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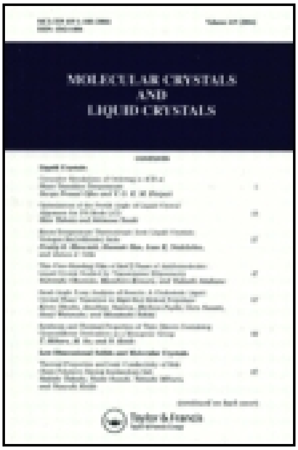

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Determination of the Viscosity Coefficients of the Liquid Crystal MBBA

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(*Received June 10, 1980*)

An experimental procedure is described which allows the determination of the viscosity coefficients η_1, η_2, η_3 and η_{12} for nematic liquid crystals as a function of temperature. Errors which arise from the surface and the flow alignment are discussed in detail. The measurements are performed on the nematic liquid crystal 4-methoxybenzylidene-4′-*n*-butyl-aniline (MBBA). At 25°C the viscosity coefficients are $\eta_1 = 0.1361$, $\eta_2 = 0.0240$, $\eta_3 = 0.0413$ and $\eta_{12} = -0.0181$ Pa s.

INTRODUCTION

The hydrodynamic theory of Leslie[1] and Ericksen[2] describes the viscous behaviour of nematic liquid crystals by five independent viscosity coefficients. Great effort has been undertaken to measure these coefficients using a variety of techniques. However, de Jeu[3] has shown that even in the case of the often used 4-methoxy-benzylidene-4′-*n*-butylaniline (MBBA) the measured coefficients are not well established. Most of the errors arise from the incomplete alignment of the liquid crystals due to surface and flow alignment. The first aim of this paper is to describe an apparatus which allows the determination of four of these viscosity coefficients. By proper choice of the experimental conditions the influence of the incomplete alignment is reduced. Furthermore, suitable corrections for these effects are given in this paper.

In order to compare the viscosity coefficients with literature values the measurements are performed on MBBA. The viscosity coefficients η_1, η_2, η_3 and η_{12} are measured at several temperatures in the nematic phase as well as the isotropic viscosity coefficient at temperatures above the clearing point.

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EXPERIMENTAL

Four of the viscosity coefficients—η_1, η_2, η_3 and η_{12} in the notation of Helfrich[4]—are related to a shear flow between infinite parallel plates and can be measured in a capillary viscosimeter with rectangular cross section of suitable dimensions.[5,6] Our apparatus consists of a capillary connected at each end to a closed vessel filled with nitrogen. A change of the pressure in one vessel induces a flow of the liquid crystal through the capillary. By measuring the pressure difference between the vessels as function of time the viscosity coefficients of the liquid crystal can be determined. Owing to the large interface between the liquid crystal and the gas the influence of the surface tension is reduced in the case of an incomplete wetting of the container walls.

The alignment of the liquid crystal is achieved through application of a magnetic field. The degree of alignment depends on the ratio of the torques resulting from the interaction of the magnetic field with the anisotropy of the magnetic susceptibility (magnetic alignment), from the viscous shear (flow alignment) and from the interaction of the liquid crystal with the surface of the capillary walls (surface alignment). Good alignment is achieved by using a strong magnetic field, small shear rates and a large capillary thickness. Furthermore, a highly oblong cross section of the capillary has to be chosen for an accurate determination of the anisotropy of the viscosity coefficients.

The capillary consists of 20 horizontal brass plates which are assembled together as shown schematically in Figure 1. The cross section of the capillary is $0.3 \times 16\ \mathrm{mm}^2$ and the total length amounts to 85.5 cm. A second capillary block with vertical plates is constructed in a similar way. The inlet and outlet of the capillary block are connected to two glass cylinders of 15 mm inner diameter for the storage of the liquid crystal. The capillary block and one half of the glass cylinders are filled with the degassed liquid crystal. Complete wetting is achieved by coating the inner surface of the glass cylinders with SnO_2. The glass cylinders are coupled by brass tubes to two buffer volumes of 85 cm^3. The remaining part of the glass cylinders, the brass tubes and the buffer volumes are filled with dry nitrogen. Because of the pressure dependence of the buffer gas compressibility a constant nitrogen pressure is chosen for all measurements. In order to achieve an isothermal expansion of the buffer gas during the measurement each buffer volume consists of 25 boreholes of 7 mm diameter in a copper block. The pressure difference between the two buffer volumes, which varies from 600 to 300 Pa, is measured with an electronic differential pressure manometer (Datametrics, Barocel 572/1018) and is printed out at constant time intervals determined by a pulse generator.

