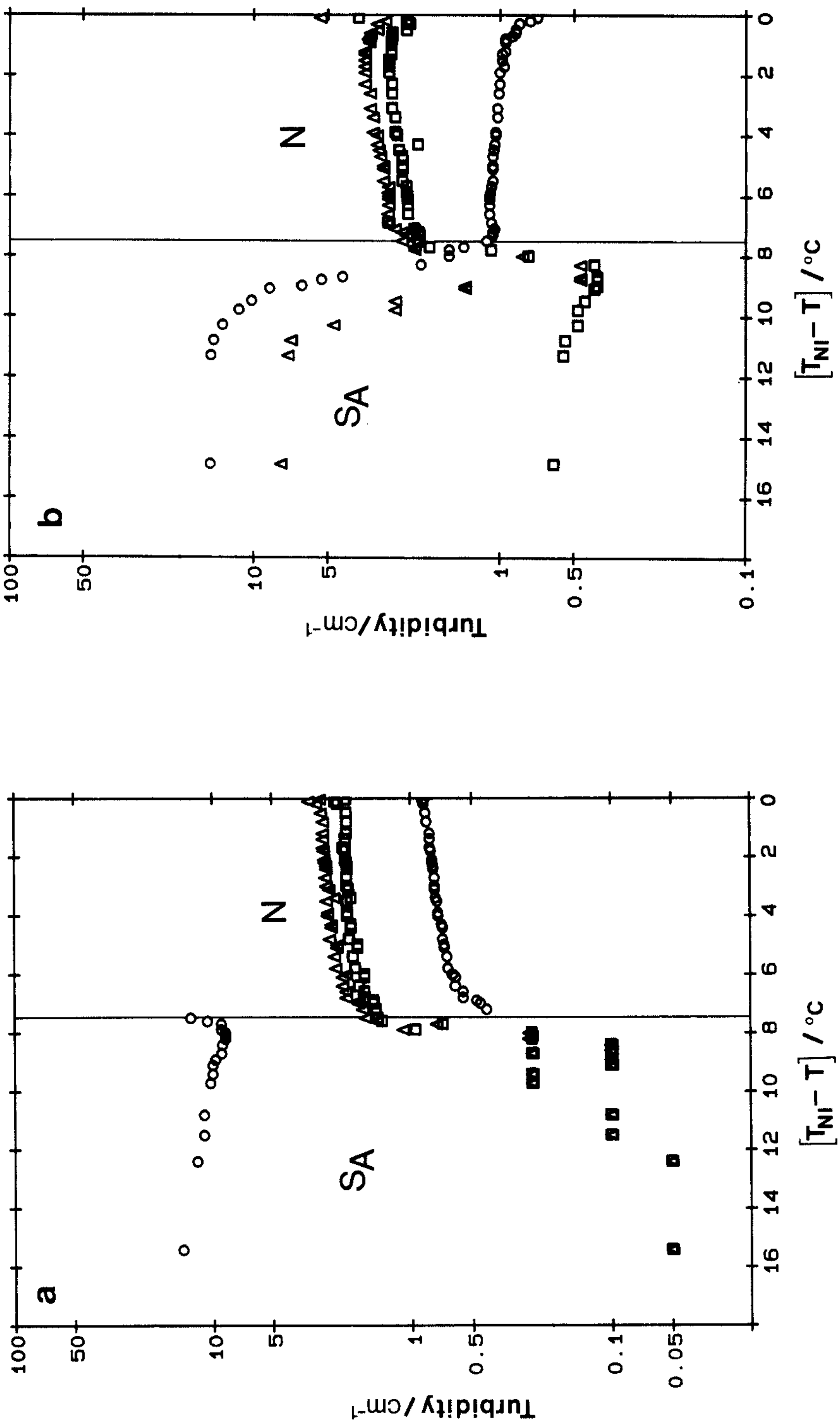
The ratios σ_j/σ_0 were evaluated for a wide range of parameters a_α and b by numerical analysis according to a gaussian-quadrature integration procedure and a Romberg extrapolation. The results were stored in the computer as a master table, where each matrix element consisted of a pair of σ_1/σ_2 and σ_1/σ_3 ratios for given a_α and b values. The evaluation of the elastic constants requires a comparison between the experimental and the theoretical turbidity ratios for the given n_0 and n_e values. A computer search routine uses the necessary iterations to converge and to find the best set of a_1, a_2 and σ_0 with a tolerance of less than 0·1 per cent. From these values and equations (4 *a*–*c*) the evaluation of the corresponding moduli K_1, K_2 and K_3 is a straightforward procedure.

