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Flexoelectric measurements of a bent-core nematic liquid crystal

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A large flexoelectric polarization might be expected for a bent-core nematic liquid crystal, due to the combination of molecular shape and transverse dipole component. In this study a bent-core nematic compound is doped to be highly chiral, and measurements of the difference in flexoelectric coefficients ($e_1 - e_3$) are carried out by exploiting the chiral flexoelectro-optic effect. The measured flexoelectric coefficients are greater than those for many conventional calamitic nematics, but several orders of magnitude lower than recent reports on other bent-core compounds. The influence of the bent molecular shape on the nematic phase is evident from measurements of the elastic constants, where an unusually low ratio of k_3 to k_1 indicates that bend distortions of the director are relatively lower in energy compared to those involving splay.

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I. INTRODUCTION

In nematic liquid crystals the director field $\hat{\mathbf{n}}(\mathbf{r})$ characterizes the local thermodynamic average molecular orientation. Flexoelectricity describes a direct coupling between splay or bend deformations of the director field and an induced flexoelectric polarization $\mathbf{P}_{\mathbf{flexo}}$. Adopting the sign convention originally defined by Meyer [1], $\mathbf{P}_{\mathbf{flexo}}$ can be expressed as

$$\mathbf{P}_{\mathbf{flexo}} = e_1(\nabla \cdot \hat{\mathbf{n}})\hat{\mathbf{n}} + e_3(\nabla \times \hat{\mathbf{n}}) \times \hat{\mathbf{n}}, \tag{1}$$

where e_1 and e_3 are the splay and bend flexoelectric coefficients, respectively. Flexoelectric effects are expected to be greatest for molecules asymmetric in shape and possessing a strong permanent dipole. Prototype molecules with (1) a "drop" shape and dipole parallel to the long axis or (2) a "crescent" shape and perpendicular dipole were suggested as good candidates for high values of e_1 and e_3, respectively [1], as illustrated in Fig. 1. The recent synthesis of bent-core nematic liquid crystals with transverse dipoles [2] has presented ideal examples of the second variety of molecule.

There is currently great interest in materials with a bent molecular shape, following accounts that an ordering of the bend axis can be present in the nematic phase leading to a biaxial symmetry [3]. Furthermore such compounds would be expected to display strong flexoelectric behavior, and indeed there have been reports of e_3 magnitudes 1000 times greater than is generally the case for nematics [4]. Additionally it has been shown that even a small concentration of bent-core compounds doped into a conventional calamitic ("rod-shaped") nematic can lead to a significant enhancement of flexoelectric behavior [5]. The "giant" flexoelectric values are attributed not only to the molecular shape anisotropy but additionally, and perhaps more crucially, to a short-range molecular clustering [4]. There is some discrepancy in the measurement of flexoelectricity in bent-core compounds, though, with other recent work finding that there is little difference in the magnitude of e_3 to conventional calamitic nematics [6,7]. There is significant technological potential for materials that exhibit "giant" flexoelectricity, ranging from electro-optic to electro-mechanical applications. However, it is clear that any exploitation of the effect requires further work exploring the relationship between molecular shape, short-range clustering, and flexoelectricity, in order to determine why "giant" flexoelectric values are observed in some systems while not others.

In this paper we report our findings on the nematic phase formed by the symmetric bent-core compound 4-cyanoresorcinol bisbenzoate (C6ban) [8], with chemical structure and phase transitions shown in Fig. 2. Initially, the dielectric and elastic properties of the nematic phase are characterized, prior to measurements of the difference in flexoelectric coefficients ($e_1 - e_3$), which are carried out by exploiting the flexoelectro-optic effect in the uniform lying helix (ULH) structure [9].

