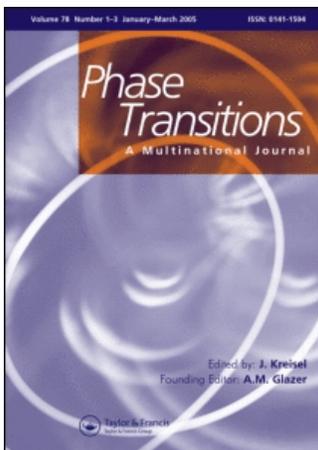


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## Physical characterisation of 4'-butyl-4-heptyl-bicyclohexyl-4-carbonitrile

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We report measurements of birefringence, bend elastic constant, principal dielectric constants and conductivities as functions of temperature in the nematic phase of 4'-butyl-4-heptyl-bicyclohexyl-4-carbonitrile (CCN-47). The compound exhibits a small birefringence and a large negative dielectric anisotropy. Surprisingly, it shows a *negative* conductivity anisotropy, which probably owes its origin to the structural details of the molecules. The orientational order parameters ( $S$ ) are estimated from both birefringence and dielectric anisotropy, and they agree well in the entire nematic range. The experimental results are compared with those on CCH7, which has a similar core structure but with a longitudinal dipole moment, in place of the transverse one of CCN-47.

**Keywords:** liquid crystals; birefringence; dielectric anisotropy; elastic constant

### 1. Introduction

A large number of nematic liquid crystals with elongated molecules exhibit positive dielectric anisotropy whereas a comparatively smaller number of compounds are known to exhibit negative dielectric anisotropy. Liquid crystals with positive dielectric anisotropy have been investigated extensively because of their industrial importance, and also as compounds with strong longitudinal dipoles exhibit smectic polymorphism [1].

The study of liquid crystal materials with negative dielectric anisotropy are also becoming important after the successful development of active matrix liquid crystal displays using the vertically aligned mode, in-plane switching mode etc. In this paper, we report the measurements of the temperature dependences of birefringence, dielectric and conductivity anisotropies and bend elastic constant of a nematic liquid crystal with a large negative dielectric anisotropy. We also compare these results with the results on a compound with a similar molecular core structure, but having a longitudinal dipole moment.

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## 2. Experimental

The sample of 4'-butyl-4-heptyl-bicyclohexyl-4-carbonitrile (CCN-47) was obtained from Merck and used without further purification. The chemical structure of the molecule is shown in Figure 1. It has the following sequence of phase transitions: Cr 28 SmA 30.6 N 59.7 I. The compound exhibits a wide temperature nematic range and a low temperature smecticA phase, which easily supercools to room temperature. The compound is chemically stable. Systematic dielectric investigations on the homologous series of CCN compounds, except CCN-47, were reported by Haase et al. [2]. Detailed high electric field experiments on CCN-47 have been reported by us recently [3–4].

Planar aligned sample cells were prepared by using two ITO (indium tin oxide)-coated glass plates, which were treated with polyimide and cured at a temperature of 280°C for 90 min before being rubbed unidirectionally. For the homeotropic alignment the plates were treated with ODSE (octadecyloxy silane) and cured at 150°C for 90 min. The plates of the cell were separated by appropriate spherical glass beads and sealed with epoxy glue. The thickness of the cell was measured by an interference technique. Typical cell thickness used for the experiments were in the range of ~6–16 μm. The electrical connections to the two plates were made through copper wires, which were soldered using an ultrasonic soldering gun. The cell was filled with the sample in the isotropic phase and on slow cooling to the nematic range, a well-aligned sample was obtained. All the experiments were conducted while cooling the sample from the isotropic phase.

