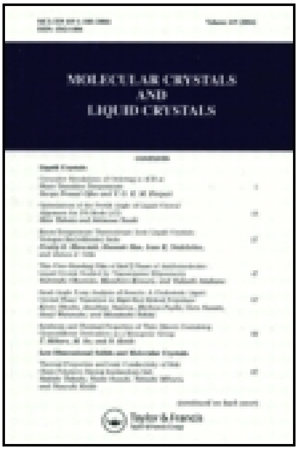

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Cold Neutron Scattering Investigation of Molecular Orientational Motion in Smectic A Phase of 4-Cyano- 4′-N-Alkylbiphenyl Liquid Crystal

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COLD NEUTRON SCATTERING INVESTIGATION OF MOLECULAR ORIENTATIONAL MOTION IN SMECTIC A PHASE OF 4-CYANO- 4'-N-ALKYLBIPHENYL LIQUID CRYSTAL

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Abstract The measured cold neutron scattering spectra obtained in the S_A phase of 4-cyano-4-alkylbiphenyl sample are investigated with emphasis: a) on the molecular uniaxial, within two potential barriers angular confined, stochastic reorientational motion, b) on the long molecular axis orientational fluctuations between two potential barriers as well as uniaxial simple rotational diffusion, and c) on the correlations of orientation between adjacent molecules due to Faber, with no localized motion implied.

It is shown that, for low Q values, where Faber approach could be utilized, the spectra are well represented by his continuum theory of molecular disorder, although the first model above, in a modified form, can not be completely ruled out.

INTRODUCTION

The single molecular dynamics in nematic and various smectic phases of liquid crystalline samples as determined by incoherent quasi-elastic neutron scattering results[1-3], has been found to be successfully interpreted in terms of translation diffusion of molecular center of mass which is, in the case of smectics in the direction parallel to the layer normal to some extent hindered, and uniaxial rotational diffusion about molecular long axes, which freezes up in the low temperature (higher symmetry) phases in such a way, that molecules either do not reorient at all or perform only occasional orientational jumps. The motion which can occur on the same time scale as the molecular rotational motion is the fluctuation of the long molecular axes[4] for which a dynamical model has been proposed recently[5].

In their comprehensive comparative study by the incoherent neutron scattering method of molecular motions in S_A, S_B and S_E phases on aligned samples of isobutyl 4 (4' phenylbenzylideneamino) cinnamate, Richardson et al.[1], have concluded that the convenient picture for the description of the localized motion in smectic phases would be given in terms of the model of a molecule confined in a cage of its neighbours. In S_A phase this cage was found to be loose enough to permit uniaxial rotational diffusion, fluctuations of the long molecular axes and a localized (perpendicular to molecular planes) translational motion, all on the same time scale. If was further concluded that with decreasing temperature the cage progressively diminishes, with the result, that axis fluctuations and uniaxial rotation become more restricted but also slower.

Thus while the single molecular dynamics in liquid crystals, also on account of the incoherent quasi elastic neutron scattering studies, seems to be understood at least in its rudimental appearances, very little is konwn about the collective motions on the molecular level. This questions seem to have received very little attention in the past, yet the neutron scattering method is most convenient method for the purpose. This is immediately apparent when vieving the only experimental study of the collective molecular motion[6], which undoubtedly due to the complexities involved, has not been followed by an adequate theoretical approach on the molecular level.

The concept of the cage let one to speculate on the way of its formation and consequently of its (dynamic) structure and its influence on the motion of a given molecule of interest. If the previous work[7] is understood in terms of potential barriers as being represented by the cage walls, then the slow reorientation of potential barriers of a sort discussed by Janik et al.[3] would be intimately connected with the life expectancy of such a cage formation and it would be neccessarily associated with single molecular characteristic long axis angular (and likely also uniaxial reorientational) motion on account of long range molecular orientational forces of liquid crystalline phases.