II. FLEXOELECTRIC MEASUREMENTS

Measurements of flexoelectric behavior are in general hindered by ionic screening of the electric field. The free-energy density associated with coupling between the flexoelectric polarization $\mathbf{P}_{\mathbf{flexo}}$ and an applied electric field $\mathbf{E}$ is described by $f_{\text{flexo}} = -\mathbf{P}_{\mathbf{flexo}} \cdot \mathbf{E}$ and results in flexoelectric reorientation dependent on the field polarity. This is in contrast to dielectric reorientation, which responds to the root mean square (r.m.s.) of the applied field. Flexoelectric reorientation tends to be relatively slow, forcing experiments to be performed at very low frequency [10], where ionic motion can lead to significant modification of the internal electric field [11,12]. Such ionic perturbations to the internal field complicate the extraction of the flexoelectric coefficients from any experimental measurements. However, by considering the flexoelectro-optic effect in chiral nematics [9], the length scale associated with a flexoelectric reorientation in the field decreases as the pitch is reduced, and as such response times for short pitch materials are sufficiently low for measurements to be undertaken at a.c. frequencies for which ionic screening effects should be reduced.

The flexoelectro-optic effect observed in chiral nematics is manifest for an electric field applied perpendicular to the helix axis [9]. A tilt of the director with respect to the helix axis about the direction of the field forms a periodic splay-bend distortion with a flexoelectric polarization parallel to the field.

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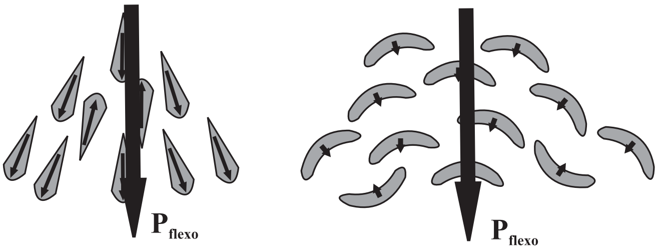

FIG. 1. Molecules with a "drop" shape and longitudinal dipole or "crescent" shape and transverse dipole may be expected to exhibit large coefficients for splay and bend flexoelectricity, respectively.

Minimizing the free energy of the system yields an expression for the tilt angle ϕ of the director planes with respect to the helix axis as

$$\tan\phi = \frac{(e_1 - e_3)pE}{2\pi(k_1 + k_3)}, \tag{2}$$

where E is the magnitude of the applied electric field, k_1 and k_3 are the splay and bend Frank elastic constants, and p is the chiral pitch length.

In the ULH structure, the helical axis of a chiral nematic is aligned in a single direction parallel to the substrates. For pitch lengths similar to the wavelength of the illuminating light, the device appears as a negative birefringence uniaxial slab with the optic axis perpendicular to the plane containing the director. For an electric field applied between the substrates, the subsequent flexoelectric reorientation is manifest macroscopically as an in-plane rotation of the optic axis by angle ϕ. A measurement of the magnitude and direction of the tilt angle yields the magnitude and sign of $(e_1 - e_3)$ from Eq. (2), provided the elastic constants and pitch of the material are also known [13].

III. EXPERIMENT

Initially devices were assembled to measure the dielectric and elastic constants of C6ban. The devices used comprised a pair of parallel glass substrates coated with a thin layer of the transparent conductor indium tin oxide and separated by glass microspheres of mean diameter 5 μm. One set of devices was constructed with a uniform layer of the surfactant *N*,*N*-dimethyl-*n*-octadecyl-3-aminopropyltrimethoxysilyl chloride spin coated onto the substrates to provide a conventional homeotropic alignment. The device was filled by capillary action while the material C6ban was in the isotropic state, and on cooling the isotropic to nematic phase transition was identified at $T_{\text{NI}} = 105\,^{\circ}$C. The device situated between crossed polarizers revealed a vanishing transmission with increasing voltage applied perpendicular to the substrates, at a frequency (1 kHz) where the material is known to display a positive dielectric anisotropy [14]. These observations provide a strong indication that the phase is macroscopically uniaxial [15] and will be assumed so in the remainder of this work.