3. Experimental

The compound 4-*n*-octyl-4′-cyanobiphenyl (8CB) was obtained from BDH Chemicals and was used as such. The transition temperatures of 8CB were $T_{S_AN} = 33{\cdot}8$ and $T_{NI} = 41{\cdot}3$°C. The corresponding n_0 and n_e values were extracted from the literature [16], with appropriate corrections made for transition-temperature differences. The samples were prepared between two glass plates or in optical cells with thicknesses of 0·02, 0·04, 0·10 and 0·20 cm. The cell was placed in a temperature-control unit located between the pole faces of a permanent electromagnet. The accuracy of the temperature measurements was ±0·05°C. A magnetic field of 1·4 T was used to orient the mesogen and to define the direction of the nematic director $\mathbf{v}_0$. The beam of a 7 mW laser was directed through a $\frac{1}{2}\lambda$ polarization rotator into the sample, and a series of intercavity and exit mirrors served to direct the beam in the three required geometries. The transmitted light was detected by a photodiode after passing through an adjustable calibrated diaphragm, which defined the solid angle of detection, Ω. The absorption coefficients σ_j in each geometry was measured from the corresponding light intensity I_j according to

$$I_j = I_j^0 \exp(-l\sigma_j), \tag{5}$$

where I_j^0 is the related intensity in the isotropic phase and l is the sample thickness. The experimental details of these measurements have been given elsewhere [10–13].


a
b
N
SA
Turbidity/cm-1
[TNI − T] /°C
100
50
10
5
1
0.5
0.1
0.05
0
2
4
6
8
10
12
14
16

