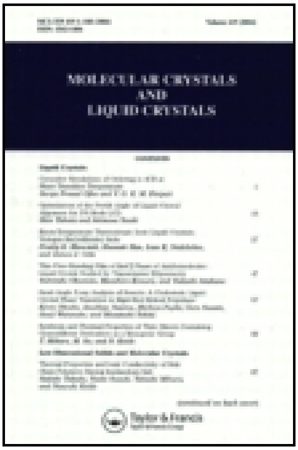

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PHASE TRANSITION AND PRE TRANSITIONAL STUDIES ACROSS ISOTROPIC—SMECTIC-F AND G PHASES IN N-(P-N-ALKOXYBENZYLIDENE)P-N-TETRADECYL ANILINES, (NO. 14): A DILATOMETRY STUDY

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Phase Transition and Pre Transitional Studies across Isotropic - Smectic-F and G Phases in N-(P-n-alkoxybenzylidene)p-n-tetradecyl Anilines, (nO.14): A Dilatometry Study

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The higher homologues of N-(p-n-alkoxy benzylidene) p-n-alkylanilines, (nO.m) viz., N-(p-n-alkoxybenzylidene)p-n-tetradecylaniline, nO.14 compounds with n=10 to 16 exhibit a direct smectic-F phase from cooling the isotropic liquid, while the compound with n=18 shows a direct smectic-G phase from isotropic liquid. Density studies are carried out across isotropic-smectic-F, isotropic-smectic-G and smectic-F-smectic-G phases. The density jumps and thermal expansion coefficient maxima across these phases infer a first order IF or FG transition. The strength of pre-transitional effects studied through α_{eff}across isotropic to nematic (IN), isotropic to smectic-A (IA) and isotropic to smectic-C (IC) phases. The α_{eff} and the fluction dominated non linear region (FDNLR) values for $T>T_c$ obtained for these compounds underlines the increase of orientational disorder extended deep into the isotropic liquid for the formation of either smectic-F or smectic-G phase than in the case of the lower homologues exhibiting IN and IA phase transitions.

Keywords: nO.14; Iso. – Smectic F; density jump; Thermal expansion coefficient maxima; Phase Transition studies; pre-transitional effects

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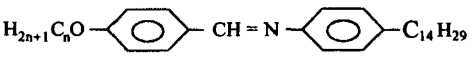
INTRODUCTION

Investigations of isotropic to mesophase transitions especially involving the smectic phases are interesting in the light of the theoretical predictions[1] for the growth of 2D and 3D crystals from the isotropic liquid phase. The growth of smectic-F and smectic-G phases from Isotropic liquid involves (in addition to the long range bond orientational order common to both the cases) the formation of 2D structural order with short range positional hexagonal correlations in the smectic-F phase. The 3D structural order is grown with long range positional hexagonal correlations in the smectic-G phase. Hence, the smectic-F to smectic-G (FG) transition characteristically involves the growth crystalline dimensionality by one order, the freezing of swift distortion. The FG transition altogether points to change of the environment of 2D bond orientational order to a 3D, mediated possibly through a first order transition.

The compounds reported to exhibit direct transitions from isotropic liquid to smectic-F and G are few[2,3,4]. The study of the nature of phase transitions and pretransitional effects across transitions is informative to reveal the details of molecular interactions. The reported[2,3] dilatometric studies across isotropic to smectic-F phases involves the 10O.14 and 12O.m compounds (with m = 12, 14 and 16) of nO.m series. The present work intended systematic studies across IF transition is achieved through the relevent synthesis of compounds (synthesis of N-(p-n-undecyloxybenzylidene)-p-n-alkylanilies, 11O.m compounds)with a direct smectic-F phase from isotropic liquid. However, the observed[4] direct smectic-F isotropic transition (with n = 11 and m > 12) and the absence of such transition in the 10O.12 further motivated the authors to study the effect of alkoxy chain length (keeping the alkyl chain length at n = 14). As is well known, since the lower alkoxy chain compounds exhibit nematic and orthogonal smectic phases in addition to tilted smectics, the synthesis has been carried out for the compounds with n starting from 7 and extended upto 18. The synthesis and characterisation of nO.14 compounds resulted[5] not only in quenching of orthogonal phases (such as smectic-A and smectic-B) when n > 10, or the onset of direct smectic-F transition (from isotropic liquid), a higher ordered direct smectic-G transition quenching even the smectic-F phase with n = 18. In this paper, we present the temperature variation of density across isotropic to smectic-F, smectic-G phases and smecic-F to smectic-G phases and the pretransitional effects observed at isotropic to smectic-F (IF) and G (IG) transitions.