The block diagram of the experimental setup is shown in Figure 2. The cell was mounted in a hot stage (INTEC HS1) which itself was kept on the rotating stage of a polarising microscope (model Leitz, ORTHOPLAN). The temperature was controlled to an accuracy of 0.008°C. The dielectric properties of the sample were measured using a lock-in amplifier (SRS-830). The sinusoidal output of the internal oscillator of the lock-in amplifier was adjusted to an rms value of ~0.2 V, and applied to the cell which was in series with a 1 μF standard capacitor  $C_m$  as shown in Figure 2. The amplitude and phase of the applied voltage were also checked using a reference circuit with two standard resistors ( $R_1 = 1\text{M}\Omega$  and  $R_2 = 100\ \Omega$ ) in series, connected in place of the cell. The phase ( $\varphi_m$ ) and the rms amplitude ( $V_m$ ) of the voltage measured across the series capacitor ( $C_m$ ) were used to calculate the capacitance ( $C_S$ ) and parallel resistance ( $R_S$ ) corresponding to the cell by using the following relations [5]

$$R_S = \frac{Y}{\omega \sin \alpha}, \quad (1)$$

$$C_S = \frac{X}{Y}, \quad (2)$$



Figure 1. The energy minimised configuration of a CCN-47 molecule.

where  $X = \cos \alpha - Q$ ,  $Y = \sin^2 \alpha + X^2/C_m Q$ ,  $Q = V_m/V_o$ ,  $\alpha = \phi_m - \phi_0$ ,  $V_o$  and  $\phi_0$  are the amplitude and phase of the input signal, respectively and  $\omega = 2\pi f$ ,  $f$  being the frequency of the applied signal. The typical frequency used in the experiment is 4111 Hz. We have checked the calibration of the setup by connecting standard capacitors in parallel with standard resistors in place of the liquid crystal cell. The measured values agree with the standard values to within 3%. We have also checked the calibration by measuring the capacitance and resistance using a Wayne–Kerr bridge which operates at 1.6 kHz. The dielectric constant is given by the ratio  $C_S/C_0$ , where  $C_0$  is the capacitance of the empty cell.

A laser (He–Ne,  $\lambda = 632.8$  nm) beam is passed through a polariser (POL) and made to be incident on the sample. The transmitted beam is passed through an analyser (ANL), which is crossed with respect to the polariser. A photodiode (PD1) is used to measure the transmitted intensity. The stability of the laser intensity is monitored by another photodiode (PD2). A multimeter (Keithley model 2000) measures the output voltages of both the photodiodes. The temperature variation of the transmitted intensity is measured in the planar aligned sample. The transmitted intensity is given by

$$I_t = \frac{\sin^2 2\theta}{2} (1 - \cos \Delta\varphi) \quad (3)$$

where  $\theta$  is the angle made by the polariser with the optic axis and the phase difference