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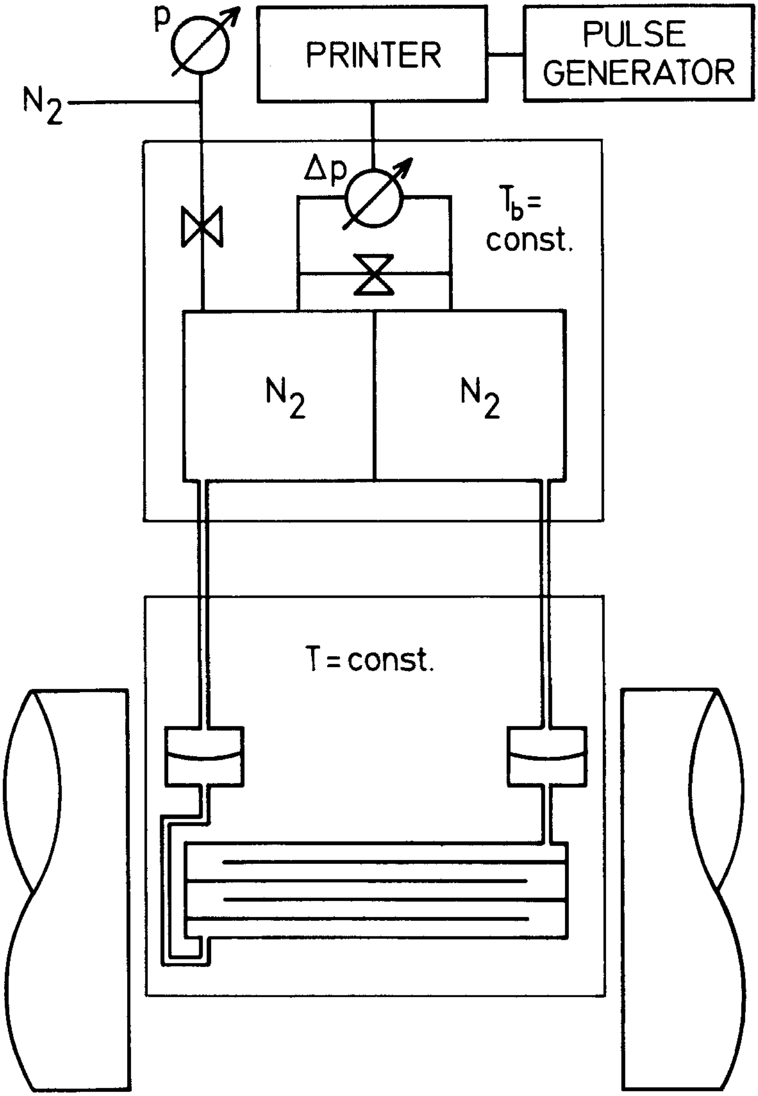

FIGURE 1 Experimental set-up.

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The capillary block, the glass cylinder and part of the brass tubes are submerged in an oil bath whose temperature T can be regulated to ± 0.01 K. The remaining part of the tubes, the buffer volumes and the manometer are placed in an aluminium case whose temperature is kept at $T_b = 22°\text{C}$.

Alignment of the liquid crystal is achieved with a magnetic field of $B = 0\text{–}1.1T$, which is controlled by a calibrated Hall probe. Rotation of the magnetic field and utilization of the two capillary blocks allow the determination of the viscosity coefficients η_1, η_2, η_3 and η_{12}.

MBBA was obtained from Riedel-de Haën AG and was used without further purification. The nematic-isotropic transition temperature of 45.1°C did not change significantly during the experiments.

Evaluation of the measurements

For a laminar flow in a long capillary, the volume flow rate $\dot{V}$ of a liquid with a viscosity coefficient η is given by

$$\dot{V} \sim \frac{\Delta p_c}{\eta} \tag{1}$$

where Δp_c is the pressure difference between the capillary ends. Assuming that the buffer gas behaviour is ideal and that the diaphragm displacement volume of the manometer is proportional to the applied pressure difference, the flow rate is proportional to the derivative of the gas pressure difference Δp with respect to time

$$\frac{\mathrm{d}\Delta p}{\mathrm{d}t} \sim -\frac{\Delta p_c}{\eta} \tag{2}$$

If the temperature T_b of the buffer volumes and the temperature T of the oil bath for the capillary are different, the thermal expansion of the gas has to be taken into account. Furthermore, the compressibility of the system depends on the gas volumes. Both effects can be described by a factor X, which is a function of T, T_b, the gas volumes and the diaphragm displacement volume.

$$X\frac{\mathrm{d}\Delta p}{\mathrm{d}t} \sim -\frac{\Delta p_c}{\eta} \tag{3}$$