EXPERIMENTAL

The N-(p-n-alkoxybenzylidene)p-n-tetradecyl anilines (nO.14, where n = 11 to 18) were synthesised by the condensation reaction between p-n-alkoxybenzaldehydes viz., undecyloxy, tridecyloxy, tetradecyloxy, pentadecyloxy, hexadecyloxy and octadecyloxy (0.01m.mol) and p-n-tetradecylanilines (0.01m.mol) as described[6] earlier. The liquid crystalline phases were texturally characterised by using a Hertel-Reuss (Super pan-II) polarising microscope equipped with an indigenous hot stage with an accuracy of 0.1 ^{0}C. The density investigations are carried out[7] by a U-shaped bicapillary pyknometer. The absolute error in density measurements is 10^{-4} gm/cm^3. The cooling rate observed (fig. 2-3) during the density measurements is 0.5 ^{0}C/hr. The Differential Scanning Calorimetry (DSC) (fig. 1) is carried out with a Perkin-Elmer DSC7 instrument. The general molecular formula of nO.14 compounds is



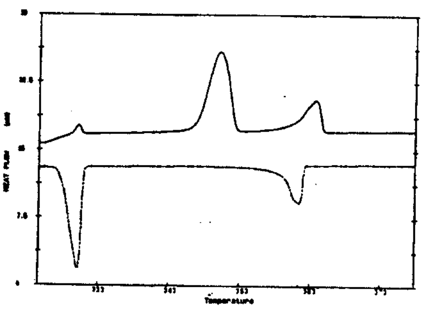


FIGURE 1 DSC Thermogram of 14O.14

RESULTS AND DISCUSSION

The observed transition temperatures (T_c) from dilatometric studies the density jumps ($d\rho/\rho$ x 100) and the volume expansion coefficient maxima (α_v) associated with the phase transitions are presented in table - 1. The lower homologues of nO.14 compounds ($n < 9$) exhibit the phase variant ABG, while the higher homologues of nO.14 compounds ($m > 10$) exhibit the FG phase sequence between the Isotropic liquid and the solid crystalline phases. That is, the higher homologues are found to possess tilted quasi two dimensional long range bond-orientational order (smectic-F phase for $n = 10$ to 16 and smectic-G phase for $n = 18$) liquid crystalline phases directly from the isotropic liquid with the quenching of orthogonal smectic (A,B) phases. The sample taken without the coverslip over it on a glass slide exhibits a six sectored polygon under crossed polars characteristic[5] (F/G) phase from isotropic liquid. With increase of flexible end chain length in nO.14 compounds along with our earlier observations in 10O.14[2] and 12O.m[3] of nO.m compounds is found to agree[8] with the

TABLE I The Transition Temperatures in K (Dilatometry), Density jumps and Thermal Expansion Coefficient maxima of nO.14 compounds.

nO.14	Phase variant	Transition Temperatures		$(\Delta\rho/\rho)$ x 100	$\alpha_{max.}$ $10^{-4}\,{}^{0}C^{-1}$	Ref
		Iso–SmF/G	SmF-SmG			
10O.14	FG	356.5	355.1	2.14	295	[2]
11O.14	FG	361.6	360.8	0.33	54	PW
12O.14	FG	360.5	359.5	0.81	136	[3]
13O.14	FG	360.9	360.0	0.50	125	PW
14O.14	FG	361.5	360.4	0.17	110	PW
15O.14	FG	361.6	359.8	1.2	132	PW
16O.14	FG	360.9	359.7	0.11	95	PW
18O.14	G	361.8		0.92	93	PW

mean field excluded volume theories developed for the micellar systems. This concurrence underlines the fact that the increase in flexible end chain length of a molecule favours the occurrence of higher ordered liquid crystalline phases. It is also observed[2,3] that the ratio of the flexible end chain (alkoxy) to the rigid core length in nO.14 compounds may attain a favourable value in nO.14 compounds the reason for the formation of a smectic-F phase directly from the isotropic liquid as has been the case of 10O.m (for m = 14) and 12O.m compounds (with m = 12, 14 and 16).