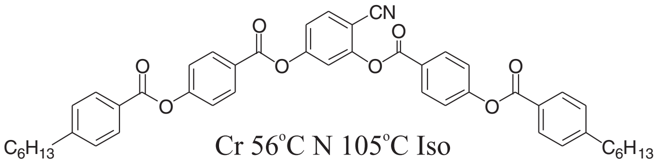

FIG. 2. Molecular structure and phase transitions for the bent-core compound C6ban under investigation (transition temperatures were obtained by polarizing microscopy of a previously melted sample after annealing for 30 minutes at 100 °C).

A second set of devices were constructed with a polyimide (JSR, JALS 9005) layer spin coated onto the substrates and rubbed to provide planar alignment. The device assembly was such that the rubbing directions on each substrate were antiparallel. The devices were filled with the bent-core nematic C6ban by capillary action and mounted in a micro-oven (Mettler-Toledo FP90, which allowed temperature control within 0.1 °C). There was a uniform tilt of the director across the device (the surface pretilt), which was measured to be 5.7° and independent of temperature via the crystal rotation method [16]. An electric field was applied perpendicular to the substrates, and an Agilent 4284A LCR meter was used to monitor the effective dielectric permittivity of the liquid crystal layer ϵ_{eff} as a function of applied voltage for a frequency of 1 kHz. As the voltage was increased through the Freedericksz threshold, the device was also observed between crossed polarizers to confirm uniform director reorientation. The values of the elastic constants k_1 and k_3 were extracted from fitting the predictions of a one-dimensional continuum theory model to the dielectric permitivity data [17]. Measurements of the Fabry-Perot spectrum for the device illuminated with light polarized parallel and perpendicular to the rubbing direction were used to establish the refractive indices [18].

As discussed above, determination of $(e_1 - e_3)$ by the flexoelectro-optic effect is preferably carried out for highly chiral liquid crystals, since the low response times allow measurements to be taken at high-frequency driving voltages minimizing the screening effects of ionic impurities. Therefore, for the flexoelectric measurements, a small amount ($\sim$1% by weight) of the chiral agent R5011 (Merck Chemicals) was added to the compound. R5011 is a right-handed dopant with a helical twisting power (HTP) of $\sim$100 μm^{-1} quoted for conventional calamitic nematics. The optical pitch, $\bar{n}p$, was determined through measurement of the selective reflection band for light normally incident on a planar device aligned in the Grandjean structure (helix axis perpendicular to the substrates). The average refractive index, $\bar{n}$, was 1.58 and approximately constant throughout the temperature range, allowing the optical pitch to be converted to an actual value p. The pitch was found to increase from 543 nm at 1 °C below the isotropic to nematic transition temperature T_{NI} to 630 nm at 10 K below T_{NI}. This relates to a variation in the HTP of 160–185 μm^{-1}. Thus the HTP is found to be significantly increased for the dopant inserted into the bent-core host C6ban relative to conventional calamitics and provides a first indication of the effects of the bent molecular shape on the properties of the nematic phase. These observations correlate with other recent studies on the chiral properties of bent-core compounds, where it is found that despite achirality of the constituent molecules spontaneously twisted structures may form [19,20], and there is a chirality enhancement when doped into an initially cholesteric phase [21].

Measurements of the flexoelectric coefficients $(e_1 - e_3)$ were carried out in the ULH structure. In order to form the

ULH, an electric field is used to orient the helix axis parallel to the surfaces, by coupling to the dielectric anisotropy in the positive $\Delta\epsilon$ regime, and the surface alignment directions break the in-plane degeneracy of the helix axis. The device was cooled from the isotropic phase with an applied electric field of 4 V μm^{-1} at a frequency of 100 Hz. As the temperature is further reduced the ULH structure grows, with the helix axis uniformly aligned roughly perpendicular to the rubbing direction. The magnitude of the flexoelectric tilt angle ϕ equals the in-plane rotation of the optic axis and was measured for a particular applied voltage by monitoring the transmission as a function of device rotation between crossed polarizers, as described elsewhere [22].