The pressure differences Δp and Δp_c are correlated over the hydrostatic pressure difference $\rho g \Delta h$, which arises from the changing levels of the liquid crystal in the glass cylinders.

$$\Delta p_c = \Delta p - \rho g \Delta h = \Delta p - (\Delta p_0 - \Delta p) A X \rho g \tag{4}$$

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Δp_0 is the gas pressure difference at the beginning of the experiment ($\Delta h = 0$, $t = 0$) and A is a constant, which depends on the cross sections of the glass cylinders. Hence Eq. (3) can be written as

$$\frac{\mathrm{d}\Delta p_c}{\mathrm{d}t} = -\frac{K}{\eta X}(1 + AX\rho g)\Delta p_c \tag{5}$$

The constant K is determined by calibration with a viscosity standard oil (Physikalisch-Technische Bundesanstalt, FRG). Because of the flow alignment the effective viscosity coefficient η_i^{eff} is a function of the pressure difference across the capillary (see Appendix II).

$$\frac{1}{\eta_i^{\mathrm{eff}}} = \frac{1}{\eta_i} + D\Delta p_c^2 \qquad i = 1, 2, 3, 45 \tag{6}$$

Taking into account this effect Eq. (5) becomes

$$\frac{\mathrm{d}\Delta p_c}{\Delta p_c(1 + D\eta_i\Delta p_c^2)} = -\frac{K}{\eta_i X}(1 + AX\rho g)\mathrm{d}t \tag{7}$$

which gives on integration

$$\ln\frac{\Delta p_c}{(1 + D\eta_i\Delta p_c^2)^{1/2}} = -\frac{K}{\eta_i X}(1 + AX\rho g)t + C \tag{8}$$

Equation (8) with Δp_c from Eq. (4) is used for the evaluation of the measurements. In the case $D = 0$ η_i is determined from the best fit of Eq. (8) to the experimental data. For $D \neq 0$ the same procedure is applied with varying values of D and the result with the best correlation coefficient is used for the determination of η_i.

RESULTS AND DISCUSSION

a Investigation of surface and flow alignment

The influence of the flow alignment and the correction procedure can be seen in Figure 2. The deviations of the experimental Δp values from an exponential function according to Eq. (8) with $D = 0$ are plotted as a function of time for two experiments. In the first experiment (η_3, 1.1 T) no correction is necessary because in this case no torque is exerted on the director. The observed small scattering of the values is mainly due to the resolution of 0.1 Pa of the differential manometer. In the second experiment (η_1, 0.6 T) strong flow alignment occurs, but a good fit is achieved with a correction of $D\eta_1 = 4 \cdot 10^{-8}$ Pa^{-2}. The change in the value of the viscosity coefficient only amounts to 7 ‰.

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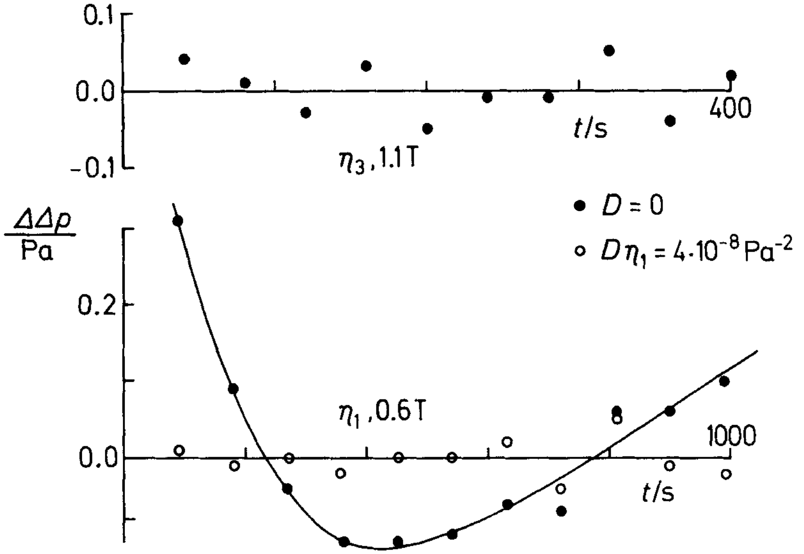

FIGURE 2 Difference of the experimental and the calculated pressure difference as a function of time at 25°C.