The estimated value of molar volume in isotropic liquid per an increment of methylene group in these compounds at $T_{IF/IG}$ + 5 K is found to be in the range of 14.5 to 17.9 x 10^{-6} cm^3 mol^{-1}, is found to agree with the reported[2,3,6,9,10,11,12,13] values for the other nO.m compounds with n = 4,5,6,7,8,9,10 and 12. The rate of the decrease of the density with temperature $(d\rho/dt)_{Iso.}$ in the isotropic phase agrees with other liquid crystalline compounds[2,3]. The temperature dependence of density ρ(T), volume expansion coefficient α(T) for 14O.14 and 18O.14 are illustrated in figures 2 and 3 respectively.

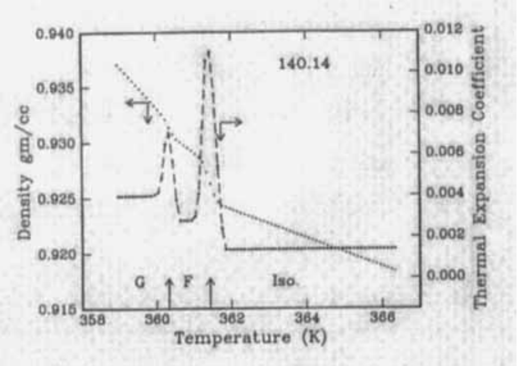


FIGURE 2 Temperature variation of Density (ρ) and Thermal Expansion coefficient (α) in 14O.14

Isotropic - smectic-F Transition

The isotropic - smectic-F phase transition is indicated by a sudden jump in density at the transition (estimated from the vertical distance between extrapolated density values from isotropic to smectic-F phase regions).

The jump in density and the following thermal expansion coefficent maxima suggests that the transition is of first order in nature. It is observed that the density jumps (Δρ/ρ x 100) show an even-odd effect (with an exception of 14O.14) unlike the case of 12O.m compounds whose values decrease[3] with the increase of alkyl chain length. The values are found to be lower compared to the reported values for 10O.14 and 12O.m compounds. The trend of decreasing density jumps with the increasing length of flexible end chain and the growth of tilted phases from isotropic liquid signifies a decreasng thermal potential barrier across the isotropic to tilted liquid crystalline interface. These results are found in agreement[14] with the reported observations across isotropic-smectic-C transition. However, the present case of isotropic to smectic-F interface in these compounds (as well as in 12O.m series) differs from that at isotropic - smectic-F interface. An additional inplane hexagonal positional correlations now to considered (in addition to the long range tilt orientation) in the former case. Hence, as a result of the comparatively higher symmetry involved at the isotropic - smectic-F phase, the higher potential barrier is supposed to result the higher density jump (with the increase of the flexible end chain). However, it is observed that the density jumps are small in the nO.14 compounds to present a distinct feature in the case of 12O.m series.

These density results demonstrate the dominant role of LC natured tilt relevant potential barrier long range orientational order, which is obviously more prone to the temperature rather than that one relevant to the positional correlations. This effect is more prominent in the case of alkoxy chain increase (in present work) than in the case of alkyl chain length 12O.m series[3].

The density jump across isotropic - smectic-F transition is found to be lower compared to that at IA interface in the lower homologues[15] of nO.14 and other nO.m and liquid crystalline compounds[13] which exhibit a direct smectic-A phase.

Smectic-F - Smectic-G Transition

The density jumps ($\Delta\rho/\rho$ x 100) and their thermal expansion maxima (α_v) at smectic-F - smectic-G transition are given in table-1. The FG transition in these compounds is accompanied by density jumps of the order of the magnitude similar to that observed[6] across I - N transition. Jump in ($\Delta\rho/\rho$) indicates that FG transition is a first order transition.

However, in view of the small thermal range of smectic-F phase the DSC measurements (figure-1) could not resolve the FG transition from IF transition inspite of slow scan rate 1 ^{0}C/hr. The density jumps observed across smectic-F – smectic-G transition in these compounds are in agreement with the values obtained[3] for the 12O.m series. However, the compounds 10O.14[2] and TB7A and 9A[16] exhibited a weak first order FG transition.