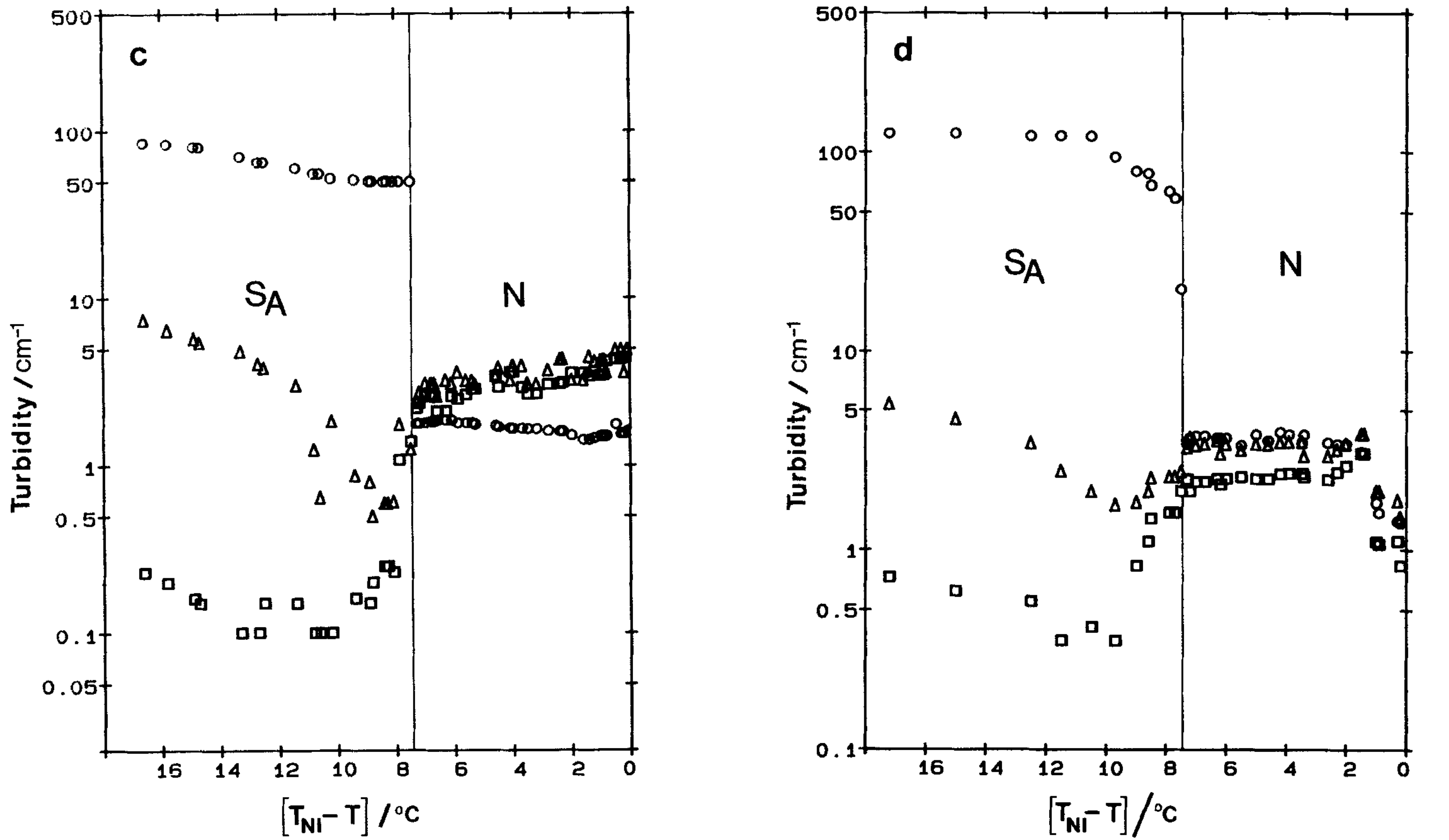


Figure 1. Temperature dependence of the turbidities σ_1 (○), σ_2 (□) and σ_3 (△) in the nematic and smectic A phases of 8CB as functions of sample thickness; (*a*) l = 0·2 cm; (*b*) 0·1 cm; (*c*) 0·04 cm; (*d*) 0·02 cm.

The overall estimated uncertainties in the measured values of the σ_j are within 5 per cent and the elastic constants are about 10 per cent.

4. Results and discussion

4.1. *Anisotropy of turbidity*

The temperature and thickness dependences of the three turbidities σ_j of 8CB in the nematic and smectic A phases are shown in figures 1 (*a–d*). The data in figure 1 (*a*) were obtained for the largest sample thickness, i.e. at $l = 0{\cdot}2$ cm; they show that in the nematic range the relative order $\sigma_3 > \sigma_2 > \sigma_1$, and their non-linear temperature dependence is in accord with previous measurements in thermotropic nematics [10–13]. In the smectic A phase both σ_2 and σ_3 are reduced almost continuously to very small values, indicating that very little light is scattered in these two geometries. On the other hand, σ_1 is very large in the smectic region and shows a discontinuous divergence at the S_A–N transition.

The different behaviour of the turbidities in the nematic and smectic regions may be reconciled if we note that the diamagnetic anisotropy of 8CB is positive [17], establishing the smectic layers perpendicular to the $\mathbf{v}_0$ axis and parallel to $\mathbf{k}$ in geometries (2) and (3), whereas it is perpendicular to $\mathbf{k}$ and parallel to the surface boundaries in geometry (1). The long-wavelength undulations of molecules in the smectic layers due to director fluctuations create density waves [18] that propagate along $\mathbf{v}_0$, which would be sensed differently in the two $\mathbf{k}$ vector orientations.

The turbidity data of figure 1 (*a*) also exhibit pretransitional behaviour near the N–I and S_A–N transitions. Near T_{S_AN} all three σ_j show a systematic decrease due to the effect of the cybotactic (smectic-like) ordering in the nematic phase. The absolute values of the turbidities in figure 1 (*a*) are about two times smaller than those of the nematogen under a lower-intensity (0·1–0·3 T) magnetic field [10]. The difference, which will be discussed later, results from the field-induced quenching of the director fluctuations caused by the use of a strong magnetic field of 1·4 T in our investigations.

Comparing the turbidity data of figure 1 (*b–d*) with that of figure 1 (*a*) clearly shows a systematic effect of the sample thickness on the temperature dependence of σ_j in both nematic and smectic regions. Regardless of the scattered data points and the magnitude of turbidities, σ_1 shows a significant increase in the nematic and smectic phases with decreasing film thickness. This effect can be explained by the successive enhancement of the surface-induced cybotactic order in the nematic region. The establishment of smectic-like ordering near the boundary surfaces would create the corresponding undulating periodic layers along the surface boundaries that strongly scatter light in geometry (1). The effect of surface anchoring can also be observed for σ_2 and σ_3 in both nematic and smectic regions. A complete understanding of the extent of the surface boundary effects on the bulk mesophase turbidity requires further detailed investigations.