All present approaches to the investigation of molecular dynamics in liquid crystals, from the incoherent neutron scattering point of view, are based on particular models in mind which all have in common the assumption of single isolated molecules and the liquid crystalline properties are introduced via the order parameters which express the distinctive feature of these phases namely that the molecules are spontaneously aligned. Since molecules interact among themselves, and most evident form of this interaction is the existence of orientational order parameter, the question, how these correlations of orientation between adjacent molecules reveal themselves in the spectra of incoherently scattered neutrons is to be examined. This effect can be however, most conveniently discussed from the point of view of Faber continuum theory of disorder in nematic phase[8-11]. The essential feature of Faber approach is the assumption that in the absence of any type of pertubation the director field and correspondingly the molecular axes are everywhere parallel to, say, z axis such that $S_2=1$. Thermally excited set of modes then cause periodic distortion of this initially uniform director field so that the local axis of uniaxial (in nematic and S_A phases) symmetry - the director - is tilted away from the z axis and varies within the sample. The degree of alignment is then given, for rigid molecules, by order parameter $S_2=\langle P_2(\cos\theta)\rangle$, where θ indicates the Euler angle of long molecular axis with respect to the director. A distinctive feature of Faber's theory is the fact that the correlations between different molecules are included from the start as any tilt of the particular molecule under investigation must have some effect on its surroundings and arguments are presented showing that these correlation of orientation are varying as 1/R.

The purpose of this paper is to investigate the effect of the correlations of orientation between adjacent molecules as predicted by Faber continuum theory of disorder, on the incoherent neutron scattering spectra and to test this predictions, against conventional isolated molecular approach utilising two different dynamical models proposed recently, i.e., the model of stochastic around long molecular axis, angular confined librational molecular motion[7], and the model of rapid molecular long axis orientational fluctuations between two perfectly reflecting barriers[5].

THEORETICAL OUTLINE

Due to the steric reason, it was argued[7, 12] the proton within assumingly rigid molecule could be constrained to move along the long molecular axis on an arc of a circle of length $2d\emptyset_o$, where d is radius of the circle and $2\emptyset_o$ is the angular span between the two potential barriers which are the cause of the constrained molecular motion. Based upon the method of onedimensional random flight problem between two barriers and including the translational motion of molecular center of mass, the following incoherent scattering function (for one proton only and averaged over all orientation of the scattering vector $\grave{Q}$) has been derived:

$$S\,(Q,\omega) = \sum_{l,m} j_l^2\,(Qr)\,Y_{lm}\,(\Theta,\phi)\,Y^{*}_{lm}\,(\Theta,\phi)\,\cdot$$
$$\cdot\left\{\frac{1-\cos\phi_o}{m\,\phi_o^2}\;\frac{Q^2D}{Q^4D^2+\omega^2} + 2\sum_{r=1}^{\infty}\right.$$
$$\left.\frac{m^2\left[1-(-1)^r\cos m\phi_o\right]}{\phi_o^2\;\left(\left(\frac{\pi r}{\phi_o}\right)^2-m^2\right)}\cdot\frac{Q^2D+\delta_r}{(Q^2D+\delta_r)^2+\omega^2}\right\} \qquad (1)$$

where,

$$\delta_r = \frac{\Pi^2 r^2 R}{\phi_0^2} \qquad (2)$$

and D is (an average) translational molecular diffusion constant, j_l is the spherical Bessel function of order l, Y_{lm} (Θ,Φ) is the spherical harmonics as defined in the molecular principal axes reference frame, $\vec{r}$ is the position vector of a proton as measured from the molecular center of mass and ℓ is an integer. R is the diffussion constant associated with the localized reorientational molecular motion. In derivation of the eq (1) above it was assumed the perfect molecular order, and we did not distinguish in the anisotropy of the translational molecular diffusion.

The solution of the problem of a symmetric one dimensional random walk of a particle between two perfectly reflecting barriers turned out to be a convenient way for the description of molecular orientational fluctuations and the incoherent neutron scattering function for this type of molecular motion incorporating also the translational motion of molecular center of mass and uniaxial rotational motion along the long axis reads[5],

$$S(Q,\omega) = \sum_{\ell} j_{\ell}^{2}(Qr) \sum_{m} \sum_{m'} \Bigg\{ F_1(\ell,\ell,m,m') \cdot \frac{Q^2 D + D_1 m'^2}{(Q^2 D + D_1 m'^2)^2 + \omega^2} + \sum_{r=1} F_2(\ell,\ell,m,m';r) \cdot \frac{Q^2 D + D_1 m'^2 + \delta_r}{(Q^2 D + D_1 m'^2 + \delta_r)^2 + \omega^2} \Bigg\} Y^{*}_{\ell m}(\Theta,\phi)\, Y_{\ell m}(\Theta,\phi) \qquad (3)$$