Isotropic to Smectic-G transition

At this transition the infinite rotational symmetry of isotropic phase is broken with the growth of a 3D long range positional correlations of bond orientational order. The compound 18O.14 exhibits a direct smectic-G transition from isotropic liquid. The variation of density, the thermal expansion coefficient with temperature in isotropic and smectic-G phases is shown in figure-3.

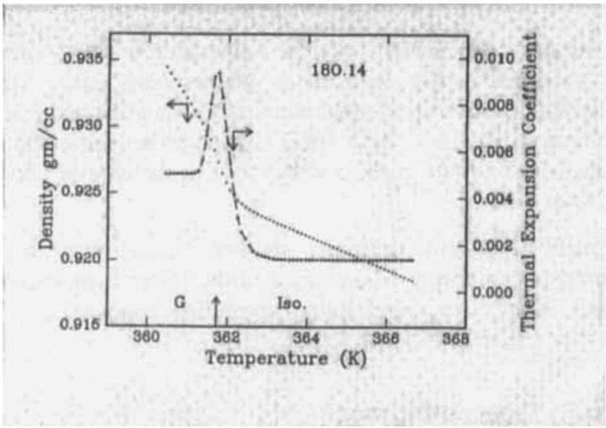


FIGURE 3. Temperature variation of Density (ρ) and Thermal Expansion Coefficient (α) in 18O.14

The density jump and the thermal expansion coefficient maxima (table-1) supports the first order nature of the transition. The density jump of 0.91% at this transition is found to be less compared to the values resulted in the case of the lower homologues[15] of this series across isotropic to smectic-A transition. However, the value is found to be higher compared to that obtained for isotropic to smectic-F transition for the compounds of nO.14 series, while the value is of comparable magnitude with the values obtained for isotropic to smectic-F transition in 12O.m series. This value is different from values at the onset of smectic-A, C, F and G phases grown directly from isotropic liquid. It is noticed that the dimensionality and the crystal structural order change is different (from smectic-A,C and F)at the isotropic to smectic-G transition resulting in a larger density jump. This decreasing trend of density jump with the increase of the flexible end chain (irrespecive of type of transition) is in agreement with that reported[14] in the case of TBnA and 12O.m series[3]. This trend of lower jumps can be explained as follows; when the end aliphatic chains are longer, the total volume occupied per methylene unit is comparatively smaller than volume required per methylene unit with smaller end chains. That is, the volume swept by methylene units in the compounds possessing small aliphatic chains is larger while the close packing of aliphatic end chains results in smaller volume variation at the transition. The above trend is more pronounced in the case of alkoxy chain rather than alkyl chain.

Pre transitional effects across isotropic liquid to first mesomorphic phase

The density observations show that isotropic liquid to a mesomorphic transition (due to the fact that isotropic infinite rotational symmetry breaks down at all such transitions) is of first order nature. The transition is accompanied by growth of density fluctuations right in the isotropic phase above the transition point. These fluctuations diverge (figure-4) at the transition point and as accompanied by the largest observed density increment at transition. However, these fluctuations also persist at lower temperature side of the phase transition which later merge to form a more ordered low temperature liquid crystalline matrix. The density experiment is carriedout with the same rate of cooling (0.5 ^{0}C/hr. as reported most of the nO.m and TBnA compounds) to study the pretransitional effects on comparative scale i.e., across I - N[6], I - A[13], I - C[14], I - F[2,3] and I - G transitions.