In general, the turbdities of uniaxially oriented mesogen are qualitatively different in the nematic and smectic phases. The inequality trends of the σ_j in various phases may be summarized as follows:

$$\sigma_1 = \sigma_2 = \sigma_3 = 0 \quad \text{(isotropic)},$$

$$\sigma_1 \leqslant \sigma_2 \leqslant \sigma_3 \quad \text{(nematic)},$$

$$\sigma_1 \approx \sigma_2 \leqslant \sigma_3 \quad \text{(cybotactic nematic)},$$

$$\sigma_1 \gg \sigma_2 \approx \sigma_3 \quad \text{(smectic A)}.$$

These qualitative characteristic trends can be utilized to study the polymorphism and to identify mesophase types in a wide range of mesomorphic systems.

4.2. *Order parameter*

The orientational order parameter in nematic liquid crystals is conveniently measured by a number of well-developed spectroscopic and macroscopic techniques. Likewise, the present light-scattering method can provide a direct evaluation of the macroscopic or director order parameter from the values of the anisotropy of the turbidity. According to the experimental conditions in geometries (1) and (3), it is readily deduced that the σ_1 and σ_3 represent the perpendicular and parallel components respectively of the anisotropy of the director fluctuations in the uniaxial nematic. In this respect, σ_2 is a mixed-component term consisting of the other two turbidities. Consequently, the value of the macroscopic order parameter S_d may be determined from the simple relation

$$S_d = \frac{\sigma_3 - \sigma_1}{\sigma_3 + 2\sigma_1}, \tag{6}$$

where the denominator on the right-hand side is the mean turbidity value of a diagonalized matrix consisting of the σ_1 and σ_3 orthogonal components.

From equation (6) and the data in figure 1 (*a*), we have evaluated the temperature dependence of the director order parameter over the nematic range of 8CB. The results are compared with those from literature [16] in figure 2. The director order parameter S_d determined by the turbidity method is larger and less temperature-dependent than that reported in the literature. We have also noticed a pretransitional increase of S_d above the S_A–N phase transition. In accord with our previous argument, discrepancies in the order of magnitude and trend of S_d could be explained by the quenching effect of the strong magnetic field on the director fluctuations and hence the turbidity values. According to the equation (6), it can be demonstrated that by

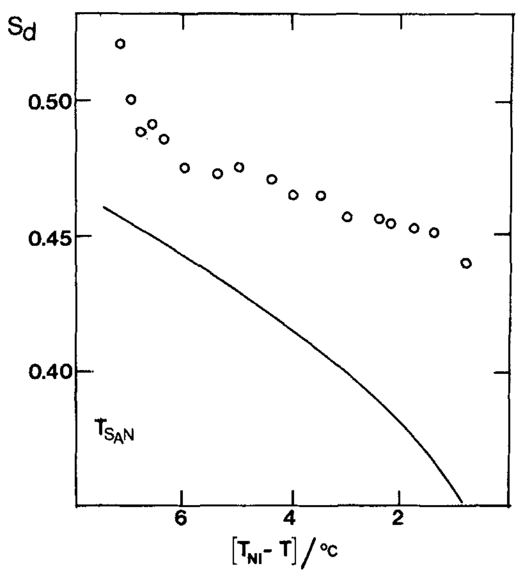


Figure 2. Temperature dependence of the order parameter in the nematic phase of 8CB calculated according to equation (7). The solid line is from [16].

systematic reduction of σ_1 and σ_3 values, the S_d is increased. A more detailed study of the director order parameter using the light-scattering method is in progress and will be reported shortly.

4.3. *Elastic constants*

The evaluation of the elastic moduli was based mainly on the turbidity data obtained for the largest thickness (i.e. at $l = 0{\cdot}2$ cm) which have the smallest contributions from the surface boundary effects. The results are shown in figure 3, indicating the temperature behaviour of K_1, K_2 and K_3 within the nematic range of 8CB. Far from the S_A–N transition (i.e. at $T_{NI}-T \leqslant 4{\cdot}0$°C) the temperature behaviours of the elastic moduli are typical of nematics, whereas within $T_{NI}-T \geqslant 4{\cdot}0$°C a clear divergence is observed between K_2 and K_3, indicating the extent of the pretransitional smectic-like ordering in the nematic phase. This is also in agreement with the literature data using the Freedericks distortions method [16]. According to the data in figure 3, the modulus K_1 undergoes a slight decrease near the S_A–N transition. This is at variance with the reported smooth increase or pretransitional divergence of K_1 in other mesogens [17, 19–21]. The major source of discrepancies in the K_1 could originate from the present field-induced quenching of the orientational fluctuations which is discussed in §4.5.