The influence of the surface alignment is studied by measuring the effective viscosity coefficient as a function of the magnetic induction. According to Eq. (13) of Appendix I a straight line is expected on plotting $1/\eta_i^{\mathrm{eff}}$ or $\eta_i/\eta_i^{\mathrm{eff}}$ versus $1/B$ (Figure 3). For η_2 only a very small dependence of η_i^{eff} on the induction of the magnetic field is observed. Obviously, in this case the surface alignment agrees with the direction of the magnetic field, which is parallel to the rolling direction of the brass plates. The strongest dependence is observed for η_1 and the extrapolation to an infinite magnetic induction runs up to 11%. Because of the good linear dependence the error of the extrapolation should not be greater than 1–2%.

b Viscosity coefficients

The viscosity coefficients η_1, η_2 and η_3 of MBBA which are corrected for flow and surface alignment are shown in Figure 4 as a function of temperature. The numerical values are given in Appendix III. Disregarding the possible influence of the different clearing points (Gähwiller 43°C, this work 45.1°C) the values of η_2 and η_3 at 25°C are in good agreement with the results of Gähwiller[5] (0.0238 and 0.0416 Pa *s* resp.). The values of η_1 are different (Gähwiller[5] 0.1035 Pa *s*, this work 0.136 Pa *s*), whereas the agreement with the results of Summerford *et al.*[7] (0.135–0.140 Pa *s*) is good.

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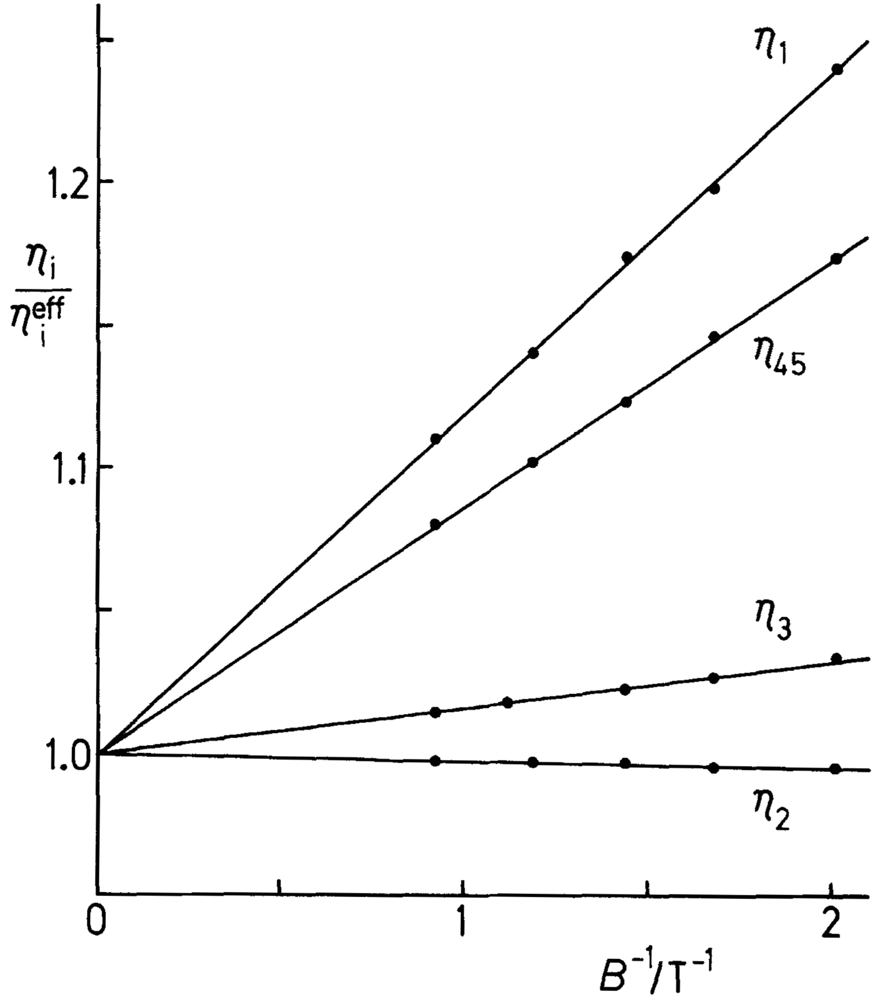

FIGURE 3 η_i/η_i^{eff} as a function of the inverse magnetic induction B^{-1}. η_i is determined from a plot of $(\eta_i^{eff})^{-1}$ versus B^{-1} at $B^{-1} = 0$.

The reason for the too small value of Gähwiller[5] was given by Summerford *et al.*[7] and de Jeu[3] who argued that under the experimental conditions of Gähwiller[5] ($B = 0.6$ T, shear rates up to 20 s^{-1}) no uniform director alignment could be achieved because of the flow alignment. Furthermore, one should expect a strong influence of the surface alignment and of the unfavourable cross section of the rectangular capillaries in Gähwiller's experiments.

