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# Pretransitional effects at the isotropic–mesomorphic phase transitions in the TBAA series

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Thermal microscopy (TM), differential scanning calorimetry (DSC) and dilatometric  $\rho(t)$  experiments were carried out to characterize the phases and to study the nature of mesomorphic transitions in two higher homologues of terephthalylidene-bis-*p-n*-alkylanilines, TBnAs, namely terephthalylidene-bis-*p-n*-tetradecylaniline, TB14A and terephthalylidene-bis-*p*-hexadecylaniline, TB16A. Results infer the first order nature of the isotropic–smectic C (I–S<sub>C</sub>) and smectic C–smectic I (S<sub>C</sub>–S<sub>I</sub>) transitions, while the smectic I–smectic F (S<sub>I</sub>–S<sub>F</sub>) transition is found to be second order. Pretransitional effects are estimated through  $\alpha_{\text{eff}}$  in the vicinity of mesomorphic fluctuation dominated non-linear regions (FDNLR), of the isotropic to mesomorphic transitions in TBnA homologous compounds and the relative strength of growth of FDNLR is discussed at the S<sub>I</sub>N, S<sub>I</sub>S<sub>A</sub> and S<sub>I</sub>S<sub>C</sub> phase transitions.

## 1. Introduction

Research in the field of liquid crystals, particularly after the 1960s, has focused considerable effort on phase transitions involving one- and two-dimensional ordering [1]. In this regard the TBnA series, namely the terephthalylidene-bis-alkylanilines, is particularly interesting in that it presents an unusually broad polymorphism exhibiting nematic, smectic A, C, I, F, B, crystal G and H phases. Thus since the first observation of the TBnA series [2, 3] there have been many studies of its phase behaviour [4–15]. Here the authors present experimental results for the phase transitions and pretransitional behaviour of two new TBnA homologues exhibiting the isotropic–smectic C–smectic I–smectic F phase sequence.

The infinite rotational symmetry of the high temperature isotropic liquid phase disappears as the temperature is decreased via transitions to the structurally ordered low temperature mesomorphic phases. The isotropic to smectic C (IS<sub>C</sub>) transition, which involves the growth of a biaxial tilted smectic phase with C<sub>2</sub> symmetry, is first order. The smectic C to smectic I (S<sub>C</sub>S<sub>I</sub>) transition, involving the growth in extent of the short range quasi-hexagonal positional order of the smectic C phase represents no symmetry change and so can only be first order or evolve continuously without a transition. The S<sub>C</sub>S<sub>F</sub> transition, which involves similar structural change similar to that of the S<sub>C</sub>S<sub>I</sub> transition, is found to

be first order [12]. The smectic I to smectic F transition is accompanied by symmetry change, the change of direction of the tilt relative to the hexagonal lattice but neither an additional long range orientational nor a short range positional ordering, resulting [8, 10] in a possible second order nature.

Phase transition studies by different methods provide valuable information about the pretransitional effects and the relevant critical properties. Our previous systematic observations in the homologues of *N*(*p-n*-alkoxybenzylidene) *p-n*-alkylaniline, (*nO.m*) compounds reveal (i) a first order [16] isotropic to smectic F transition involving the growth of bond-orientational order; (ii) interdigitated (frustrated) smectic phases [17] (as mediated through sterically enhanced orientational disorder) appearing with increasing flexible end chain length; (iii) direct transitions from the isotropic liquid to tilted phases obtained [17, 18] by tuning the length of the flexible end chain  $m > 12$ .

Owing to the interest in the tilted smectic phases (motivated by the potential electro-optic applications of the chiral smectic C) we studied the higher homologues of the TBnA series with the expectation of an isotropic to direct tilted smectic phase transition within an experimentally feasible thermal range. This motivated us to synthesize the TBnA compounds with  $n = 14$  and 16 (higher homologues) for a possible direct transition from isotropic liquid to smectic I phase by quenching the high temperature smectic A and smectic C phases (which have been found in the lower homologues of these

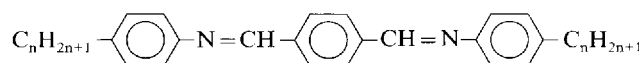
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compounds). Further, as a part of our systematic study of the TBnA homologues series, we report (i) the synthesis of the compound, terephthalylidene-bis-*p-n*-hexadecyl aniline, TB16A and its characterization by thermal microscopy (TM) and differential scanning calorimetry (DSC); (ii) phase transition studies of TB16A by dilatometry (along with the reported [19] DSC of TB14A) of the isotropic to smectic C ( $I-S_C$ ), smectic C to smectic I ( $S_C-S_I$ ) and smectic I to smectic F ( $S_I-S_F$ ) transitions. This work is directed toward understanding their order and

to study the relative strength of pretransitional effects accompanying the transitions from the isotropic liquid.