IV. RESULTS

The relative dielectric permittivity of the device is plotted as a function of voltage in Fig. 3 for a range of temperatures. The dielectric anisotropy is observed to be extremely small ($\Delta\epsilon \lesssim 0.25$) and decreases as the temperature is lowered, because the dielectric relaxation frequency for the material nears the driving frequency of the applied voltage. The device lacks a sharp Fréedericksz threshold voltage for reorientation of the director due to the large pretilt angle, measured as 5.7° from the crystal rotation method. Therefore it is not possible to use analytic expressions for the extraction of the elastic constants k_1 and k_3 [17], and a numerical model is used to fit the predictions of continuum theory to the dielectric permittivity data. If θ is used to denote the angle between the director and the substrate, and the director is confined to a single plane, the Euler-Lagrange equation minimizing the free energy of the system is

$$(k_{11}\cos^2\theta + k_{33}\sin^2\theta)\theta''' + [(k_{33}-k_{11})(\theta'')^2 + \epsilon_0\Delta\epsilon E^2]\sin\theta\cos\theta = 0, \quad (3)$$

where z is the direction normal to the substrate, $E(z)$ is the local electric field, and primes indicate differentials with respect to z. Equation (3) is solved using a relaxation method to determine $\theta(z)$, which can accordingly be used to calculate the effective dielectric permittivity ϵ_{eff} of the layer. The boundary conditions employed are a pretilt θ_s of 5.7° with a surface anchoring strength $W_\theta = 1 \times 10^{-3}$ Jm^{-2}, and the latter was adjusted to provide the optimum fit for the data. We note that such a value of W_θ is typical for conventional calamitic materials on similar alignment layers. Extracted values for k_1 and k_3 are plotted as a function of temperature in Fig. 4.

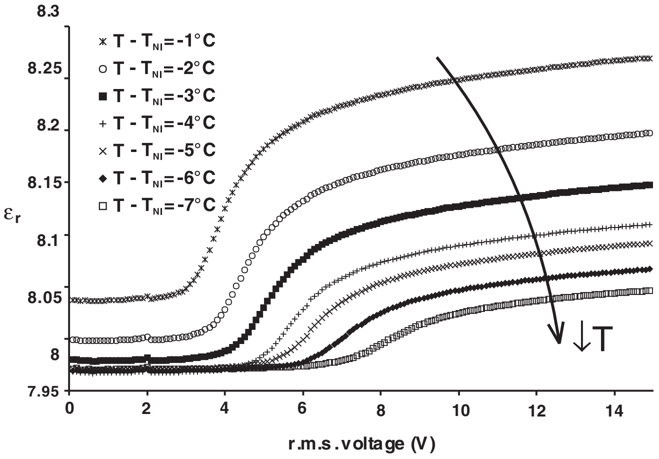

FIG. 3. A plot of the relative dielectric permittivity of the achiral planar device as a function of the applied root mean square (r.m.s.) voltage at a frequency of 1 kHz, displayed for a range of temperatures.