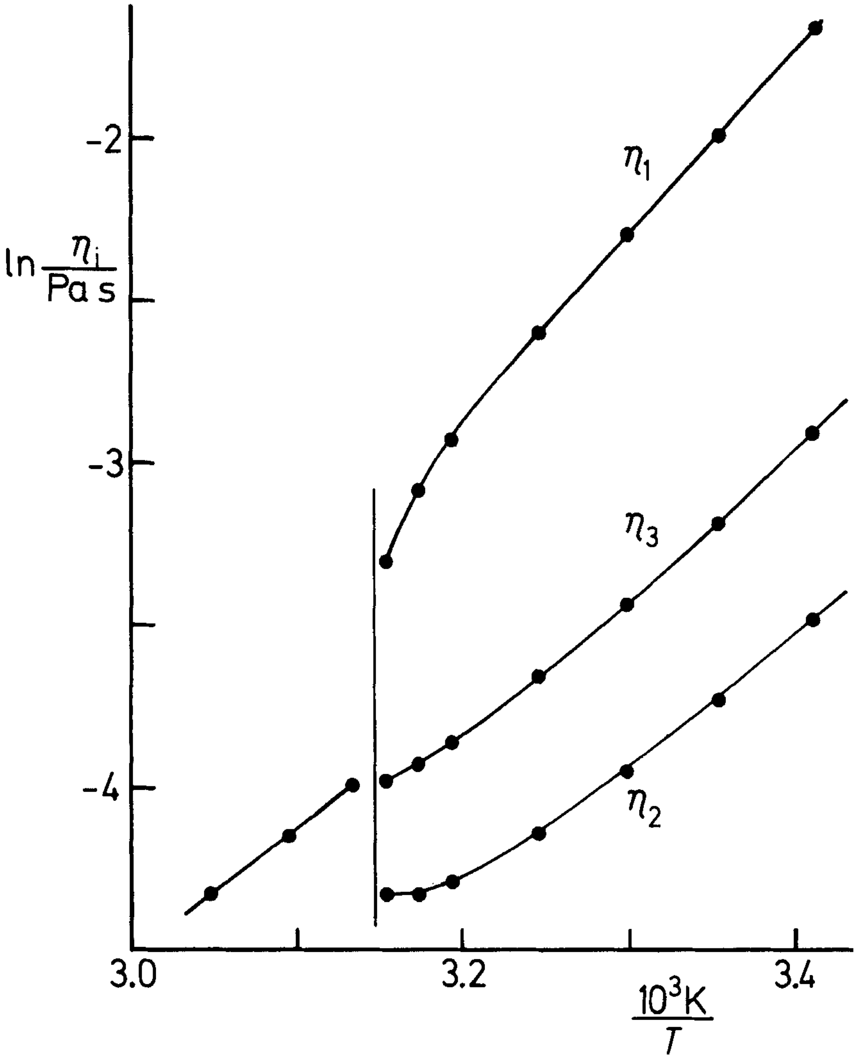

FIGURE 4 Viscosity coefficients η_1, η_2 and η_3 as a function of temperature.

The temperature dependence of the viscosity coefficients seems to be very complicated. For the viscosity coefficients η_2 and η_3 no linear part in the plot of $\ln \eta$ versus T^{-1} can be observed. The linear dependence of $\ln \eta_1$ at lower temperature probably results from a superposition of a concave bending of the curve due to a normal isotropic liquid behaviour and a convex bending due to the temperature dependence of the degree of order.

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From the three values in the isotropic phase an activation energy of 32.5 kJ mole^{-1} is calculated. The determination in an Ubbelohde viscosimeter (see Appendix III) gives 32.6 kJ mole^{-1} using the lowest three temperatures.

On the basis of the Leslie–Ericksen theory Imura and Okano[8] derived equations which describe the temperature dependence of the viscosity coefficients. According to this theory the difference η_1-η_2 should be proportional to the degree of order. In contrast, Figure 4 shows that at low temperatures, where the degree of order changes slowly with the temperature, the ratio η_1/η_2 rather than the difference is nearly constant. The theories of Hess[9] and Tseber[10] introduce other sets of viscosity coefficients from which the η coefficients can be calculated. Unfortunately, the temperature dependence of these new sets of coefficients is not yet known.