## 2. Experimental

The TB14A and 16A compounds were synthesized from their relevant terephthalaldehydes and alkyl anilines following the procedure reported [6] earlier. Thermal microscopy was carried out using a Hertel Reuss super pan polarizing microscope equipped with a heating stage for the textural observations. Dilatometry was carried out with a U-shaped pycnometer kept in a controlled temperature chamber. The temperature was accurate to  $\pm 0.1^\circ\text{C}$  in the thermal microscopy and  $\pm 0.2^\circ\text{C}$  in the density measurements. The absolute error in measuring the density was  $\pm 0.1 \text{ kg m}^{-3}$ . Differential scanning calorimetry (DSC) was carried out for both the compounds (see figure 1) using a Perkin-Elmer 7 system. The general molecular formula of the TBnA series of compounds is given below and the phase transition temperatures for the  $n=14$  and 16 compounds observed through microscopy and DSC are given in table 1.



## 3. Results and discussion

### 3.1. Phase transitions in TB14A and TB16A

#### 3.1.1. Thermal microscopic studies

The characteristic [20] thermal microscopic textures observed for the compounds of TB14A and TB16A are

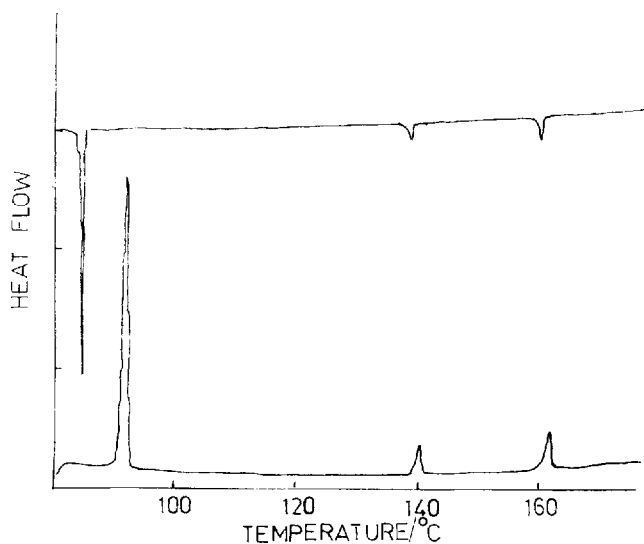


Figure 1. Differential scanning calorimetry trace for TB16A.

Table 1. Phase transition temperatures from thermal microscopy (TM), dilatometry and DSC along with the enthalpy values and pressure dependence of transition temperatures for different transitions.

Compound	Method	$I-S_C$	$S_C-S_I$	$S_I-S_F$	$S_I-Cr$
TBAA-14	T.M.	170.0	144	120.1	90.0
	DSC				
	Heating	169.54	144.01	—	85.84
	$\Delta H$	12374	7165	—	65403
	Cooling	168.35	142.95	—	78.89
	$\Delta H$	11962	6427	—	54126
			170.0	144.5	120.0
	Density	170.0	144	120.2	—
	$dT/dp$	33.03	51		
TBAA-16	T.M.	160.0	138.8	133.8	89.0
	DSC				
	Heating	161.73	140.41	—	91.91
	$\Delta H$	8469	6608	—	62882
	Cooling	160.24	138.96	—	84.9
	$\Delta H$	8440	6176	—	52075
	Density	160.0	138.8	133.8	—
	$dT/dp$	29.99	41.4		

Transition temperatures are in  $^\circ\text{C}$  and pressure dependence in  $\text{K kbar}^{-1}$ .  $\Delta H$  in  $\text{J mol}^{-1}$ .

schleiren in smectic C, broken focal conic in smectic I and arc-focal-conic in smectic F phases. Further, the TB16A phases are found to be miscible with the reported [19] phases of TBAA-14 in their entire thermal range to confirm the phases in TB16A to be CIF variant.