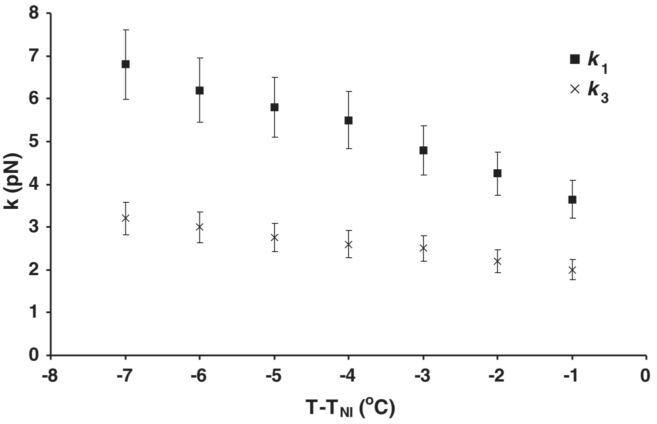

FIG. 4. Values of k_1 and k_3 for C6ban as a function of the shifted temperature $T_s = T - T_{\text{NI}}$. The values were obtained from fitting a one-dimensional continuum theory model to measurements of the dielectric permittivity as a function of applied voltage in planar devices. It is highly unusual for the ratio of k_3/k_1 to be lower than unity in calamitic nematics.

The ratio of $k_3/k_1 \sim 0.5$ found here for C6ban is unusually low in comparison to common calamitic nematics, for which k_3/k_1 is generally greater than unity. The elastic constants derived elsewhere for pure bent-core nematic compounds are a similar order of magnitude to the results here, but a higher ratio $k_3/k_1 \sim 1$ is still found [23]. It has been shown that using appropriate mixtures of compounds k_3/k_1 can be suppressed below unity [24–26], but the result is rare in pure compounds. The low value of k_3/k_1 thus seems to provide a direct link between the molecular shape and the macroscopic properties of the nematic phase. It suggests the bent shape of the molecules favors distortions of the director involving bend as opposed to those related to splay.

In determining k_1 and k_3, flexoelectric terms were omitted from Eq. (3), since the oscillation in the electric field (1 kHz) is assumed too fast for flexoelectric reorientation. However, the splay-bend distortion at each surface due to the dielectric torque has an associated flexoelectric polarization perpendicular to the substrate, leading to a d.c. offset in the internal electric field. It has been shown that this modification of the internal field reduces the value of the midplane tilt angle relative to the case of zero flexoelectric polarization [27]. As the director distribution is altered, so is the predicted effective dielectric permittivity at each voltage. Equally good fits to experimental data can be made using a range of flexoelectric values, by reducing k_3 as the magnitude of $(e_1 + e_3)$ rises. However, the exlusion of flexoelectricity from the model here is justified since it is anticipated that ions will largely screen out the induced d.c. flexoelectric offset in the field [28]. It is important to note, though, that in the absence of ionic screening the ratio k_3/k_1 would be rendered even smaller by the inclusion of flexoelectricity in the data fitting.

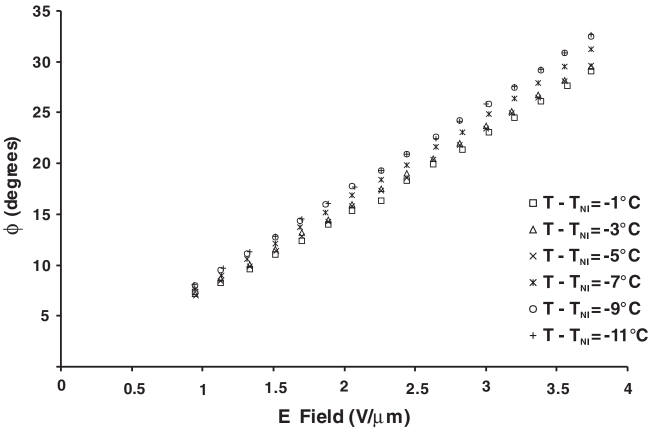

FIG. 5. The magnitude of the flexoelectrically induced rotation ϕ of the optic axis in the ULH structure as a function of applied electric field, for a range of temperatures.