In an attempt to calculate the elastic moduli from the turbidity data at other thicknesses, we first used an approximation procedure to correct the σ_1 values. This was done by assuming that at each temperature the bulk σ_1/σ_2 ratio should be

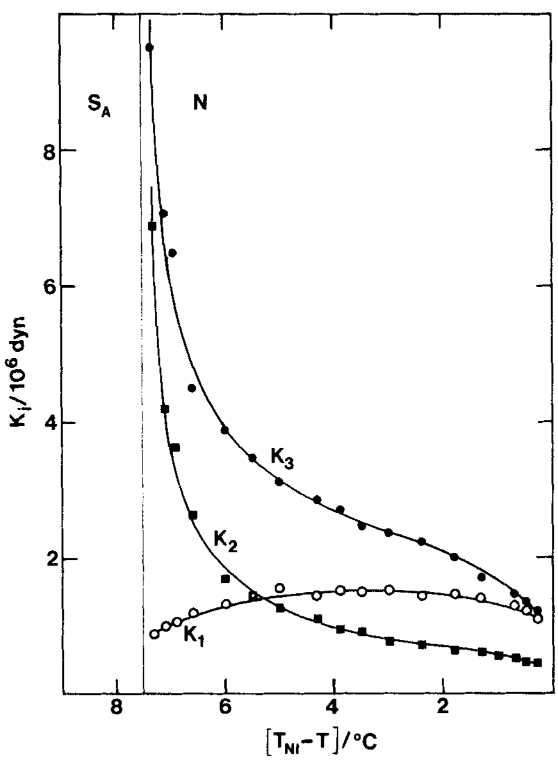


Figure 3. Temperature dependence of the three elastic constants K_1 (●), K_2 (■) and K_3 (○) in the nematic phase of 8CB for $l = 0{\cdot}2$ cm.