$$\Delta\varphi = \frac{2\pi}{\lambda} \Delta n(d). \quad (4)$$

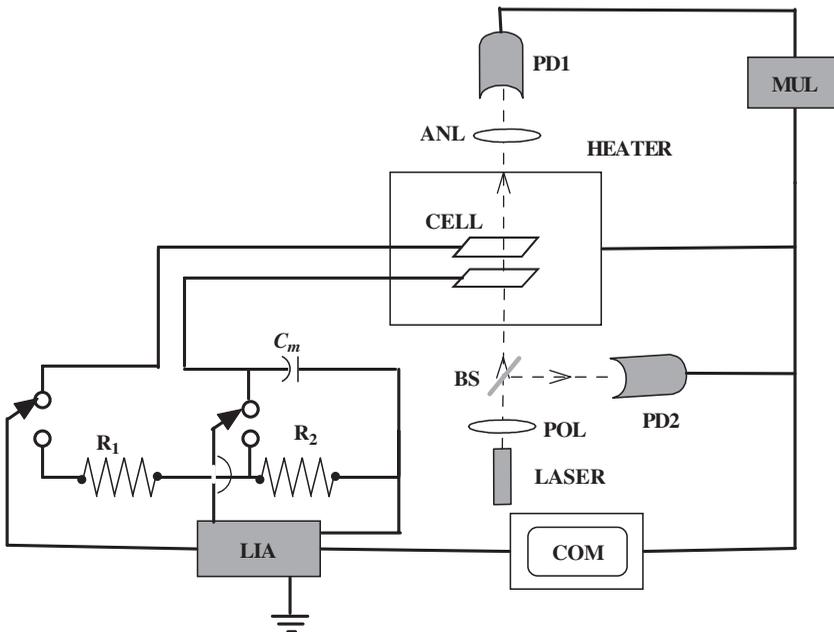


Figure 2. Schematic diagram of the experimental setup: photodiodes (PD1, PD2), polariser (POL), analyser (ANL), multimeter (MUL), voltage amplifier (AMP) lock in amplifier (LIA), resistances ( $R_1, R_2$ ), computer (COM).

$\Delta n = n_e - n_o$ , where  $n_e$  and  $n_o$  are the extraordinary and ordinary refractive indices of the liquid crystal medium, and  $d$  is the sample thickness. The angle  $\theta$  is set at  $45^\circ$  to optimise the measurements. The birefringence is calculated from the measured intensity. The measurements are completely controlled by a computer, using a suitable program.

### 3. Results and discussion

The minimum energy configuration of the molecule was obtained using a standard software package (Gaussian 98). The optimised structure is shown in Figure 1. The estimated transverse dipole moment is 3.14D. The variation of  $\Delta n$  over a wide temperature range ( $\sim 20^\circ\text{C}$ ) in the nematic phase is shown in Figure 3.  $\Delta n$  is  $\sim 0.016$  at  $0.5^\circ\text{C}$  below  $T_{\text{NI}}$  and increases to 0.031 at  $20^\circ\text{C}$  below  $T_{\text{NI}}$ . The relatively small optical anisotropy reflects the saturated nature of the rings of the molecules. We had measured earlier [6] the temperature dependence of birefringence of CCN-47 using a tilting compensator and cells of thicknesses 1.5 and  $6.7\ \mu\text{m}$ . The birefringence and hence the order parameter measured in the two cells were found to agree [6]. In the present study, the birefringence has been measured from the transmitted intensity on a cell of thickness  $16.8\ \mu\text{m}$  and the values of  $\Delta n$  are a few percent lower than those reported earlier. Measurements on other samples show that the birefringence and hence the order parameter increase as the thickness of the cell is decreased, whose origin is not yet understood [6]. The birefringence of *trans*, *trans*-4-*n*-heptyl-4'-cyanobicyclohexane (CCH-7) has been measured by Ibrahim et al. [7]. It has a molecular core structure similar to that of CCN-47, but with a longitudinal orientation of the polar cyano group instead of the transverse one in the latter. We also show the variation of  $\Delta n$  of CCH-7 with temperature in Figure 3. CCH-7 exhibits  $\sim 30\%$  larger birefringence near the NI transition point compared to CCN-47, and the difference is increased as the temperature is lowered in the nematic phase. The longitudinal dipole moment in CCH-7 increases the longitudinal polarisability and hence the birefringence in CCH-7 is larger as compared to that of CCN-47 with a transverse dipole moment. Further, the temperature variation of  $\Delta n$  is steeper in CCH-7 compared to that in CCN-47. In CCH-7 the strongly polar cyano group is essentially along the long axis of the molecule, leading to antiparallel near neighbour interactions [8]. This leads to the formation of antiparallel pairs, as seen by X-ray