Here, D_1 represents the uniaxial molecular rotational diffusion constant, and the functions F_1 and F_2 are defined by

$$F_1(\ell,\ell,m,m') = \left[\frac{1}{\phi_0} \int_0^{\phi_0} d^{\ell}_{mm'}\left(\phi - \frac{\phi_0}{2}\right) d\phi \right]^2$$

$$F_2(\ell,\ell,m,m';r) = \left[\frac{\sqrt{2}}{\phi_0} \int_0^{\phi_0} d^{\ell}_{mm'}\left(\phi - \frac{\phi_0}{2}\right) \cos\left(\frac{\pi r \phi}{\phi_0}\right) d\phi \right]^2 \qquad (4)$$

and are for the case l=1,2 evaluated in the appendix of[5]. In the expressions above like in the previous one a perfect molecular order is assumed. $d^{1}_{mm'}$ are the rotational matrices as defined in Rose[13].

In Faber's alternative theory[8-11] to mean field theories, director fluctuations play the dominant role. It is assumed that but for these fluctuations the molecular alignment would be perfect, i.e. the order parameters of all orders would be identically equal to 1. Faber thus equates the molecular orientation with the director field described in terms of a unit vector $\vec{N}$ ($\vec{R}$) and attributes the director fluctuations to a set of thermally excited modes involving periodic distortions of the initially uniform director field. It is rather clear that Faber approach does not deal with the type of motion as described by eq. (3), which describes the molecular long axis fluctuation arround its average direction but referes to this average molecular direction instead. But then, depending on the relative strenght of the correlated orientational interactions between the molecules the above type of long axis fluctuations might, after all, not be possible at all.

Following the method of ref.[8], the calculated incoherent neutron scattering function, for the case when the director fluctuations are viewed as the superposition of a spectrums of excited periodic distortion modes (i.e. the order parameter is then defined as $S_2=\langle P_2$ $(\cos \psi)\rangle$ where ψ is the local angle of tilt of the director associated with a mode of wavevector $\vec{q}$) and the molecule is subjected to the translational diffusion motion, averaged over all orientations of the scattering vector, for the case of l=0,1,2,3, reads,

$$S(Q,\omega) = \sum_{l} (2\ell+1) j_l^2(Qr) P_l^2(\cos\,\delta) h_\ell(D, S_2, \tau_c) \tag{5}$$

Denoting,

$$g(p) = \frac{Q^2 D + p\frac{1}{3\tau_c}}{\left(Q^2\;D + p\frac{1}{3\tau_c}\right)^2 + \omega^2} \tag{6}$$

the functions h_1 $(D,\ S_2,\ \tau_c)$ are given by,

$$h_0 = g\;(p = 0)$$

$$h_1 = \left(1-\frac{5}{4}X + \frac{1}{4}X^2\right)\sqrt{S_2}\;g(p=0) + \left(2X-\frac{9}{4}X^2\right)\cdot$$
$$\cdot\sqrt{S_2}\;g(p=1) + \frac{1}{2}X^2\sqrt{S_2}\;g(p=2)$$

$$h_2 = 4S_2^2\;g(p=0) + (12X+3X^2)\;S_2^2\;g(p=1) +$$
$$+ \left(15X^2 - \frac{23}{2}X^3\right) S^2\;g(p=2) \tag{7}$$

where

$$X = -\frac{2}{3}\,\ell n\;S_2 \tag{8}$$

and S_2 denotes the usual order parameter defined in terms of the molecular (or preferentially local director) local angle of tilt. The functions

h_ℓ are written in the isotropic approximation assuming all elastic constants to be equal. The functions h_l are associated with the time dependent correlation function of the form

$$< Y_{\ell m}(\Theta(t)\ \phi(t))\ Y^{*}_{\ell m}(\Theta(0)\phi(0))>$$

and $(0, \phi)$ gives the instantaneous molecular long axis orientation with respect to the space z-axis as caused by the excitation of a single mode which twists the long axis about a fixed axis in the (x, y) plane[8]. The angle δ in eq.(5) is an angle between the proton position vector as measured from the molecular center of mass and the z-principal axis. The parameter τ_c is given by

$$\tau_c = \frac{\eta}{Kq_c^2} \tag{9}$$

where K is and (average) elastic constant, η the viscosity and q_c is the mode spectrum cut off on a spherical surface in q-space of radius q_c[8]. In deriving the expressions h_l we have utilized for the time dependent part of the distortion mode correlation function $\xi(t)$ as defined by ref.8.

$$\sum_{\text{all modes}} <\psi(t)\ \psi(0)> = \xi(t)\ X \tag{10}$$

the following form

$$\xi(t) = \frac{1}{q_c}\int_0^{q_c} e^{-\frac{t}{\tau_c q_c^2}q^2}\, dq \rightarrow e^{-\frac{t}{3\tau_c}}$$

what ensures the correct ξ behaviour for $t \rightarrow 0$.