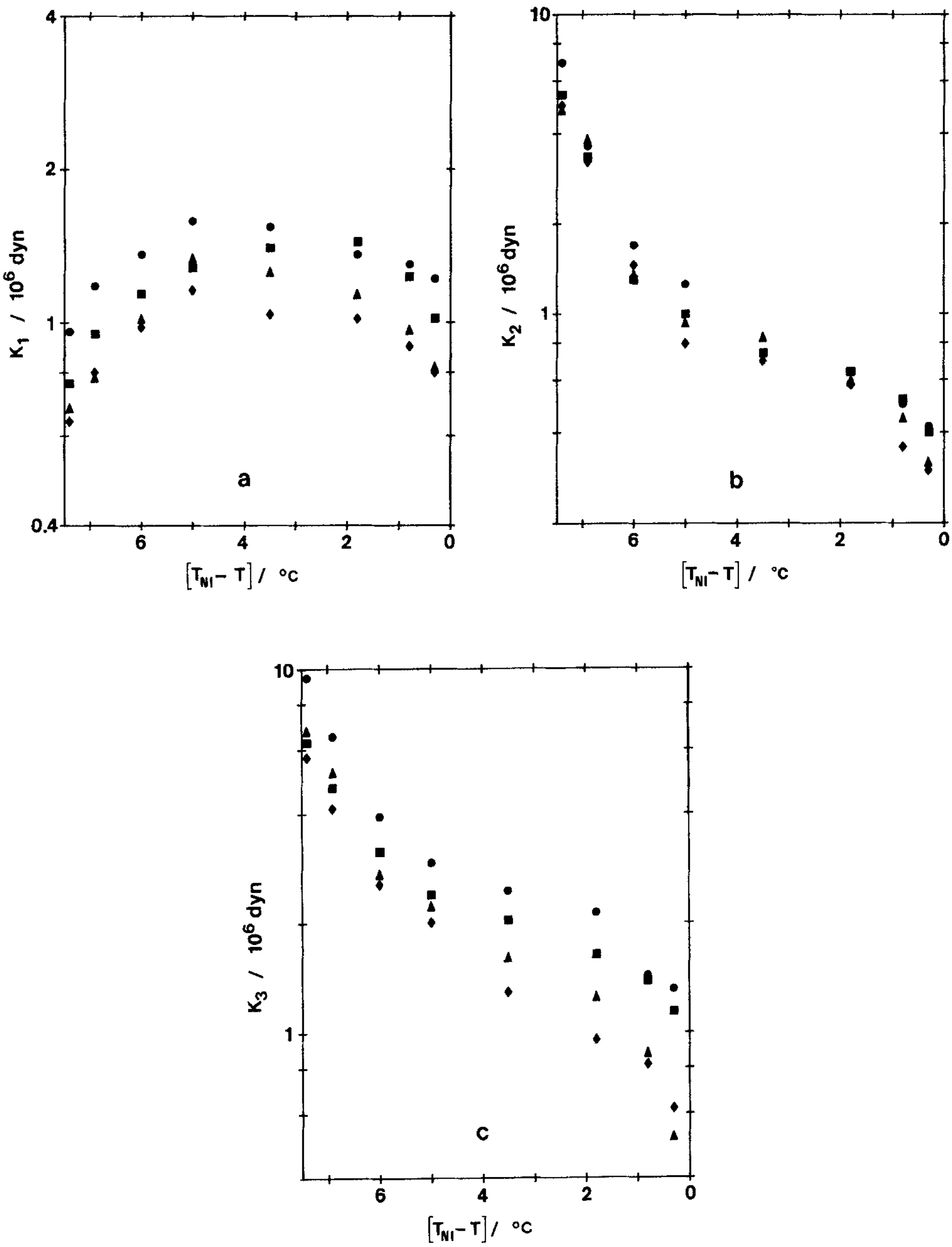


Figure 4. Temperature dependence of the three elastic constants K_1 (*a*), K_2 (*b*) and K_3 (*c*) in the nematic phase of 8CB at sample thicknesses: ●, l = 0·2 cm; ■ 0·1 cm; ◆ 0·04 cm; ▲, 0·02 cm.

independent of the sample thickness and surface effects. From this assumption, and by using the σ_1/σ_2 data at $l = 0{\cdot}2$ cm as reference, we calculated the corresponding moduli K_i from the turbidities at other thicknesses. In figures 4(*a–c*) we present the results of these calculations, including those obtained at $l = 0{\cdot}2$ cm. Within the limits of the experiment, the elastic data are in satisfactory agreement with each other, indicating that, to a first approximation, the correction procedure is sufficient to remove the major contrbution of the surface-induced light scattering in geometry (1).

4.4. *Smectic-A–nematic phase transition*

It is known that near the S_A–N transition both moduli K_2 and K_3 are expected to diverge according to the scaling relation

$$K_i(T) - K_i^0 \sim (T - T_{S_AN})^{-\nu}, \tag{8}$$

where K_i^0 ($i = 1, 2, 3$) are the reference elastic constants from a purely nematic contribution and ν is the critical exponent. In analogy with the second-order phase transition in superfluid helium-4 de Gennes [22] has predicted ν to be 0·67. This has been verified experimentally with X-ray, calorimetric and light-scattering techniques for mesogens including 8CB [23–26]. Further detailed experimental investigations of this model have revealed that, owing to a difference between the longitudinal ($\xi_\perp$) and transverse ($\xi_\parallel$) correlation lengths, the corresponding critical exponents are not equal: $\nu_\parallel > \nu_\perp$. In fact, the X-ray experiments have shown that $\nu_\parallel = 0{\cdot}67$ and $\nu_\perp = 0{\cdot}51$, and light scattering has found a K_3 divergence consistent with $\nu_\parallel = 0{\cdot}62$ [24].

