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Phase transition studies in 9O.6 and 9O.8

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The variation of density with temperature across different phases for two homologues of the $N(4-n\text{-alkyloxybenzylidene})\text{-}4'\text{-}n\text{-alkylanilines}$ (9O.6 and 9O.8) exhibiting the $S_A S_C S_T S_G$ phase variant is determined. The influence of pretransitional effects in the fluctuation dominated non-linear region in the vicinity of the isotropic-smectic A transition and the nucleation growth analogy of this transition are discussed.

1. Introduction

Liquid-crystalline materials have been long known [1-6] for their subtle mesomorphism. Although considerable research has successfully advanced the understanding of the static and dynamic behaviour at the isotropic-nematic (IN) transition [7] to exploit the underlying molecular mechanism in electrooptic displays (for example, the twisted nematic cell) the isotropic-smectic A (IS_A) transition [8-22] is still an untapped source of information for its suitability in liquid-crystalline displays. The growth of molecular orientational as well as translational orders in a completely disordered isotropic liquid characterizes the IS_A phase transition. Systematic experimental investigations of the IS_A transition characteristics with regard to qualitative and quantitative variations of the flexible end part of a liquid crystal molecule as well as effect of external applied field and temperature has become the interest of recent research in order to study the non-linear response [10, 16, 21, 22]. Much attention has been paid [8] to the IS_A transition in understanding its observed analogy with nucleation growth and furthermore to the accompanying pretransitional fluctuations. The infinite isotropic symmetry is broken at the IS_A transition it is of interest to study whether the orientational correlations set in first before the positional correlations characteristic of a layered smectic A phase or both of them manifest simultaneously in the isotropic liquid and grow into the S_A structure. Though recent electric field experiments on non-chiral liquid crystal molecules exhibited [22] a considerable pretransitional anomaly in the vicinity of the IS_A transition, it may be mentioned that chiral IS_A transitions differed in the nature of their sign because of an altogether different inherent dipolar nature of the molecule.

The smectic A-smectic C transition involves the growth of a tilted ordering of molecules in the smectic A layered structure resulting in a biaxial S_C phase. As much importance is attached to the $S_A S_C$ transition and its formation is described [23] as due to the application of uniaxial compressional stress normal to the smectic layers while its analogy with the helium superfluid transition results in a second order continuous $S_A S_C$ transition. Smectic A-smectic C tricritical points are also reported [24-29] either as

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fluctuation mediated weak first order $S_A S_C$ transitions, weak first order transitions due to the strong coupling between the chiral group to the orientational order, or due to the narrow smectic A thermal range and also in the vicinity of $NS_A S_C$ multicritical points.

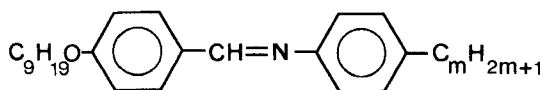
The identification and discovery [5] of bond orientationally ordered hexatic S_B , S_F , S_I , etc. phases aroused much interest as they represent the very first experimental evidence to support the Thouless–Kosterlitz theoretical predictions of melting in two dimensions [30]. The pretransitional effects that accompany the growth of such bond orientational order at the interface is equally interesting to study. The smectic C–smectic F transition involves the growth of in-plane positional correlations of tilted hexagonal nets with poor registry over a number of layers in a direction normal to the layers as manifested by swift distortion resulting in large discontinuities of thermodynamical properties at the phase separation boundary.

The smectic F–smectic G transition is accompanied by the development of strong long range correlations of hexagonal nets within the layers as well as between the layers in addition to the long range orientational order to establish a structurally more ordered S_G phase. During the $S_F S_G$ transition the higher temperature S_F phase suffers a loss of possible cooperative librational degrees of freedom as the hexagons attain an orthogonal arrangement in the low temperature S_G phase. The study of the $S_F S_G$ transition presents a scope to investigate the accompanying critical fluctuations.