It is through the combination of X terms in the expression for h_1, that the correlation of molecular orientations enter the above incoherent scattering law[8].

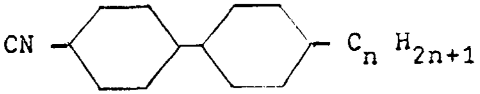
EXPERIMENT AND DATA TREATMENT

The molecular dynamical behaviour in liquid crystals become more pronounced with elevated temperatures but also depend on the nature of particular phase of the sample under investigation. For the reason of simplicity we decided to perform the scattering experiment on an unoriented sample of eutectic smectic biphenyls, 4-cyano - - 4'-n-alkylbiphenyls, denoted S-2, and provided by BDH-liquid crystals England. The phase transitions are at

$$K \xleftrightarrow{-1^{\circ}C} S_A \xleftrightarrow{48^{\circ}C} N \xleftrightarrow{49^{\circ}C} I$$

and the sample is therefore room temperature S_A liquid crystal.

The homologues in the cyanobiphenyl series in the present study are



for n=9,12 and the S_A phase at 22°C and amorphous phase at -16°C were studied, respectively.

The experiment was performed at the time-of-flight spectrometer for cold neutrons at the R2 reactor in Studsvik, Sweden. The energy of the incident neutrons was 3.34 meV corresponding to a neutron wave length of 4.95 Å. The detectors were placed at different scattering angles ranging from 17° to 83° which is equivalent to a covered wave vector transfer region of Q=0.3 $Å^{-1}$ to Q=1.6 $Å^{-1}$. The energy and a resolution at zero energy transfer were nearly constant and equal to 0.20 meV and Δ Q=0.1 $Å^{-1}$, respectively.

The measurement spectra of scattered neutrons were corrected for trivial experimental effects by standard methods and normalized to absolute cross section units via vanadium calibration measurements. The contribution from multiple scattering was also calculated.

Models describing the molecular motions in liquid crystals usually yield a scattering function for the quasi-elastic scattering only. Thus all scattering of inelastic nature should be removed from the measured spectra in making a comparison between predictions and experimental results meaningful. For this purpose the measured S (Q, E) in the amorphous phase were used. A smooth interpolation of the inelastic part in under the elastic peak was performed at all scattering angles. The inelastic S (Q, E) thus obtained were then substracted from the corresponding ones determined in the smectic A phase. The elastic peak of the amorphous phase spectra were also used to determine the resolution function.

RESULTS AND DISCUSSION

The choice of the sample was dictated by the desire to keep the complications resulting from the molecular rotational motion to a minimum. It is reasonable to expect that the different length alcyl chains in the eutectic smectic A sample would be hindered in particular so since the temperature is rather low. Assuming the uniaxial hindered molecular rotational motion we have atempted to fit the experimental spectra with the scattering law as given by eq. (1). The structural data were calculated from known data for bond angles and bond distances assuming the two benzene rings lying in the same plane. Very good fit in the range of $Q = 0{,}375$ Å^{-1} to $Q = 1.210$ Å^{-1} investigated is obtained for the values of $\emptyset_{o}$ □ 20^{o} and $R=10^{11}$s, taking D □ $5\text{x}10^{-8}$ cm^{2}/s a fixed value for the translational diffusion constant, which unfortunately is not known. Although the scattering law in question is derived under the assumption of perfect molecular order an estimate of the order parameter influence on the quality of fittings, and consequently on the value of obtained, shows almost negligible change. For the order parameter the value $S = 0{,}85$ was taken as follows from mean field theory of McMillan[14].