However, neither the expected quenching of smectic phases ( $S_A$  or  $S_C$ ) nor the direct appearance of a bond orientational smectic I phase has been observed as a result of increase ( $m=14$  or  $16$ ) of the flexible end chain as expected in these compounds. This may be occurring due to the presence of the three aromatic rings in the rigid core part of the TBnA molecule (in comparison with the two rings [16] of a  $nO.m$  molecule). Hence, it is noticed that the ratio of the flexible chain length (for the present compound with  $n=14$  or  $16$  in the TBnA series) to the rigid core length is not sufficient to quench the smectic A or C phases to exhibit a bond orientational smectic F or I phase directly from the isotropic liquid (as observed [16] for  $m=14$  in the  $nO.m$  series). This suggests that a further increase of chain length  $>16$  may quench the smectic C phase to result in a direct bond-orientationally ordered tilted smectic phase from the isotropic liquid.

The temperature variation of density,  $\rho$  and the volume expansion coefficient,  $\alpha$  for the compounds TB14A and 16A are shown in figures 2 and 3, respectively. The estimated contribution of molar volume per methylene ( $-\text{CH}_2-$ ) group increment (from the observed molar volumes) in the isotropic liquid at  $T_C+5^\circ\text{C}$  is found to be in the range of  $14$  to  $16.5 \times 10^{-6} \text{ m}^3 \text{ mol}^{-1}$  in agreement with the reported

values [8, 10] for other compounds of the TBnA and  $nO.m$  series [16] of compounds. The rate of increase of density observed in the equilibrium isotropic liquid phase ( $[\text{d}\rho/\text{d}T]=10.2 \times 10^{-4} \text{ g cm}^{-3} \text{ }^\circ\text{C}^{-1}$  for TB14A and  $9.29 \times 10^{-4} \text{ g cm}^{-3} \text{ }^\circ\text{C}^{-1}$  for TB16A) is found to agree with the reported [8, 10, 16] values of other LC compounds.

### 3.1.2. Isotropic to smectic C ( $IS_C$ ) transition

The  $IS_C$  transition is accompanied by a large density jump ( $[\Delta\rho/\rho] \times 100 = 1.2$  in TB14A and  $0.75$  in TB16A) and a thermal expansion coefficient maxima ( $\alpha = 23.87 \times 10^{-3} \text{ }^\circ\text{C}^{-1}$  in TB14A and  $11.91 \times 10^{-3} \text{ }^\circ\text{C}^{-1}$  in TB16A) to infer the first order nature of the transition. The slope of the density plot (calculated away from the phase transition, i.e. in the thermally stable linear region) is found to attain a higher value with the growth of the relatively densely packed smectic C phase. The observed density jump (see table 2) is found to be comparable and falls on the higher side of the density jumps reported [21] across the isotropic-smectic A transition in the TBnA series and other compounds. However, it is less than the density jump (2.14 per cent) observed [16] across the isotropic to smectic F transition (in compound 10O.14 of the  $nO.m$  series), which involves the largest change of structural order. The observed density jumps across the  $IS_C$  (in TB14A and TB16A) are found to be higher than those across  $IS_A$  or  $IS_F$  transitions (except for TB10A) and lower than that across  $IS_F$  (in 10O.14) phase transitions. It is due to the growth of characteristic intermediate smectic C order (in between structural