Here, we attempted to estimate the critical exponent according to equation (8) from the observed K_2 and K_3 divergence of 8CB near T_{S_AN} (see figure 3). The results, as shown by two examples in the double-logarithmic plot in figure 5, gives an average value of $\nu = 0{\cdot}65 \pm 0{\cdot}05$ in both K_2 and K_3 cases. This suggests that the S_A–N phase transition of 8CB should be second-order. Note that to estimate ν, we have used the temperature-independent reference values $K_2^0 = 0{\cdot}4 \times 10^{-6}$ and $K_3^0 = 1{\cdot}2 \times 10^{-6}$ dyn. Although these reference values are not expected to be good approximations for

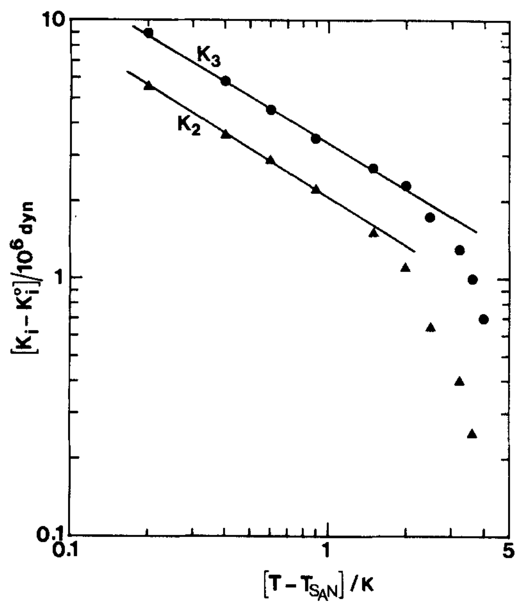


Figure 5. Double-logarithmic plots of the K_2 and K_3 data in figure 3 according to the scaling law of equation (8). The linear portions of the curves near T_{S_AN} give average slopes of $\nu = 0{\cdot}65$.

proper applications of equation (8), their use is sufficient in the limit of present experimental capabilities. In fact, owing to the limit of the temperature measurements, the relatively large K_i, and a possible effect of the field-quenched director fluctuations on the K_2 and K_3 divergence, we did not expect to find any difference between the $\nu_{\parallel}$ and $\nu_{\perp}$ values. Obviously, more accurate measurements of temperature and the elastic constants near $T_{S_A N}$ are required to verify this phenomenon. The effort depends on the experimental improvement of the present technique that is currently in progress.

4.5. *Quenching of the director fluctuations*

The quantitative reliability of the present light-scattering method depends significantly on the ability to evaluate accurate values of the elastic constants from precise measurements of the turbidities and refractive indices. In order to investigate the contributions of these experimental parameters to the elastic constants, we have evaluated the moduli K_i as functions of the turbidities and the refractive indices. The overall conclusion of these semi-experimental evaluations is that the elastic constants vary *strongly* and non-linearly with these parameters. In figures 6 and 7 we present two examples of these calculations, showing variations of the K_i with σ_1 and n_e respectively. Further calculations of the elastic moduli with respect to the variations in the absolute values of the turbidities confirm that a linear decrease in σ_j results in a linear increase of the corresponding K_i values. An example of these calculations is given in figure 8. This clearly explains the reason for the relatively large elastic-constant values obtained in these studies. The source of this difference, which has not been discussed in our previous papers [11–13], is the relative quenching of the fluctuations of the nematic director due to the presence of a strong magnetic field. Since the electromagnet used provides a constant field of 1·4 T, we were not able to study the effect of a variable field on the turbidities. However, from a preliminary study on 4-methoxybenzylidene-4′-*n*-butylaniline (MBBA), we measured the σ_j values

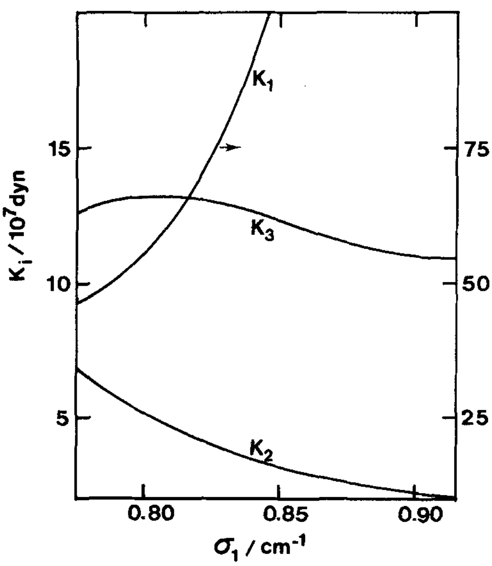


Figure 6. Variations of the three elastic constants with σ_1; $T = 314\,K$, $n_0 = 1{\cdot}528$, $n_e = 1{\cdot}632$, $\sigma_2 = 2{\cdot}35\,cm^{-1}$, $\sigma_3 = 2{\cdot}86\,cm^{-1}$.