Leadbetter and Norris[15] were able with X-ray scattering investigation of nematic and smectic A phase of EABAC to infere that in both phases the molecular long axis fluctuations are completely dominated by collective fluctuations involving the coupled motions of several molecules for which a lower limit of the order of ten molecules has been proposed. The orientational

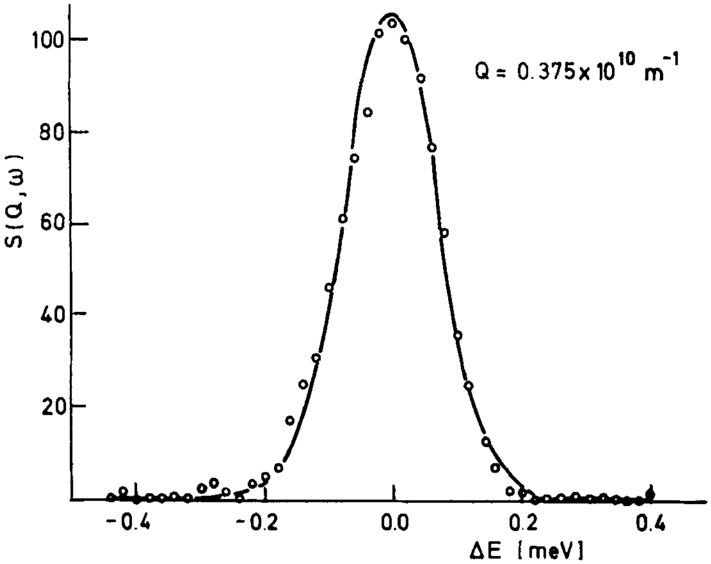


FIGURE 1. The measured spectra of incoherently scattered neutrons on an unoriented S_A phase of S-2 eutectic liquid crystal at T □ 22°C are compared to the model of uniaxial, angular confined, molecular motion within two potential barriers and translational diffusion of molecular center of mass. The value of parameters are: D □ 5 x 10^{-12} m^2/s and $\tau = 1/R = 10^{11} s$.

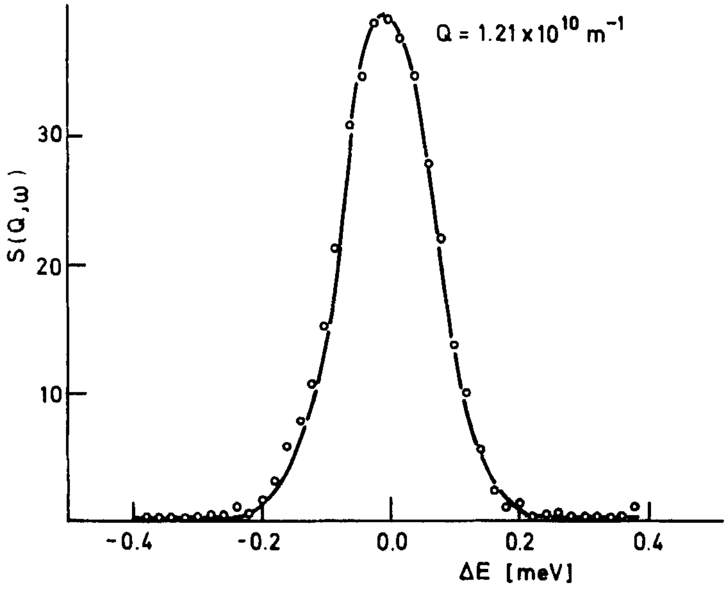


FIGURE 2. The measured spectra of incoherently scattered neutrons on an unoriented S_A phase of S-2 eutectic liquid crystal at T □ 22°C are compared to the model of uniaxial, angular confined, molecular motion within two potential barriers and translational diffusion of molecular center of mass. The value of parameters are: $D = 5 \times 10^{-12}$ m²/s and $\tau = \frac{1}{2}$ □ 10^{11} s.

distribution function of the local director for clusters of these molecules (relative to the average directors), which form is to a very good approximation described by a single particle mean field theory of Maier-Saupe type, led authors to conclude that the motions of an individual molecules against a cage of neighbours represents only a minor contribution to the total distribution function. The same conclusions turned out to be valid for nematic and smectic C phase of OOAB and in nematic phase of HOAB sample.