Figure 5 presents the flexoelectrically induced rotation ϕ of the ULH optic axis as a function of the applied electric field for several shifted temperatures $T_s = T - T_{\mathrm{NI}}$. A large flexoelectro-optic effect is found, with complete optical modulation ($\phi = 22.5^\circ$) achieved for a field of 2.8 V μm^{-1}. The linearity of the rotation in the applied field is intriguing, since it is predicted from Eq. (2) that $\tan\phi$, not ϕ, should be linear in the field. In the derivation of Eq. (2) the chiral pitch p is allowed to vary from its unperturbed value p_0, and it is predicted that p will decrease as $p = p_0 \cos\phi$ [9]. An alternative treatment considering the case of a fixed chiral pitch yields a flexoelectric tilt angle dependence on the applied field as [29]

$$\tan\phi = \frac{(e_1 - e_3)pE}{4\pi k_2} - \frac{(k_1 - 2k_2 + k_3)\sin\phi}{2k_2}. \qquad (4)$$

Expanding the terms in ϕ to third order, it is found that when $k_1 + k_3 = 6k_2$ the tilt angle displays a linear dependence in the field, in agreement with the experimental data. The value of the elastic constant k_2 remains unknown from the experiments presented here, but the observed linearity in the flexoelectric response as a function of field suggests $k_2 = (k_1 + k_3)/6 \sim k_1/4 \sim k_3/2$ given that $k_3 \sim \frac{1}{2}k_1$ from Fig. 4. The tilt angle is then described by $\phi \approx (e_1 - e_3)E/q(k_1 + k_3)$.

The flexoelectric difference $(e_1 - e_3)$ is determined from the gradient of $\phi(E)$, utilizing the values of the pitch and elastic constants discussed above. The measurements of the elastic constants presented in Fig. 4 extend only to a temperature 7 °C below the isotropic to nematic transition since the dielectric relaxation of C6ban prevented further measurements on the splay Fréedericksz transition. Therefore values of k_1 and k_3 are extrapolated at lower temperatures when evaluating $(e_1 - e_3)$.

Figure 6 displays the resulting temperature dependence of $(e_1 - e_3)$ found for C6ban. The magnitude of $(e_1 - e_3)$ increases with a reduction in temperature reflecting a rise in the nematic order. The values of $|(e_1 - e_3)|$ for C6ban are found to be similar to those reported for an alternative fixed bent-core nematic compound [7] and flexible bimesogenic molecules that are optimized to form bent conformations [13,30].

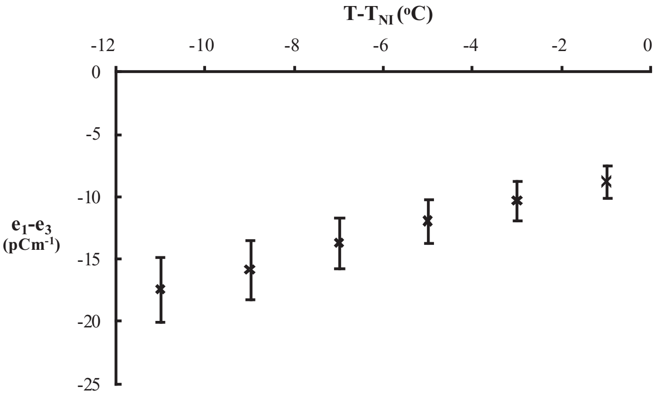

FIG. 6. The difference in flexoelectric coefficients $(e_1 - e_3)$ as a function of the shifted temperature $T - T_{\mathrm{NI}}$ for the bent-core compound C6ban.