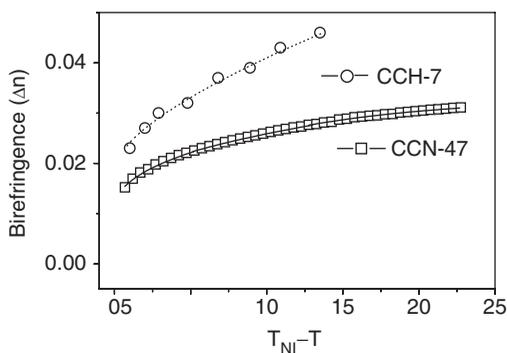


Figure 3. Variations of  $\Delta n$  as functions of temperature in CCN-47 and CCH-7. Open squares are the experimental data on CCN-47 and the continuous line is a theoretical fit to Equation (6). Cell thickness:  $16.8\ \mu\text{m}$ . Open circles correspond to data of CCH-7 obtained from ref. [7] and the dotted line is drawn as a guide to the eye.

scattering studies [9]. The pair formation can be expected to become efficient at lower temperatures of the nematic phase, which effectively increases the molecular length and hence the anisotropies in polarisabilities and refractive indices. In CCN-47 the dipoles are essentially in a transverse direction, and in the absence of the above mechanism, the temperature variation of birefringence is weaker.

We have measured the perpendicular component of dielectric constant ( $\epsilon_{\perp}$ ) using a homogeneously aligned sample and parallel component of dielectric constant ( $\epsilon_{\parallel}$ ) using a homeotropically, aligned sample. The cell thicknesses used for the experiments are 8.5 and 7  $\mu\text{m}$ , respectively, in the two cases. Both the measurements are made at an applied voltage of 0.2 V and a frequency of 4111 Hz. The applied voltage is much lower than the threshold voltage for Fredericksz transition in the homeotropic cell. Electro-hydrodynamic instability was not seen in the nematic phase in either geometry, except very close to the nematic–smecticA transition temperature. The variations of dielectric constants  $\epsilon_{\parallel}$  and  $\epsilon_{\perp}$  are shown as functions of temperature in Figure 4. The dielectric constant in the isotropic phase obtained from the two different geometries matched to within 1%.  $\epsilon_{\perp}$  is larger than  $\epsilon_{\parallel}$  in the nematic as well as in the smectic phases, the N–SmA transition occurring at  $T_{\text{NI}} - T = 29^{\circ}\text{C}$ . The variation of  $\epsilon_{\parallel}$  across the N–SmA transition temperature is smooth whereas a small increase is observed in  $\epsilon_{\perp}$ , reflecting increased correlations of the transverse dipoles in the layered structure. The dielectric anisotropy  $\Delta\epsilon = (\epsilon_{\parallel} - \epsilon_{\perp})$  near the nematic–isotropic transition point is about  $-3$  and increases to about  $-6$  at  $30^{\circ}\text{C}$  below  $T_{\text{NI}}$ . The mean dielectric constant  $\bar{\epsilon}$  is calculated using the formula  $\bar{\epsilon} = (\epsilon_{\parallel} + 2\epsilon_{\perp})/3$ . At  $T_{\text{NI}}$ ,  $\bar{\epsilon} = \epsilon_{\text{is}} \approx 7.2$  and as the temperature is lowered to the N–SmA transition point,  $\bar{\epsilon}$  increases. The increase in  $\bar{\epsilon}$  is  $\sim 4\%$  at  $T_{\text{NI}} - 15^{\circ}\text{C}$  (N phase) and  $\sim 10\%$  at  $T_{\text{NI}} - 30^{\circ}\text{C}$  (in the SmA phase). The contribution of the molecular dipole moment to the average dielectric constant is given by [10]

$$\bar{\epsilon} = 1 + 4\pi \frac{N_A \rho h F}{M} \left( \bar{\alpha} + \frac{F \mu^2}{3k_B T} \right). \quad (5)$$

where  $N_A$  is the Avogadro number,  $\rho$  the density,  $M$  the molecular weight,  $h$  is the cavity field factor and  $F$  is the reaction field factor for spherical cavity and  $\bar{\alpha}$ , is the