We have attempted to test the proposed cage concept by trying to gain information on the short range dynamical behaviour of an individual molecule within the cage, for which the dynamical model of long molecular axis orientational fluctuations[5] as given by the eq.(3) seems most suitable. This would be indeed likely if only the values of uniaxial rotational diffusion constant and at least an estimate for the reorientational time of the orientational fluctuation motion would be, independently, available. As an example we had taken $D_1 = 10^{11}$ s, $R = 10^{11}$ s^{-1} (corresponding to the value obtained with the model of the angular confined uniaxial librational motion above), then for $D \square 10^{-7}$ cm²/s the apex angle, $\emptyset_o$, of the molecular long axis would be of the order 20° to 60° which would seem to support the cage concept. Unfortunately, there are other combinations od parameters yielding similar range of the apex angle and for this reason no conclusion is possible to draw in the above case. Is should be mentioned that in the approach described, the cage concept is tested only implicitely, assuming that the cage dynamics would be invisible on our time scale.

It is possible to investigate the direct cage contribution to te spectra, by utilizing Faber's continuum theory of disorder, on the assumption that the effect of an individual molecule to the measurements is primarily reflected in the traslational diffusion motion. The drawback in this approach is however in the fact that it is possible to analyze low Q spectra only as the analytical expression for the roots (which are functions of time dependent parameter $\xi(t)$, eq.(10)) of the algebraic equation with which the general solution of coupled linear differential equations for time correlation functions of spherical harmonics is expressed, can be given for the up and including l □ 3 term. Yet, as shown on Figure 3, the calculated scattering law is rather sensitive to the value of parameter τ_c. Figure 3 shows the normalized resolution broadened incoherent neutron scattering function calculated from eq. (5) for the values of D □ 5×10^{-8} cm²/s, Q = 1 Å^{-1}, Qr = 2.3 and the order parameters S_2 = 0.6, for various values of the parameter τ_c. In general it was found that for the values of parameter $\tau_c < 10^{-11}$ s the calculated broadened incoherent scattering law shows similar but slightly less distinct behaviour with the increasing value of S_2.

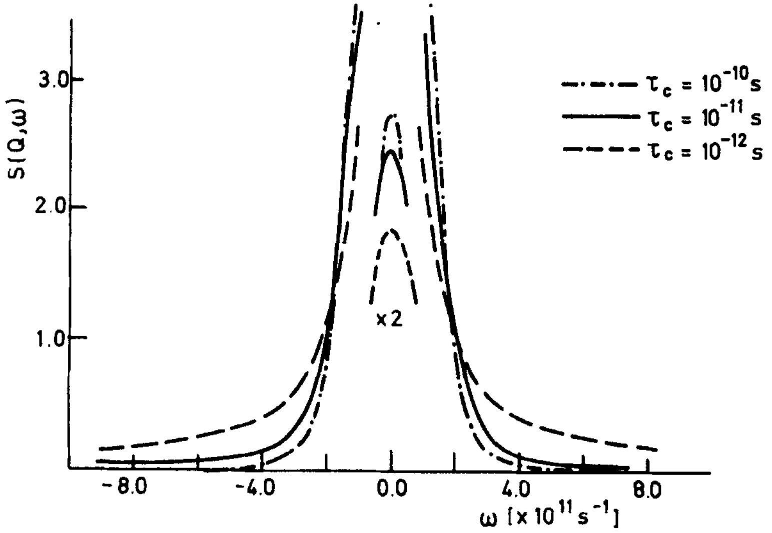


FIGURE 3. Orientationaly averaged normalized incoherent resolution broadened scattering function assuming the translational diffusion of molecular center of mass and the contribution of correlations between neighbouring molecules as introduced by Faber[8]. The uniaxial molecular rotational motion was taken to average out. The values of parameters are: $D = 5\text{x}10^{-11}$ m²/s, Qr = 2.3, τ_c is the parameter associated with the spectrum of distortion modes cut-off.

The best fit to the two low Q spectra is shown on Figure 4 for Q = 0.375 Å^{-1} for the following set of parameters S_2 □ 0.8, τ_c □ 10^{-11} s, for

$D = 5\times10^{-8}$ cm²/s. The value of τ_c which is associated with the spectrum of distortion modes cut--off is to a large degree, as follows from Faber's theory, fixed. The Figure 3 shows great sensitivity of the resolution broadened scattering law on the value of this parameters and the fact that τ_c as obtained in the fitting of measured spectra is within the order of magnitude equal to the expected value, seem to corroborate the cage concept advanced in ref.[15]. In fact, generalizing the reasoning which had led to the derivation of eq. (3) in such a way to incorporate the quantities X, which are associated with long range molecular orientational correlations (and are function of S_2, eq. (8)) one obtaines the possibility to investigate simultaneously also the intracage motion of an individual molecule. It is noted that the effect of molecular long range orientational correlation contribution on incoherent neutron scattering spectra has not been investigation at all and this work represents first such an attempt in this direction.