In Figure 5 the viscosity coefficient η_{12} is plotted versus the temperature. The error in this coefficient can be substantial because of the indirect method of determination by the equation $\eta_{12} = 4\eta_{45} - 2(\eta_1 + \eta_2)$. For instance, the point for 20°C does not lie on the smooth curve connecting the other points. Contrary to our result Gähwiller[5] found positive values which did

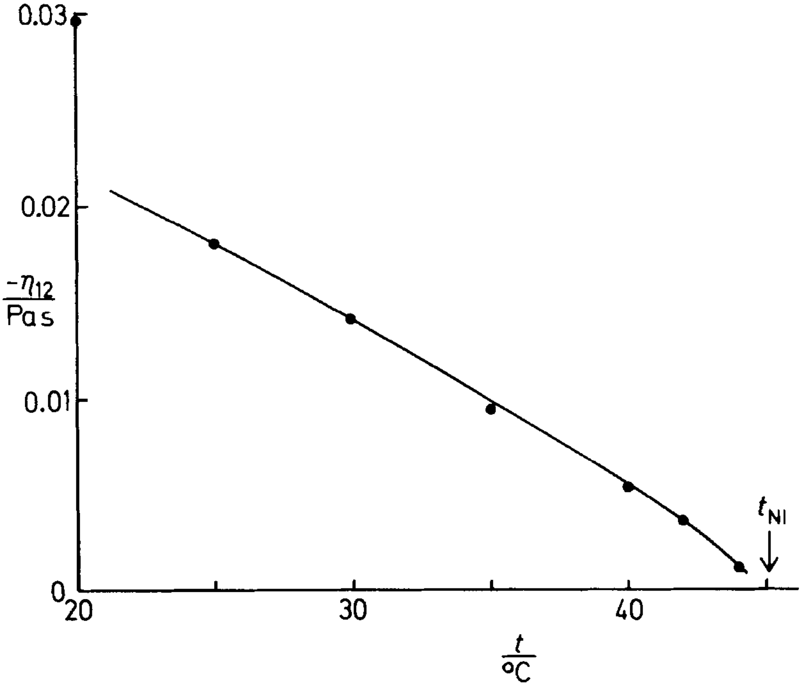

FIGURE 5 Viscosity coefficient η_{12} as a function of temperature.

not depend on the temperature. Probably this is due to the fact that his η_1 coefficients are too small. Negative values for η_{12} are not excluded by any physical reason. Thermodynamics only requires the fulfillment of some inequalities[11] which are satisfied by our values. Since the error of the η_{12} determination is very large, no definite statement about the dependence of η_{12} on temperature or degree of order can be made.

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Appendix I

INFLUENCE OF THE SURFACE ALIGNMENT

We assume that the alignment of the liquid crystal is entirely determined by the inner capillary surface within the magnetic coherence length ξ

$$\xi = \sqrt{\frac{k}{\chi_a \mu_0}} \cdot \frac{1}{H} \approx \sqrt{\frac{k\mu_0}{\chi_a}} \cdot \frac{1}{B} \tag{9}$$

which depends on one of the elastic constants k, on the anisotropy χ_a of the magnetic susceptibility and on the induction B of the magnetic field. Outside of the coherence length the alignment is determined by the magnetic field direction. If the viscosity coefficient η_s of the liquid crystal at the inner surface of the capillary is different from the bulk viscosity coefficient η_i the effective viscosity η_i^{eff} depends on the induction B of the magnetic field. With these assumptions the velocity profile (Figure 6) in a capillary with a

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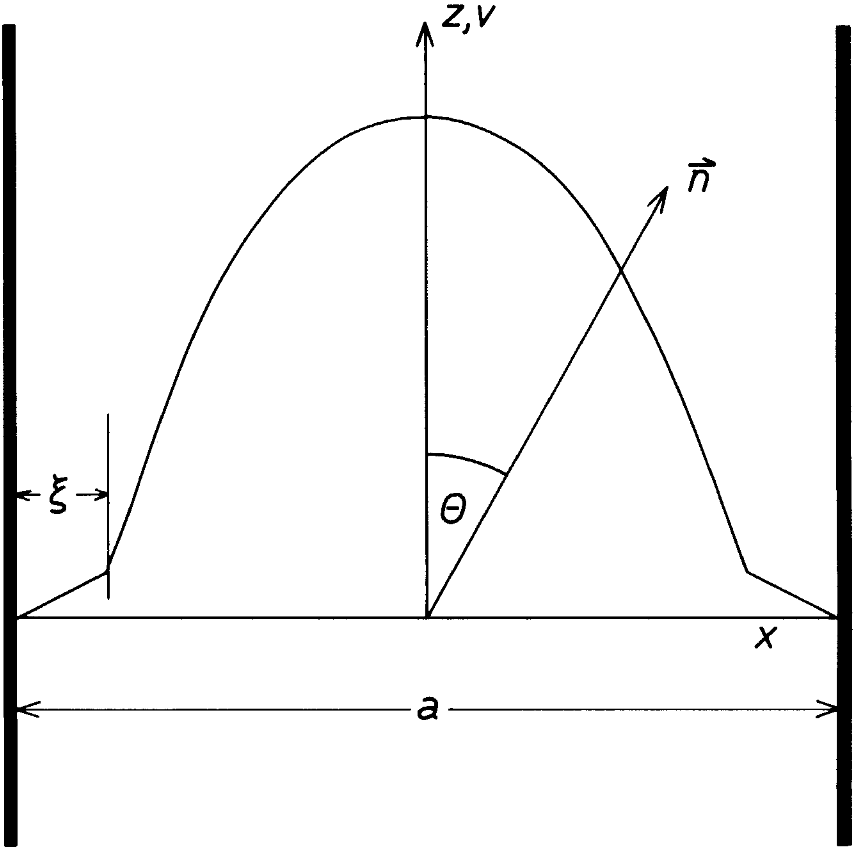