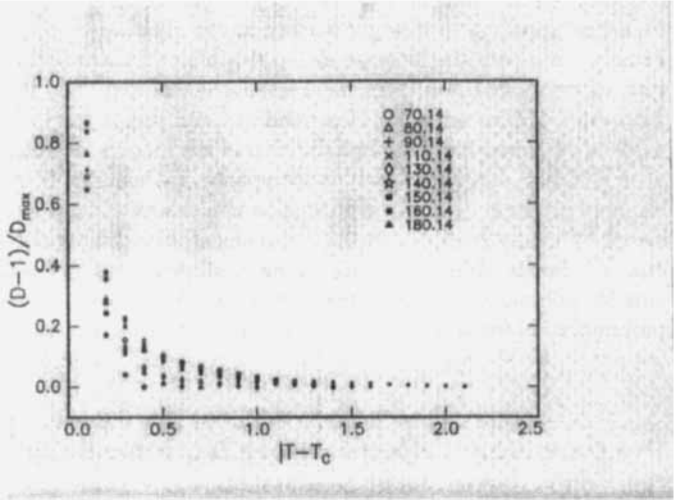


FIGURE 4 Divergence of Density Fluctuations (D-1)/D_{max} (where $D=\Delta\rho/\Delta\rho_{max}$) near the Transition point as function of |T – T_C| in nO.14 compounds

The observed nonlinear density variation on the isotropic side is representative of the volume swept by the molecules (with characteristic hindered rotational degrees of freedom) accompanied by the relevant density fluctuations that grow with the mesomorphic phase. This region known as FDNLR denotes the density fluctuations. In the vicinity of phase transition, the volume occupied by a molecule (with specified potential energy) is influenced by the growth of the oncoming molecular structural order of the low temperature liquid crystalline phase. The molecules at a phase boundary are subjected to smooth shift distortion (as at IF interface) they attain a denser environment because of closer packing at lower temperature phase. In the light of the data[3,6,13,14] available on the pretransitional effects at IN, IA, IC and IF phase transitions, the growth of the smectic-F and smectic-G (from isotropic liquid) phases is estimated to a first approximation through the exponent α from the observed density increment in the density fluctuation dominated nonlinear region (FDNLR) for $T > T_C$. The density increment in the fluctuation dominating nonlinear region can account for the growth of smectic-F and smectic-G positional correlations. However, the bare density data by themselves may not be able to resolve their contributions independently due to the simultaneous onset of orientational, translational and tilt order parameters at IF transition. Further, a comparison between the growth of smectic-A phase with IC, IF and IG transitions is made to estimate the relative growth of hexagonal order from density data.

The observed density data in FDNLR (for $T > T_C$) is fitted to the relation

$$|\rho_{I\text{-}LC} - \rho_{Ti}| \propto |T_{I\text{-}LC} - T_i|^{1-\alpha_{eff.}} \qquad \text{.......(1)}$$

$\rho_{I\text{-}LC}$ and $T_{I\text{-}LC}$ are the observed densities at transition and transition temperature respectively, while ρ_{Ti} and T_i are the observed density and corresponding temperature T_i of interest in the FDNLR and $\alpha_{eff.}$ describes the power law dependence of fluctuations on the high temperature side of the transition.

The goodness of the fit (1) is demonstrated through the p-values ($p > 0.99$) follows chi - square test. The estimated values and the thermal stabilities along with the literature data at IN, IA, IC along with IF and IG are given in table-2.

TABLE II The $\alpha_{eff.}$ Values estimated from density results in the FDNLR for first order IN, IA, IC, IF and IG transitions.

.nO.m	Trans.	$\alpha_{eff.}$ $T>T_C$	$(\Delta T)_{FDNLR}$ range	Ref	nO.m	Transition	$\alpha_{eff.}$ $T>T_C$	$(\Delta T)_{FDNLR}$ range	Ref
4O.4	IN	0.78	0.3	[6]	7O.14	IA	0.62	1.4	[15]
4O.5	IN	0.74	0.5	[6]	8O.14	IA	0.24	0.7	[15]
4O.6	IN	0.70	0.6	[6]	9O.4	IA	0.64	1.1	[13]
4O.7	IN	0.83	0.5	[6]	9O.5	IA	0.50	0.6	[13]
4O.8	IN	0.79	0.4	[6]	9O.6	IA	0.62	1.3	[13]
4O.9	IN	0.52	0.5	[6]	9O.8	IA	0.65	1.3	[13]
4O.10	IN	0.62	0.2	[6]	9O.14	IA	0.26	1.5	[15]
4O.12	IN	0.79	0.3	[6]	TB8A	IA	0.44	1.4	[14]
6O.3	IN	0.34	0.6	[13]	TB9A	IA	0.34	1.6	[14]
6O.4	IN	0.62	0.3	[13]	TB10A	IA	0.86	0.9	[14]
6O.5	IN	0.67	0.4	[13]	11O.14	IF	0.26	1.4	PW
6O.6	IN	0.26	0.4	[13]	12O.12	IF	0.38	2.5	[3]
6O.7	IN	0.53	0.3	[13]	12O.14	IF	0.40	1.9	[3]
6O.8	IN	0.51	0.4	[13]	12O.16	IF	0.39	2.2	[3]
TB5A	IN	0.43	1.0	[14]	13O.14	IF	0.39	1.0	PW
TB6A	IN	0.55	1.6	[14]	14O.14	IF	0.23	0.4	PW
TB7A	IN	0.57	0.7	[14]	15O.14	IF	0.20	2.2	PW
TB14A	IC	0.73	1.9	[14]	16O.14	IF	0.29	0.6	PW
TB16A	IC	0.49	2.4	[14]	18O.14	IG	0.41	1.7	PW