The $nO.m$ $N(4-n$ -alkyloxybenzylidene)-4'- n -alkylanilines compounds are well-known for their rich and interesting smectic polymorphism [1–6]. The synthesis followed by miscibility studies [31–33] of the higher homologues of the $nO.m$ compounds motivate a systematic study of the IS_A transition and the phase transitional phenomena involving exotic phases (S_F). The density investigations and the study of the pretransitional effects in the fluctuation dominated non-linear regions of the IS_A , $S_A S_C$, $S_C S_F$ and $S_F S_G$ phase transitions carried out in liquid-crystalline compounds, viz. $N(4-n$ -nonyloxybenzylidene)-4'- n -hexylaniline (9O.6) and $N(4-n$ -nonyloxybenzylidene)-4'- n -octylaniline (9O.8), of the $nO.m$ homologous series in the light of reported data on 9O.4 and 9O.5 compounds [34, 35] (and other compounds exhibiting the IS_A transition) along with a detailed study of the nucleation analogy of the IS_A transition are presented.

2. Experimental

The transition temperature recorded with a Hertel Reuss Super Pan II polarizing microscope with an indigenously fabricated heating stage and the enthalpy values from DSC are given in table 1. The observed phases were identified [5] from their standard characteristic textures as well as by miscibility studies. The density was measured with a pycnometer with an absolute accuracy of 10^{-4} g cm $^{-3}$ as reported earlier [12, 15]. The accuracy of the temperature measurements for both the thermal microscopy and the density measurements was $\pm 0.1^\circ\text{C}$. The general molecular formula for the 9O. m homologous series is



3. Results and discussion

The synthesis and characterisation of phases in the compounds 9O.6 and 9O.8 were reported earlier [36]. The variation of density (ρ) with temperature is given in figures 1 and 2 for compounds 9O.6 and 9O.8, respectively. The estimated contribution of molar

Table 1. The phase transition temperatures and enthalpy values (kJ mol^{-1}) obtained from thermal microscopy (TM) and DSC as well as density (g cm^{-3}) measurements for the IS_A , $S_A S_C$, $S_C S_F$ and $S_G S_K$ transitions for 9O.6 and 9O.8.

Method	9O.6					9O.8				
	$IS_A/^\circ\text{C}$	$S_A S_C/^\circ\text{C}$	$S_C S_F/^\circ\text{C}$	$S_F S_G/^\circ\text{C}$	$S_G S_K/^\circ\text{C}$	$IS_A/^\circ\text{C}$	$S_A S_C/^\circ\text{C}$	$S_C S_F/^\circ\text{C}$	$S_F S_G/^\circ\text{C}$	$S_G S_K/^\circ\text{C}$
TM	87.0	80.5	77.5	75.0	49.0	85.5	76.0	75.2	72.0	47.0
DSC	84.8	78.1	75.3	—	50.0	85.3	76.4	74.7	—	45.0
Density	87.0	80.5	77.5	75.0	—	85.5	76.0	75.2	72.0	—
ΔH	7.636	—	3.906	—	47.629	9.127	—	4.200	—	43.468