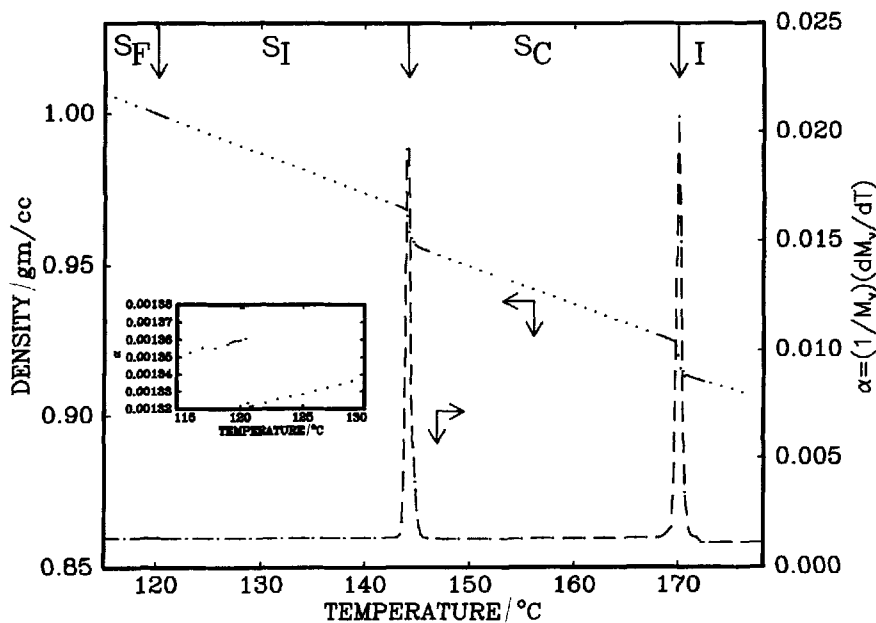


Figure 2. Temperature variation of density  $\rho$  and volume expansion coefficient  $\alpha$  for the compound TB14A.

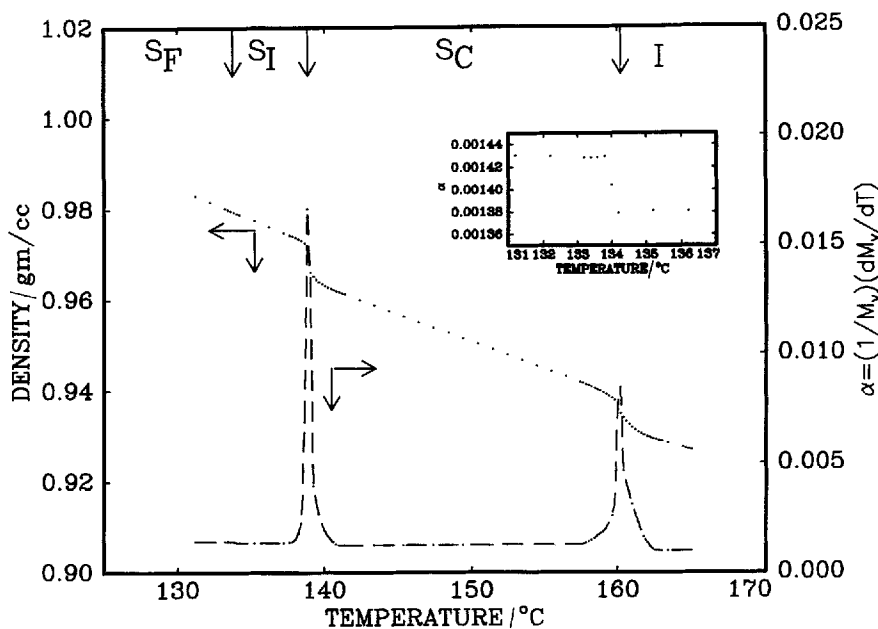


Figure 3. Temperature variation of density  $\rho$  and volume expansion coefficient  $\alpha$  for the compound TB16A.

orders that grow across  $IS_A$  and  $IS_F$  transitions) from the isotropic liquid. The differing density jumps across  $IS_I$ ,  $IS_A$ ,  $IS_C$  and  $IS_F$  transitions with the highest value at the  $IS_F$  transition reflect upon the impact of short range positional order across the  $IS_F$  transition. The estimated pressure dependence (namely  $dT/dp$  in table 2) for  $IS_C$  transition temperatures (from the extrapolated density values onto the  $T_{IS_C}$  and the DSC enthalpy ( $\Delta H$ ) values), using the Clausius–Clapeyron equation in TB14A and TB16A are lower compared to the values reported (see table 2) across the  $IS_A$  transition in the TB9A and 10A compounds, while it was found [16] to be strikingly incomparable with that across the  $IS_F$  transition. Thus the observations suggest the comparability of equilibrium thermodynamic situation of the  $IS_C$  transition to the  $IS_A$  transition (as both of them represent model systems for melting of one-dimensional crystals), rather than the case of melting of the quasi-two-dimensional crystal at the  $IS_F$  transition. It may be recalled [21, 22] that the  $IS_A$  transition is a case of nucleation (i.e. the growth of the translucent  $S_A$  phase can be seen visually at the bottom of pycnometer bulb as the transparent isotropic liquid appears to float over it with a clear cut separating boundary, emphasizing the coexistence of the two phases). However, no such visual observation has been noticed for the growth of the  $S_C$  phase from the isotropic liquid (and is similar to the observed uniform growth of nematic phase from the isotropic melt).