A relative study of the strength of the pre transitional effects over IN, IA, IC, IF and IG transitions (table-2) through $\alpha_{eff.}$ values throws light on the higher values with IN transition than at IA transition. However, it is noticed that the values at IC transition falls in the higher side of the values reported for IA transition. The pre transitional effects studied through $\alpha_{eff.}$ for nO.14 compounds (for n = 11 to 16 and 18) are characteristic with lower values. The pre-transitional effects are studied[15] for 7O.14, 8O.14 and 9O.14 (the lower homologues of nO.14 series) across IA transition. However, the compounds 8O.14 and 9O.14 strikingly bear the lower values as a prelude to the on coming smoothening effect of orientational degree of freedom with the increasing chain length. This trend (of an altogether lower values of observed $\alpha_{eff.}$) may be clearly visible and when the comparison is extended study of pre-transitional effects to IF transition for nO.14 compounds (for n = 11 to 18). It may be argued (from the lowest reported $\alpha_{eff.}$ values along with the decreasing trend of $\alpha_{eff.}$ exhibited at IN transition in lower homologues on nO.m compounds, for n = 4 to 18), that the squeezing of orientational degrees of freedom is more effective at the IN interface (with largest $\alpha_{eff.}$) in lower compounds. The disorder contributions are clearly observed due to the effect of increasing alkyl flexible end chain length (m in nO.m compounds) in the presence of fixed, but effective orientation of alkoxy chain (n) to the essential rigid core through electronegative oxygen atom. That is, the decreasing trend of $\alpha_{eff.}$ becomes negligible with the increase of n value in nO.m compounds. The present case of higher homologues of nO.14 compounds (n = 11 to 16) is rather different, that the inductomeric effect lessen the coupling of n-chain configuration to the core. The higher homologues of nO.14 molecules behave rather resolved (between end chain and rigid cores contributions). This leads to the decreasing of coupling strength of orientational fluctuations tuned by the temperature as relative field. The lower observed values of $\alpha_{eff.}$ across IF transition in higher homologues of nO.14 series distinctly speakout the successfully introduced orientational disorder in these compounds. Further, the observed $\alpha_{eff.}$ is found to attain lower values before smectic-F phase is formed. The $(\Delta T)_{FDNLR}$ for $T > T_C$ is found to be comparable to the observed value for nO.14 compounds may be attributed to the fact that the growth of quasi 2-dimensional smectic-F liquid crystalline phase is rather slow. It is also noticed that the $\alpha_{eff.}$ values for IF transition exhibit odd-even effect (except 13O.14) similar to the trend observed at IN[6] and IA[13] interfaces. The reason for this alternating values of $\alpha_{eff.}$ is argued on the basis of the alternating contributions of axial polarisabilities with the increment of additional methylene unit as detailed in Marcelja's model[17]. It is observed that 18O.14 exhibits an IG transition, G phase differing from -F in the dimensionality of the crystal growth involved. The observed thermal span to be 1.7 ^{0}C for FDNLR infers the comparable growth process of smectic-G hexagon to that of smectic-F from the isotropic liquid. Furthermore, the $\alpha_{eff.}$ observed for the 18O.14 across IG transition is relatively higher than the observed across IF transition and is due to the effect of orientational disorder in higher homologues. The possession of relatively higher value of 0.41 may be due to higher dimensionality involved with smectic-G crystal compared to smectic-F as expected with the possible orientational disorder in higher homologues.

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