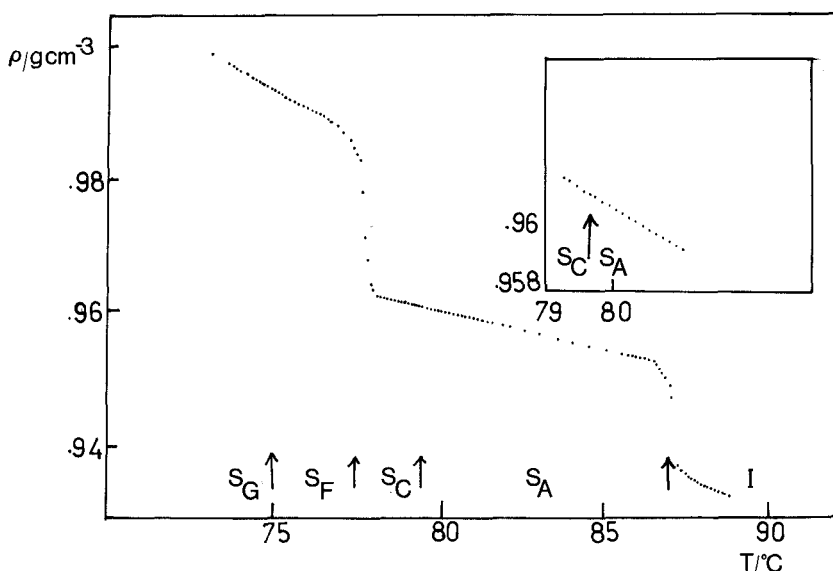


Figure 1. Variation of density (ρ) with temperature for the compound $N(4\text{-nonyloxybenzylidene})4'\text{-}n\text{-hexylaniline}$, 9O.6.

volume per methylene group increment for the 9O. m series (in the light of data on 9O.4, 9O.5, 9O.6 and 9O.8) in the isotropic liquid at $T_{IS_A} + 5^\circ\text{C}$ is found to be $16 \times 10^{-6} \text{ m}^3 \text{ mol}^{-1}$ in agreement with reported values for the 4O. m [37], 5O. m [38] 6O. m [39] and 7O. m [33] series of $nO.m$ compounds.

3.1. Isotropic–smectic A (IS_A) transition

The density jump ($\Delta\rho/\rho \times 100$) associated with the IS_A transition (1.51 for 9O.6 and 1.95 for 9O.8) and the large volume expansion coefficient ($\alpha = (1/M_V)dM_V/dT = 44 \times 10^{-3} \text{ }^\circ\text{C}^{-1}$ for 9O.6 and $49 \times 10^{-3} \text{ }^\circ\text{C}^{-1}$ for 9O.8) indicate the first order nature of the transition. The observed higher density jumps across the IS_A transition compared to the isotropic–cholesteric transition density jump ($\Delta\rho/\rho \times 100 = 0.3$) is due to the development of higher structural ordering in the isotropic liquid at the IS_A transition. The density jump data and the enthalpy values associated with the IS_A transition and the pressure dependence of the transition temperatures estimated from the Clausius–Clapeyron equation

$$dT_i/dp = T_i(\Delta V/\Delta H),$$

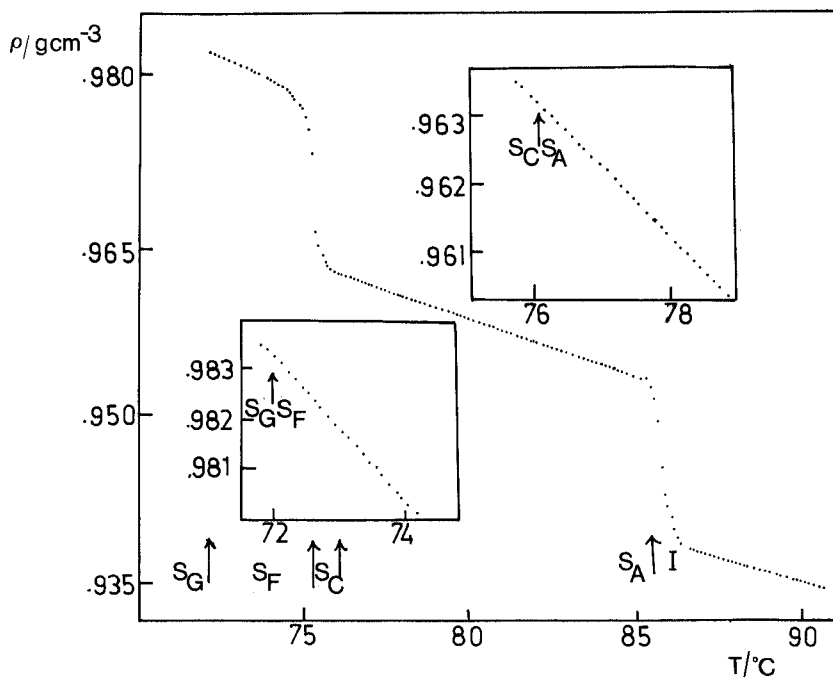


Figure 2. Variation of density (ρ) with temperature for the compound *N*(4-*n*-nonyloxybenzylidene)4'-*n*-octylaniline, 9O.8.

where T_t , ΔV and ΔH are the transition temperature, change in volume and enthalpy values of the transition, respectively. The data are presented in table 2 for the compounds 9O.6 and 9O.8 along with other compounds which exhibit the IS_A transition.