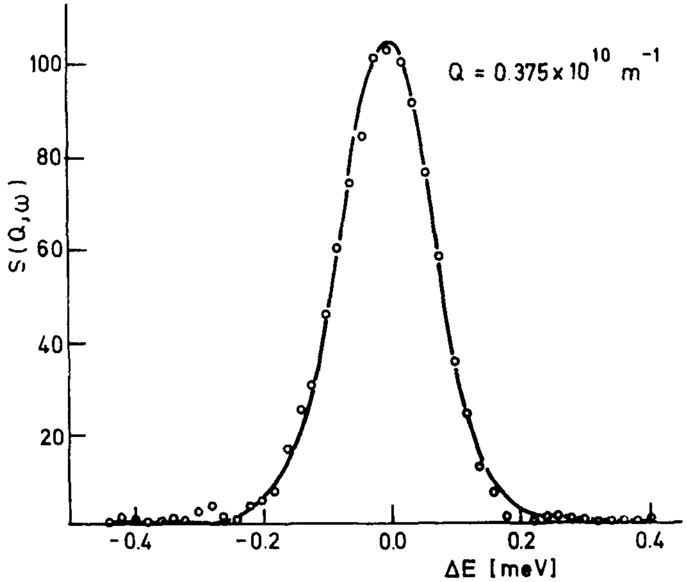


FIGURE 4. The incoherent neutron scattering spectrum measured on an unoriented S_A phase of S-2 eutectic liquid crystal at T = 22°C is compared to the calculated scattering law assuming simple translational diffusion of molecular center of mass and correlations between neighbouring molecules due to Faber. No uniaxial rotational motion is included. The values of parameters are: D □ 5×10^{-12} m²/s, τ_c □ 10^{-11} s, Q □ 0.37 Å^{-1}.

The above results of the model fittings could be therefore interpreted in the following way: in the smectic A phase of S-2 eutectic liquid crystal the molecules form associations owing to the orientational correlations between adjacent molecules what in consequence results in the fact, that the intracage rapid uniaxial rotational motion of an individual molecule, as indicated by the angular separation of potential barriers, is strongly hindered. Occasional jumps of the molecules over the potential barriers induce the decay of the present cage formation and a new one is formed. The time interval of the reorientation of the potential barriers would be an indication of the life span of a cage and it seems that it is too slow to be measured by neutron scattering method.

The models of confined librational molecular motion which could, in the context above, represent intracage motion are not neccessarily in contradiction with the conclusion drawn from ^{14}N NQR[16]. The fact that in S_A phases the measured asymmetry parameter, η , of electric field gradient tensor at the site of nucleus in question is in general zero, would be in agreement with the dynamical model, providing one is invoking Janik et al.[3] assumption of rotating potential barriers which would be a slow process on neutron time scale but could be possibly detected by NMR. The fast, rapid within the barriers (intracage) localized diffusive molecular reorientational motion is expected to be for NMR invisible. However, if it happens that the potential barriers reorient themselves slowly also on NMR frequency scale or on the average not at all, than a case of uniaxial localized reorientational motion with biasing would be taking place. Of course, the asymmetry parameter would than be expected to be nonzero.

CONCLUSION

The measured incoherent scattering spectra at T □ $22^{\circ}C$ in S_A phase of an unoriented sample of eutectic S-2 liquid crystal were, for low Q values, interpreted on account of translational diffusion of molecular center of mass and contribution of correlations between neighbouring molecules due to Faber's continuum theory of molecular disorder. The intracage molecular motion was described by two different models of localized molecular dynamics (over all Q range).

On the basis of these approaches the picture of molecular dynamics was proposed for this phase, according to which the strong intermolecular orientational interactions are responsible for the rapid, between two potential barriers angular confined, uniaxial reorientational motion of a single molecule with the net result that molecular associations are formed, which are, due to orientational correlations, of a dynamic nature. The average time interval between reorientation of the hindering potential barriers could serve as an indication of a life span of such a cage formation.

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