FIGURE 6 Velocity profile in the capillary which is assumed for the calculation.

thickness a is obtained as

$$v = \frac{1}{2\eta_s} \cdot \frac{\mathrm{d}p}{\mathrm{d}z}\left(x^2 - \frac{a^2}{4}\right) \quad \text{for} \quad -\frac{a}{2} \leq x \leq -\frac{a}{2} + \xi$$

$$\text{and} \quad \frac{a}{2} - \xi \leq x \leq \frac{a}{2}$$

$$v = \frac{1}{2\eta_i} \cdot \frac{\mathrm{d}p}{\mathrm{d}z}\left[x^2 - \left(\frac{a}{2} - \xi\right)^2\right] + \frac{1}{2\eta_s} \cdot \frac{\mathrm{d}p}{\mathrm{d}z}\left[\left(\frac{a}{2} - \xi\right)^2 - \frac{a^2}{4}\right]$$

$$\text{for} \quad -\frac{a}{2} + \xi \leq x \leq \frac{a}{2} - \xi \qquad (10)$$

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The volume flow rate $\dot{V}$ is given by

$$\dot{V} = b\int_{-a/2}^{+a/2} v\,\mathrm{d}x = -\frac{2b}{3}\cdot\frac{\mathrm{d}p}{\mathrm{d}z}\left[\frac{a^3}{8\eta_i} + \left(\frac{3a^2\xi}{4} - \frac{3a\xi^2}{2} + \xi^2\right)\left(\frac{1}{\eta_s} - \frac{1}{\eta_i}\right)\right] \tag{11}$$

and the effective viscosity becomes

$$\frac{1}{\eta_i^{\mathrm{eff}}} = \frac{1}{\eta_i} + \left[6\frac{\xi}{a} - 12\left(\frac{\xi}{a}\right)^2 + 8\left(\frac{\xi}{a}\right)^3\right]\left(\frac{1}{\eta_s} - \frac{1}{\eta_i}\right) \tag{12}$$

Neglecting terms higher than first order in ξ/a one obtains

$$\frac{1}{\eta_i^{\mathrm{eff}}} = \frac{1}{\eta_i} + \sqrt{\frac{k\mu_0}{\chi a}}\cdot\frac{6}{aB}\left(\frac{1}{\eta_s} - \frac{1}{\eta_i}\right) \tag{13}$$

Plotting $1/\eta_i^{\mathrm{eff}}$ against $1/B$ one should get a straight line.

Appendix II

INFLUENCE OF THE FLOW ALIGNMENT OF THE LIQUID CRYSTAL

a Measurement of η_1

The influence of the flow alignment on the effective viscosity is calculated according to the theory of Leslie[1] and Ericksen.[2] In order to simplify the calculations we use the following approximations

1) $\eta_2 = \eta_{12} = 0; \alpha_3 = 0$
2) There are no elastic contributions to the alignment of the liquid crystal.