The density jump observed for both 9O.6 and 9O.8 fall on the higher side of the density jumps across the IS_A transition. It is apparent that both the compounds exhibit large density jumps with the value for 9O.8 almost equal to the maximum value (within 2 per cent) reported in the literature. Furthermore, the density jumps as well as the ΔH values exhibit an odd-even effect at the IS_A transition for the 9O.*m* series (9O.6 and 9O.8 along reported 9O.4 and 9O.5 values). The even numbered alkyl chain members, viz. $m=4, 6$ and 8 , bear higher values than those of odd numbered members. The even numbered members apparently extend outwards further away from the average long molecular axis compared to the odd numbered members in the comparative all *trans* configuration. Thus the more extended (from the average long axis) carbon bonds of even members account for the relatively large enthalpy and density jumps underlining the higher entropy involved with the IS_A transition in attaining the characteristic periodic S_A structure. The observed results suggest that even numbered members exchange more thermal energy in attaining a more ordered layered S_A phase. Hence, it is imperative that even numbered carbon members (9O.*m*) possess relatively large thermodynamical potential barriers in order to attain the S_A orientational translational orders. A similar odd-even effect for the density change is observed across the IN transition for the *nO.m* compounds [37] and is explained [39, 40] on the basis of Marcelja's model whereby contributions of axial polarizabilities due to the end chains in a series of compounds are taken into consideration to account for the nematic orientational order at the IN transition. The observed poor agreement in the pressure

Table 2. The isotropic to smectics A transition temperature, density jumps, enthalpy values and estimated pressure dependences for various compounds.

Compound	$T_{IS_A}/^{\circ}C$	$\Delta\rho/\rho \times 100$	$\Delta H/kJ mol^{-1}$	$dT_i/dP/K kbar^{-1}$
di- <i>n</i> -Hexadecyl 4,4'-azoxy cinnamate	135.0	0.40	6.05	26.70
di- <i>n</i> -Decyl 4,4'-azoxy cinnamate	160.2	0.35	8.59	34.90
di- <i>n</i> -Decyl 4,4'-azoxy α -methylcinnamate	85.7	1.21	12.70	18.90
<i>n</i> -Amyl 4(4- <i>n</i> -dodecyloxy benzylidene) amino-cinnate	137.2	1.28	16.60	33.70
Diethyl 4-4'-azoxy benzoate	123.0	2.00	15.90	43.00
Terephthalidene bis(4'- <i>n</i> -octylaniline) TBAA8	202.4	0.96	11.10	42.00
TBAA9	199.0	1.00	12.44	41.5
TBAA10	190.2	1.82	12.53	72.20
7O.6	80.5	1.07	16.88	24.66
7O.8	83.0	1.04	14.40	27.30
7O.9	84.0	0.80		
7O.10	82.8	1.08		
8O.4	81.2	1.10	15.53	26.50
8O.8	86.2	0.70		
9O.4	80.9	1.20	16.60	39.50
9O.5	87.5	1.18	9.14	50.00
9O.6	85.5	1.50	22.38	25.48
9O.8	87.0	1.95	17.53	43.56