### 3.1.3. Smectic C to smectic I ( $S_C S_I$ ) transition

The  $S_C S_I$  transition is accompanied by a large density jump (1.25 per cent in TB14A and 0.88 per cent in TB16A) and an expansion coefficient peak value (see figures 2 and 3) to infer the first order nature. It may be pointed out that the growth of sixfold symmetry (as underlined by the growth of the smectic I hexagons in the one-dimensional smectic C liquid) may be continuous as expected [11] at the second order transition boundary in the vicinity of  $S_C S_F$  tricritical point. However, the possibility for the occurrence of the first order nature of the  $S_C S_I$  transition is not ruled out due to the structural analogy of the  $S_C S_I$  transition in the wake of the observed [11] tricritical behaviour for the  $S_C S_F$  phase transition. However, our density results in TB14A and 16A yielded a clear first order nature for the  $S_C S_I$  transition in agreement with the reported results at the  $S_C S_F$  TCP. It is also noticed that our previous dilatometric experiments inferred [14] a first order  $S_C S_F$  transition which involves a structurally similar change at the  $S_C S_I$  transition. The observed first order nature of the  $S_C S_I$  transition cannot be argued as a fluctuation mediated one, since considerably large thermal ranges were observed for the preceding smectic C phase (26°C in TB14A and 21.2°C in TB16A). This suggests the important role played by the strong coupling between the exponential growth of hexatic-positional correlations (low temperature smectic I or F phase hexagons) and the long range tilt order (in the high temperature  $S_C$  phase), thereby pronouncing the first order nature of the

Table 2. Pretransitional effects estimated through  $\alpha_{\text{eff}}$  from dilatometric measurements across IN, IS<sub>A</sub> and IS<sub>C</sub> transitions in TBnA homologues.

Compound	Transition	$T_C/^\circ\text{C}$	$[\Delta T]_{\text{LC}}$	$\frac{\Delta H}{dT_C/dP}$ ( $\Delta\rho/\rho$ )	FDNLR < $T_C$ > $T_C$	$\alpha_{\text{eff}}$ for < $T_C$
TB5A	I-N	235	21.4	1530 0.356 56.05	1.0 0.8	0.43
TB6A	I-N	207.8	8.2	1440 0.35 60.07	1.6 0.8	0.55
TB7A	I-N	211.6	1.6	2590 0.48 47.9	1.0 0.7	0.57
TB8A	I-S <sub>A</sub>	202.4	10.6	5740 0.96 42.0	2.2 1.4	0.44
TB9A	I-S <sub>A</sub>	199.0	6.4	6680 1.0 91.5	2.0 1.6	0.34
TB10A	I-S <sub>A</sub>	190.2	1.9	7080 1.82 72.2	1.6 0.9	0.86
TB14A	I-S <sub>C</sub>	170.0	26.0	11961 1.21 33.03	2.0 1.9	0.73
TB16A	I-S <sub>C</sub>	160.0	21.2	8440 0.745 29.99	2.4 2.4	0.49

$T_C$ , the transition temperature;  $[\Delta T]_{\text{LC}}$ , the liquid crystalline thermal span in  $^\circ\text{C}$ ;  $\Delta H$ , the enthalpy of the isotropic liquid to the first LC phase transition in  $\text{J mol}^{-1}$ ; FDNLR, the fluctuation dominated non-linear region above and below the transition temperature while  $\alpha_{\text{eff}}$  is estimated for the former case and  $dT_C/dP$  is the pressure dependence of the transition temperature in  $\text{K kbar}^{-1}$  estimated by the Clausius-Clapeyron equation.

transition. The pressure dependence of the S<sub>C</sub>S<sub>I</sub> transition temperatures (see table 1) are found to be comparable to that of the reported values [12] ( $39.1 \text{ K kbar}^{-1}$ ) for the S<sub>C</sub>S<sub>I</sub> transition in TB9A.