V. CONCLUSION

It is often assumed that nematic liquid crystals with a bent molecular shape and a transverse dipole component will exhibit a large flexoelectric polarization. The assumption is confirmed by the experimental results presented for the flexoelectric difference of C6ban, where $|(e_1 - e_3)|$ values are found to be greater than those for conventional calamitic nematics possessing permanent dipole moments of similar magnitude (arising predominantly from the polar cyano group). For example, the cyanobiphenyl materials have $|(e_1 - e_3)|$ values typically less than 12pCm^{-1} at temperatures well below T_{NI} [22], while for C6ban $|(e_1 - e_3)| = 17.4$pCm^{-1} at 11 K below T_{NI}, a value that would be expected to rise further with additional increase in the nematic order if the temperature were reduced. However, $|(e_1 - e_3)|$ for C6ban is several orders of magnitude less than might be anticipated from the direct e_3 measurements of Harden *et al.* [4] for an alternative bent-core nematic compound 4-chloro-1,3-phenylene bis 4-[4′-(9-decenyloxy) benzoyloxy] benzoate. Harden *et al.* attribute the giant value of e_3 for ClPbis10BB to the existence of local polar clusters containing a few tens of molecules in the nematic phase [4]. Kumar *et al.* developed this idea further by proposing that "giant" flexoelectricity in bent-core nematics need not arise from polar clusters, but instead could be associated with apolar clusters and be quadrupolar in nature [6]. The flexoelectric coefficients would consequently be expected to scale linearly with the number of molecules in a cluster. If an external mechanical distortion could reorient the clusters, a much larger flexoelectric polarization would be expected than for reorienting individual molecules.

Small-angle X-ray scattering data on C6ban has indicated the presence of SmC cybotactic groups throughout the nematic range [8]. The clusters are not necessarily polar, although the SmC phase of bent-core compounds is often ferroelectric, even if the constituent molecules are not chiral, due to a double symmetry breaking within the smectic layers [31]. This therefore suggests that the compound might be expected to exhibit large flexoelectric behavior, similar to that observed in ClPbis10BB. However, it appears that any enhancing effects of the clusters on the flexoelectric coefficients are suppressed in the measurements reported here. It is possible that the cluster flexoelectricity is not observed due to the speed of oscillation of the electric field. The effective rotational viscosity for reorienting a cluster would be expected to be larger than that for a single molecule, and as such the flexoelectric response time longer. Therefore the flexoelectric response observed here may be that arising from aligning individual molecules, while a secondary larger effect due to the reorientation of clusters may be lost due to the finite period of the field. Alternatively if the cluster flexoelectricity is quadrupolar as suggested by Kumar *et al.* [6], the dependence of the flexocoefficients on the molecular shape is removed, and e_1 should be of similar magnitude to e_3. Therefore in the experimental geometry used, which provides a measurement of $(e_1 - e_3)$, quadrupolar cluster contributions to splay and bend flexoelectricity may largely counteract each other. Accordingly, work will be undertaken in the future to evaluate the sum of the flexoelectric coefficients $(e_1 + e_3)$ for C6ban in order to address this issue. Additionally it is worth noting that anti-parallel dipolar ordering may be present at both the intra- and intercluster level. This would tend to reduce dipolar contributions to flexoelectricity, but may not strongly influence quadrupolar contributions.

In summary, we have characterized the nematic phase formed by the bent-core compound C6ban. The influence of the bent molecular shape on the nematic phase is evident through the unusually low ratio of k_3 to k_1 that is found, expressing that bend distortions of the director are relatively lower in energy compared to those involving splay. When doped with a chiral agent, the resultant phase is significantly more twisted than with standard materials (equivalent to an 85% rise in effective HTP of the dopant). The molecular shape anisotropy is also seen to have a pronounced effect on the difference in flexoelectric coefficients where, although we do not find evidence of “giant” flexoelectricity, the $(e_1 - e_3)$ values reported are large in magnitude compared to conventional calamitic materials with similar dipoles. We note that the final two points may both be linked to the biaxial molecular structure of the bent-core compounds. A cholesteric phase is known to be locally biaxial [32], related to a difference in molecular fluctuations along and perpendicular to the helix axis. Meanwhile, a flexoelectric distortion is associated with a local biaxial order of the director. Hence, it seems possible that a material with a pronounced biaxial molecular structure may adopt distortions where there is a local biaxial order more easily than molecules of largely uniaxial shape. Therefore while there are no indications of a bulk biaxial phase, high flexoelectric coefficients and increased twist in a cholesteric phase are found.

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