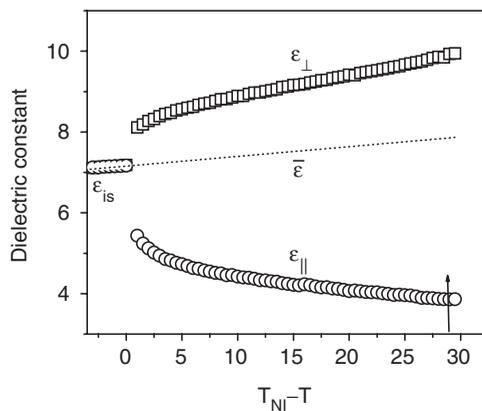


Figure 4. Variations of parallel ( $\epsilon_{\parallel}$ ) and perpendicular ( $\epsilon_{\perp}$ ) components of dielectric constant as functions of temperature. Applied voltage: 0.2 V and frequency: 4111 Hz.  $\epsilon_{\text{is}}$  is the dielectric constant in the isotropic phase. The mean value in the nematic phase is  $\bar{\epsilon} = (\epsilon_{\parallel} + 2\epsilon_{\perp})/3$ , which is shown by a dotted line. The vertical arrow denotes N–SmA transition temperature.

average polarisability. The second term in the bracket is due to the contribution of the orientational polarisation of the dipole moments.  $\bar{\epsilon}$  can increase with decreasing temperature due to the increase in the density ( $\rho$ ) and the contribution from orientation polarisation. Experimentally, it is reported that in a non-polar compound  $\bar{\epsilon}$  increases by  $\sim 2\%$  at  $15^\circ\text{C}$  below NI transition temperature due to an increase in density alone [11]. The excess variation in the highly polar compound used by us is due to the contribution of the orientation polarisation, which increases with decreasing temperature as seen in Equation (5). It may be pointed out that in the case of cyanobiphenyl compounds there is a slight increase in the  $\bar{\epsilon}$  when the temperature is increased from nematic to isotropic phase due to a reduction in short range antiferroelectric order [8]. In the case of CCH compounds,  $\bar{\epsilon}$  exhibits a maximum as the temperature is increased above the NI transition point. This trend was interpreted [12] as arising from the formation of antiparallel dimers at lower temperatures, due to local antiferroelectric correlations between the highly polar end groups, as was discussed earlier. In CCN-47 with a *transverse* dipolar group, such an increase is not observed. Similar observations have also been made by other workers on other compounds of the homologous series of CCN [2].

We have also measured the anisotropic ionic conductivities of the sample as functions of temperature at a frequency 4111 Hz. The variation of normalised conductivity  $\sigma/\bar{\sigma}$  is shown in Figure 5 where the mean conductivity  $\bar{\sigma}$  is calculated using the formula  $\bar{\sigma} = (\sigma_{\parallel} + 2\sigma_{\perp})/3$ . Curiously, the perpendicular component of conductivity ( $\sigma_{\perp}$ ) is larger than the parallel component ( $\sigma_{\parallel}$ ) in the nematic as well as in the smectic phases. Both the conductivities vary continuously across the NI transition. The conductivity anisotropy  $\Delta\sigma = (\sigma_{\parallel} - \sigma_{\perp})$  at  $T_{\text{NI}} - 3^\circ\text{C}$  is  $\approx -1.5 \times 10^{-8}(\text{ohm m})^{-1}$  and increases to  $-6 \times 10^{-8}(\text{ohm m})^{-1}$  at  $30^\circ\text{C}$  below  $T_{\text{NI}}$ . At  $T_{\text{NI}}$ ,  $\bar{\sigma} \approx 9.6 \times 10^{-8}(\text{ohm m})^{-1}$  and as the temperature is lowered to the N-SmA transition point,  $\bar{\sigma}$  decreases to  $\approx 9 \times 10^{-8}(\text{ohm m})^{-1}$ . The negative anisotropy of conductivity in the entire liquid crystalline range of CCN-47 is very unusual. In the nematic phase made of rod like molecules,  $\Delta\sigma$  is usually positive, as the ions can move more freely along the director than in a perpendicular direction [1]. In the SmA phase, the ion motion is subjected to the periodic smectic potential along the director, and the resulting hopping motion can lower  $\sigma_{\parallel}$  substantially. On the other hand, the ion motion along the liquid layers is not affected by the smectic