dependence of the IS_A transition temperature among various compounds, which accounts for the mesophase thermal stability in the domain of the changing chemical environment, may be due to the parameters being obtained by different experimental conditions. The higher slope value for the density curve for the smectic A phase as opposed to the isotropic phase ($10.4 \times 10^{-4}^{\circ}C^{-1}$ compared to $10.0 \times 10^{-4}^{\circ}C^{-1}$ for 9O.6 and $11.2 \times 10^{-4}^{\circ}C^{-1}$ compared to $9.5 \times 10^{-4}^{\circ}C^{-1}$ for 9O.8 for the S_A and I phases, respectively) suggests denser packing and higher structural ordering in the S_A phase compared to the isotropic liquid. Visual observations of density studies noted significant physical changes in the material studied in the bulb of the pycnometer at the IS_A transition. The translucent S_A phase grew uniformly in the lower part of the bulb first and less dense isotropic liquid appeared to float above the smectic A phase with a clear boundary between them. This type of coexistence with smectic A embryos appearing simultaneously at the bottom of the bulb suggests nucleation growth analogy of the IS_A transition. Further, the S_A embryos which are developed at the bottom of the bulb appeared in the phase with a characteristic translucent appearance rather than with a uniform, opaque, milky nematic appearance throughout the bulb. This suggests the simultaneous development of orientational and translational order parameters rather than the preferred growth of orientational order over the translational order. For a systematic study of nucleation growth analogy of the IS_A transition and the pretransitional effects on the fluctuation dominated non-linear regions on either side of the IS_A transition, the density data is fitted to a relation

$$|\rho_{IS_A} - \rho_T| \propto |T_{IS_A} - T|^{1-\alpha_{eff}},$$

to study the effect of alkyl chain increment in a series of compounds as the transition temperature is approached for the compounds 9O.6 and 9O.8 along with the data reported on 9O.4 and 9O.5. The results are given in table 3. The fitting of the data is demonstrated by χ^2 test whose p values are found always to exceed 0.9 with an insignificant error. The rate of cooling for the density studies of these compounds is 0.5°C h^{-1} . The width of the fluctuation dominated non-linear region is found to be more pronounced on the higher temperature (isotropic) side of the IS_A transition in all the compounds. This suggests that S_A embryos take comparatively longer times in attaining a critical size before they merge out to form a periodic smectic A matrix. The α_{eff} values are found to be higher in the isotropic side of the IS_A transition than on the smectic A side suggesting dominant pretransitional effects in the isotropic liquid and the long range development of smectic A like regions. The lower range of the fluctuation dominated non-linear region on the S_A side indicates relatively quicker growth of S_A regions which merge out to form an equilibrium smectic A phase rather than developing before attaining a critical size. Furthermore, the estimation of α_{eff} values in fluctuation dominated non-linear regions from the density investigations revealed (see tables 3 and 4) the influence of strong pretransitional effects at the IS_A transition than at the first order NS_A transition in the 6O.*m* series (non-linear density variation is not observed at the second order NS_A transition in 4O.*m* compounds). In spite of comparable exponent values at the IS_A transition with the values across the IN transition for some members of the 4O.*m* series [37], it may be noticed that a large non-linear thermal region is involved with the IS_A transition, i.e. on the time scale and in the environment of identical thermodynamic conditions, the growth of simultaneous orientational and translational orders is much stronger which ratifies our visual observation.

3.2. Smectic A–smectic C transition

The smectic A–smectic C transition is accompanied by a small density jump and a small volume expansion coefficient (0.01 and $12.04 \times 10^{-4}^\circ\text{C}^{-1}$ for 9O.6 and 0.012 and $12.29 \times 10^{-4}^\circ\text{C}^{-1}$ for 9O.8, respectively) indicating the second order nature of the transition in agreement with the reported results for $n\text{O}.m$ and other compounds. An increase of the equilibrium density value for the smectic C phase ($d\rho/dt = 10.6 \times 10^{-4}^\circ\text{C}^{-1}$ for 9O.6 and $11.85 \times 10^{-4}^\circ\text{C}^{-1}$ for 9O.8) from the preceding S_A phase indicates a closely packed, tilted molecular structure highly ordered in the S_C phase. It is apparent from the density results that the observed density jump ($\Delta\rho_{\text{obs}}$) is almost identical to the estimated density jump ($\Delta\rho_{\text{est}}$) ($\Delta\rho_{\text{est}}$ is obtained by the linear extrapolation of ρ_{obs} values about $T_{\text{S}_A\text{S}_C}$ for $\pm 0.5^\circ\text{C}$), supporting the continuous second

Table 3. The α_{eff} values estimated from density results in the fluctuation dominated non-linear regions (FDNLR) for the 9O.*m* ($m=4, 5, 6$ and 8) series at the isotropic–smectic A transition.