### 3.1.4. Smectic I to smectic F [S<sub>I</sub>S<sub>F</sub>] transition

The observed small density jump ( $\Delta\rho/\rho \times 100 = 0.026$  for TB14A and 0.028 for TB16A) and a small volume expansion coefficient anomaly (inset in figures 2 and 3) suggest a nearly second order nature of the S<sub>I</sub>S<sub>F</sub> transition. The S<sub>I</sub>S<sub>F</sub> transition is an ordered-ordered intrasmectic transition and is expected to be a second order transition. However, it is noticeable that the S<sub>I</sub>S<sub>F</sub> transition in TB9A is reported [12] with a small but noticeable enthalpy of  $19 \text{ J mol}^{-1}$  attributed to the pretransitional anomaly. However, a further meticulous observation into the observed density increment at the S<sub>I</sub>S<sub>F</sub> transition temperature (when the experimental error is considered) in TB14A and 16A confirms it to be a second order transition in agreement with the earlier dilatometric [8, 10] studies in TB10A and 12A as well as the pressure [23] studies in TB10A compounds. The observed increase of slope of density  $d\rho/dT$ , (as ana-

logous to a change of slope of the  $P$ - $T$  plot) reflects the denser packing of molecules in the lower temperature smectic F phase.

### 3.2. Pretransitional effects at the isotropic to first mesomorphic transition in TBAA series

The density observations reveal that a first order isotropic liquid to LC transition (due to the fact that isotropic infinite rotational symmetry breaks down at all such transitions underlined by the lower temperature nematic or smectic phase) is accompanied by the growth of density fluctuations right in the isotropic phase above the transition point. These fluctuations converge at the transition point and is accompanied by the largest observed density increment. However, these fluctuations also persist on the lower temperature side of the phase transition, which later merge out to form the more ordered low temperature liquid crystals (LC) matrix. As one carries out the density experiment with the same rate of cooling ( $1^\circ\text{C h}^{-1}$ ) in all the compounds, it provides an opportunity to study the pretransitional effects on a comparable scale, i.e. across IN, IS<sub>A</sub> and IS<sub>C</sub> transitions (in a homologues series such as TBnA).

We aim to study the thermal range of density fluctuations, their qualitative growth from isotropic liquid reflecting the mean extensions (average volume) in the fluctuation dominated non-linear regions (FDNLR), and their average lifetimes. The density results are fitted to a relation given as  $|\rho_{I-LC} - \rho_T| \propto |T_{I-LC} - T|^{1-\alpha_{\text{eff}}}$  where  $\rho_{I-C}$  and  $T_{I-C}$  are the observed density and transition temperature values,  $\rho_T$  and  $T$  are the observed density and corresponding temperature relevant to the fluctuation dominated non-linear regime of density values and the  $\alpha_{\text{eff}}$  describes the power law dependence of fluctuations on the high temperature side of the transition.

The goodness of the fit is demonstrated through the obtained  $p$ -values ( $p > 0.99$ ) by performing the  $\chi^2$ -test. The estimated  $\alpha_{\text{eff}}$  values and their thermal stabilities are given in the table 2.

The enthalpy value of transition (see table 2) involved in the isotropic to the first liquid crystalline transition is found to attain higher values, as one goes from IN, IS<sub>A</sub> and to IS<sub>C</sub> transitions, due to the difference in the growth of relevant LC order from the isotropic liquid. Consequently, the pressure dependence of the relevant transition temperature is also found to be highest for the IN transition implying the steeper  $P$ - $T$  interface. However, it is noticed that the thermal range of the fluctuation dominated non-linear regions on either sides of the IS<sub>C</sub> transition is found to be symmetrical, while it differs considerably in the case of IN and IS<sub>A</sub> phase transitions. This may be due to the growth of orientationally self-balanced twofold degenerate biaxial thermal fluctuations at the IS<sub>C</sub> interface. However, the situation differs at both the IN and IS<sub>A</sub> interfaces, where the development of the growth of the uniaxial thermal fluctuations are not so relatively ordered. Further, the  $\alpha_{\text{eff}}$  values estimated on the higher temperature side of the I-LC transition are observed to attain higher values at the IN interface. However, on the equal time-scale, i.e. from the observed range of FDNLR regions (namely FDNLR for IS<sub>C</sub> > IS<sub>A</sub> > IN), it is found that pretransitional effects are relatively strong at the IS<sub>C</sub> transition followed by the IS<sub>A</sub> transition. This suggests the comparatively steeper growth of fluctuation dominated regions as well as higher average thermal ranges of such fluctuations before they merge to form the low temperature LC matrix.

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