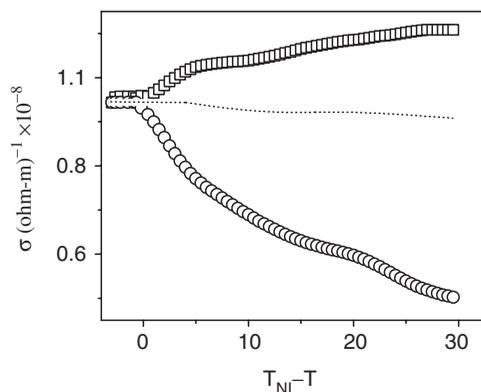


Figure 5. Variations of normalised conductivities  $\sigma_{\perp}/\bar{\sigma}$  (open squares) and  $\sigma_{\parallel}/\bar{\sigma}$  (open circles) as functions of temperature. Applied voltage: 0.2 V and frequency: 4111 Hz. The mean value of the conductivity in the nematic phase is  $\bar{\sigma} = (\sigma_{\parallel} + 2\sigma_{\perp})/3$ , which is shown by a line.

potential, and  $\Delta\sigma$  often changes sign near the N-SmA transition point [13], becoming negative in the smectic phase. In the case of CCN-47, the negative anisotropy even close to  $T_{\text{NI}}$  probably owes its origin to the chemical structure of the compound. The unknown ions in the sample are likely to be associated with highly polarisable species. These will have a much larger affinity to the cyano group of the CCN-47 molecule (Figure 1) than to the remaining weakly polarisable part. When the ions move under the action of an external electric field, presumably, they tend to do so by hopping between the cyano groups of neighbouring molecules. The statistically averaged separation between the cyano groups can be expected to be considerably smaller in a direction orthogonal to the director than in one parallel to it, in view of the rod shape of the molecules. This can in turn lead to the negative conductivity anisotropy. As the temperature is lowered towards the smectic phase, the smectic like short range order builds up, and the anisotropy of average separation, and hence that of the conductivity, also get enhanced (Figure 5).

We can estimate the orientational order parameter in the liquid crystalline phases using the birefringence as well as dielectric anisotropy data.  $S = \Delta n / \Delta n_o$ . The birefringence  $\Delta n_o$  of a completely ordered sample is determined from the temperature dependence of  $\Delta n$ , which can be approximated well for nematic liquid crystals by [14]

$$\Delta n = \Delta n_o \left(1 - \frac{T}{T_1}\right)^\beta \quad (6)$$

where  $\Delta n_o$ ,  $T_1$  and  $\beta$  are adjustable parameters. A least squares fit to the experimental data with Equation (6) is shown in Figure 3. The value of the fitted parameters are  $\Delta n_o = 0.0576$  and  $\beta = 0.23$ . The value of  $\beta$  is typical for nematic liquid crystals [1]. The variation of orientational order parameter is shown as a function of temperature in Figure 6. The jump in the order parameter at  $T_{\text{NI}}$  is approximately 0.27. The orientational order parameter was also estimated using the formula  $S \approx \Delta\epsilon / \Delta\epsilon_o$ . The value of  $\Delta\epsilon_o$  was estimated comparing the order parameter value measured from  $\Delta n$  data at  $T_{\text{NI}} - T = 1^\circ\text{C}$ . The temperature variations of the order parameter estimated from the two sets of experimental data agree well in the entire temperature range except very close to the N-SmA transition point.