In this case the Leslie–Ericksen theory reduces to an equation for the motion

$$\frac{\mathrm{d}p}{\mathrm{d}z} = \eta_1 \sin^2\theta\cdot\frac{\mathrm{d}^2 v}{\mathrm{d}x^2} \tag{14}$$

and an equation for the alignment

$$-\alpha_2 \sin^2\theta\cdot\frac{\mathrm{d}v}{\mathrm{d}x} = \chi_a\mu_0 H^2 \sin\theta\cos\theta \approx \frac{\chi_a}{\mu_0} B^2 \sin\theta\cos\theta \tag{15}$$

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Elimination of d^2v/dx^2 from the Eqs. (14) and (15) and integration of the differential equation gives

$$\theta = \frac{\pi}{2} + \beta_1 x \quad \text{with} \quad \beta_1 = \frac{\alpha_2 \mu_0}{\eta_1 \chi_a B^2} \frac{dp}{dz} \tag{16}$$

Using Eqs. (15) and (16) and expanding $\tan \beta_1 x$ up to the third power, v and $\bar{v}$ are obtained as

$$v = \frac{1}{2\eta_1} \frac{dp}{dz} \left\{ x^2 - \left(\frac{a}{2}\right)^2 + \frac{\beta_1^2}{6} \left[x^4 - \left(\frac{a}{2}\right)^4 \right] \right\} \tag{17}$$

$$\bar{v} = -\frac{a^2}{12\eta_1} \frac{dp}{dz} \left(1 + \frac{\beta_1^2 a^2}{20} \right) \tag{18}$$

The effective viscosity becomes

$$\frac{1}{\eta_1^{\text{eff}}} = \frac{1}{\eta_1} + \frac{\beta_1^2 a^2}{20\eta_1} \tag{19}$$

b Measurement of η_2

The Leslie–Ericksen theory predicts a stable flow alignment for $\alpha_2, \alpha_3 < 0$ without any magnetic field. In this case the angle θ_0 between the velocity and the director becomes[2]

$$\tan^2 \theta_0 = \frac{\alpha_3}{\alpha_2} \tag{20}$$

In general, $|\alpha_3| \ll |\alpha_2|$ is valid and θ_0 becomes very small. Thus only minor influences of the flow alignment are to be expected in the case of the measurement of η_2.

For the following calculations the approximation of no elastic contributions is still maintained. In this case the Leslie–Ericksen theory gives

$$\frac{dp}{dz} = (\eta_1 \sin^2 \theta + \eta_2 \cos^2 \theta) \frac{d^2 v}{dx^2} \tag{21}$$

and

$$\frac{dv}{dx} = \frac{\chi_a B^2}{\mu_0(\alpha_2 \tan \theta - \alpha_3 \operatorname{ctg} \theta)} \tag{22}$$

Assuming $\tan^2 \theta \ll \alpha_3/\alpha_2$ and $\theta \ll 1$ the same procedure as in Section a gives

$$\theta = \beta_2 x \quad \text{with} \quad \beta_2 = -\frac{\alpha_3 \mu_0}{\eta_2 \chi_a B^2} \frac{dp}{dz} \tag{23}$$

For small values of θ Eq. (21) can be written as

$$\frac{d^2v}{dx^2} = \frac{1}{\eta_2}\frac{dp}{dz}\left[1 - \theta^2\left(\frac{\eta_1}{\eta_2} - 1\right)\right] \tag{24}$$

Insertion of Eq. (23) and triple integration gives

$$\bar{v} = -\frac{a^2}{12\eta_2}\cdot\frac{dp}{dz}\left[1 - \frac{\beta_2^2 a^2}{20}\left(\frac{\eta_1}{\eta_2} - 1\right)\right] \tag{25}$$

The effective viscosity coefficient is obtained as

$$\frac{1}{\eta_2^{\text{eff}}} = \frac{1}{\eta_2} - \frac{\beta_2^2 a^2}{20\eta_2}\left(\frac{\eta_1}{\eta_2} - 1\right) \tag{26}$$

c Measurement of η_3

The Leslie–Ericksen theory predicts that in this case no torque is exerted on the director. Thus, no change of the effective viscosity coefficient with the magnetic induction should be observed.

Appendix III

a NUMERICAL VALUES OF THE VISCOSITY COEFFICIENTS

t/°C	η_1/Pa s	η_2/Pa s	η_3/Pa s	η_{12}/Pa s
20	0.1891	0.0308	0.0548	−0.0297
25	0.1361	0.0240	0.0413	−0.0181
30	0.1007	0.01927	0.0322	−0.0141
35	0.0741	0.01596	0.0257	−0.0095
40	0.0534	0.01377	0.0211	−0.0054
42	0.0456	0.01320	0.01972	−0.0036
44	0.0367	0.01323	0.01871	−0.0012
		= = = = =	= = = = =	
46		0.01852		
50		0.01585		
55		0.01323		

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b NUMERICAL VALUES OF THE VISCOSITY COEFFICIENTS IN THE ISOTROPIC PHASE MEASURED WITH AN UBBELOHDE VISCOSIMETER

t/°C	η/Pa s
46.00	0.01865
49.99	0.01597
55.00	0.01332
59.97	0.01127
65.00	0.00959

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