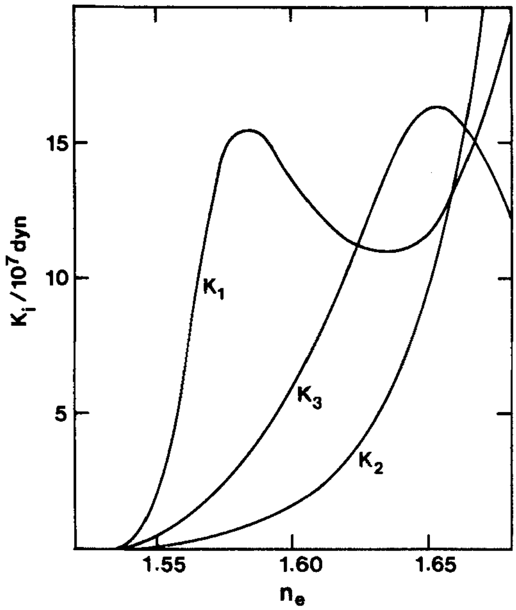


Figure 7. Variations of the three elastic constants with the refractive index; $\sigma_1 = 0{\cdot}80\,\mathrm{cm}^{-1}$, $\sigma_2 = 2{\cdot}35\,\mathrm{cm}^{-1}$, $\sigma_3 = 2{\cdot}86\,\mathrm{cm}^{-1}$, $n_0 = 1{\cdot}528$, $T = 314\,\mathrm{K}$.

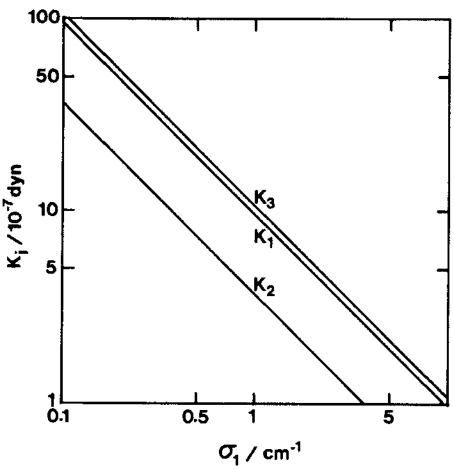


Figure 8. Inverse relation between the elastic constants K_i and the turbidities σ_i; $\sigma_1/\sigma_2 = 0{\cdot}34468$, $\sigma_1/\sigma_3 = 0{\cdot}28322$, $n_0 = 1{\cdot}528$, $n_e = 1{\cdot}631$, $T = 314\,\mathrm{K}$.

and compared the evaluated K_i with those reported in another study using a 0·3 T field intensity [10]. The results are given in the table; they clearly indicate that damping of director fluctuations by the magnetic field reduces the turbidities and results in increases of the moduli K_i. Furthermore, Langevin and Bouchiat [10] have shown that the $\chi_a H^2$ term in the denominator of equation (1) should make a negligible contribution to the K_i values. For example, in MBBA at 22°C, assuming that $\chi_a = 1{\cdot}2 \times 10^{-7}\,\mathrm{emu\,g^{-1}}$ and $H = 0{\cdot}3\,\mathrm{T}$, we find that the term $\chi_a H^2$ gives a value of 1·1. This term at $H = 1{\cdot}4\,\mathrm{T}$ is about 20 times larger. From this simple calculation it is again concluded that the contribution of the magnetic-field term may not necessarily

The effect of the magnetic field on the turbidities and the elastic constants of MBBA.

	[10]	This work
β/T	0·3	1·4
σ_1/cm^{-1}	4·9	3·2
σ_2/cm^{-1}	12·1	8·1
σ_3/cm^{-1}	14·9	9·5
$K_1/10^7$ dyn	6·7	11·8
$K_2/10^7$ dyn	4·2	7·9
$K_3/10^7$ dyn	8·4	19·7

be negligible at higher field intensities, and its increase results in a reduction of the overall scattering in all geometries. This effect finds support from the experimental results in the table and the calculated data in figure 8.

Since the quenching of the director fluctuations is not necessarily a linear function of the magnetic flux density, any attempt to correct the present elastic-constant data must await detailed measurements of the turbidities and elastic constants as functions of the applied magnetic field. The extent of the field-induced effect in light scattering from the smectic phase has yet to be studied.

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