We have also measured the Fredericksz threshold voltage ( $V_{\text{th}}$ ) at several temperatures in a homeotropically aligned cell. The measurement is made at a frequency of 4111 Hz and the voltage was increased in steps of 10 mV. The threshold voltage is given

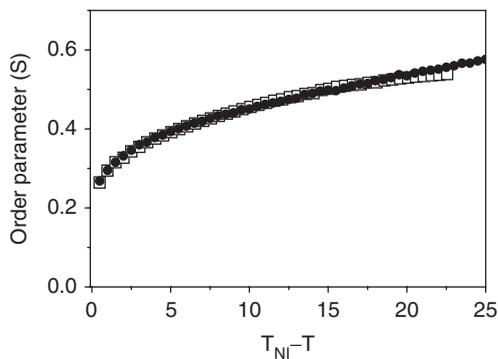


Figure 6. Variation of orientational order parameter ( $S$ ) with temperature estimated from the birefringence data (solid circles) and from dielectric data (open squares).

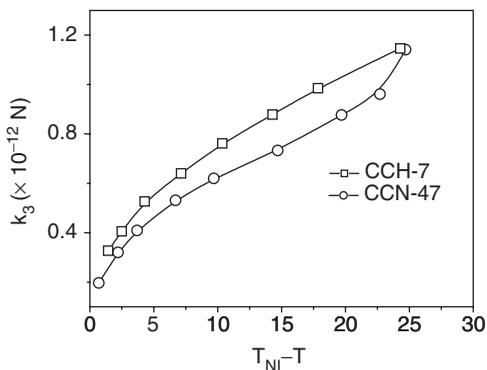


Figure 7. Variations of the bend elastic constant  $K_3$  as functions of temperature in CCN-47 and CCH-7. Points are experimental data and the continuous lines are drawn as guides to the eye. Open circles: CCN-47 and open squares: CCH-7 (from ref. [15]).

by  $V_{th} = \pi \sqrt{K_3 / \epsilon_0 \Delta \epsilon}$  where  $\Delta \epsilon$  now stands for the magnitude of dielectric anisotropy,  $K_3$  is the bend elastic constant and  $\epsilon_0$  is the dielectric constant of the free space. Using  $V_{th}$  and  $\Delta \epsilon$  (Figure 4) we calculate the bend elastic constant ( $K_3$ ). The variation of  $K_3$  as a function of temperature is shown in Figure 7.  $K_3$  is  $\sim 0.2 \times 10^{-12}$  N at the temperature of  $T_{NI} - 1^\circ\text{C}$  and follows an  $S^2$ -dependence near  $T_{NI}$ , where  $S$  is the order parameter.  $K_3$  increases rapidly as the nematic to smectic transition point is approached, as the smectic like short range order builds up [1]. The temperature variation of  $K_3$  of CCH-7 was measured by Schad et al. [15], and that data is also shown in Figure 7. It is found that  $K_3$  in CCN-47 is smaller in the entire nematic range compared to that of CCH-7. The higher value in CCH-7 probably owes its origin to the formation of antiparallel dimers which increase the effective length of the molecules, as described earlier.

#### 4. Conclusions

We have measured the birefringence, dielectric and conductivity anisotropies and the bend elastic constant of the compound 4'-butyl-4-heptyl-bicyclohexyl-4-carbonitrile as functions of temperature. The material exhibits a low birefringence and a large negative dielectric anisotropy. The estimated order parameters from the birefringence and dielectric anisotropy agree well in the entire nematic range. Unusually, the compound also exhibits a *negative* conductivity anisotropy probably because of the chemical structure of the compound. Electrohydrodynamic instabilities (EHD) are *not* observed in the nematic phase, as the Carr–Helfrich mechanism does not work when both  $\Delta \sigma$  and  $\Delta \epsilon$  are negative [1]. However, the compound exhibits a weak EHD near the smectic transition point under a low frequency ac field, which probably owes its origin to a flexoelectric mechanism [16]. The measured bend elastic constant ( $K_3$ ) increases as the temperature is lowered in the nematic phase and diverges near the nematic–smectic phase transition due to the rapid increase in smectic correlation length.

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