Property	9O.4		9O.5		9O.6		9O.8	
	I	S_A	I	S_A	I	S_A	I	S_A
FDNLR/ $^\circ\text{C}$	1.1	0.5	0.6	0.5	1.3	0.8	1.5	0.8
α_{eff}	0.64	0.62	0.50	0.48	0.62	0.41	0.65	0.52
$\kappa^2 \times 10^{-5}$	0.67	1.65	5.21	2.25	4.27	2.08	5.92	1.62

Table 4. The α_{eff} values estimated from density results in the fluctuation dominated non-linear regions for first order NS_A and IN transitions of some $n\text{O}.m$ compounds.

Transition	Compound	α_{eff} $T > T_C$	FDNLR/ $^{\circ}\text{C}$
IN	4O.4	0.776	0.3
	4O.5	0.742	0.5
	4O.6	0.701	0.6
	4O.7	0.826	0.5
	4O.8	0.794	0.4
	4O.9	0.523	0.5
	4O.10	0.622	0.2
	4O.12	0.785	0.3
IN	6O.3	0.342	0.6
	6O.4	0.620	0.3
	6O.5	0.671	0.4
	6O.6	0.259	0.4
	6O.7	0.530	0.3
	6O.8	0.510	0.4
NS_A	6O.3	0.310	0.2
	6O.4	0.160	0.5
	6O.5	0.410	0.4
	6O.6	0.510	0.3
	6O.7	0.300	0.2
	6O.8	0.330	0.4

order nature of the S_AS_C transition in both compounds. However, it may be recalled that weak first order S_AS_C transitions have been reported in the literature underlining the influence of the smectic A thermal range in the vicinity of the NS_AS_C multicritical point and the S_AS_C critical point. Here, the S_A range in either compound is much larger than those previously reported and the introduction of a fluctuation mediated, weak first order AC transition is not conceivable.

3.3. Smectic C–smectic F transition

The density jumps (1.48 per cent and 2.41 per cent) and volume expansion coefficients ($72.5 \times 10^{-3} \text{C}^{-1}$ and $66.7 \times 10^{-3} \text{C}^{-1}$) for 9O.6 and 9O.8, respectively, characterize the smectic C–smectic F transition as first order. The growth of smectic F hexagonal nets and their swift distortion for small changes in temperature below the S_AS_C transition are pronounced with fluctuation dominated non-linear regions of 0.2°C for 9O.6 and 2°C for 9O.8 reflecting the large density discontinuity. The pretransitional effects are observed to last for large thermal ranges ranging from 1.0°C for 9O.6 and 2.1°C for 9O.8 in the smectic F phase below the S_CS_F transition. The estimated pressure dependence of the S_CS_F transition temperatures from the Clausius–Clapeyron equation (dT_i/dP), using DSC and density results are 51.5 K kbar^{-1} for 9O.6 and 96.9 K kbar^{-1} for 9O.8. The value for 9O.8 is found to be higher than the reported values for 5O.5 [38] and TBDA [15] whilst that for 9O.6 agrees reasonably well. The pressure dependence (43 K kbar^{-1}) for the S_CI transition in TBDA is found to be smaller than the S_CS_F transition which differs in the formation of the direction of tilt of the hexagonal smectic F net. Furthermore, the dT_i/dP values for the S_CS_F transition in

these compounds are found to be higher than the reported [37] values for the $S_A S_B$ transition in $nO.m$ compounds indicating a steeper $S_C S_F$ interface underlining the more susceptible swift distortions than the crystallising hexagonal fluctuations at the $S_A S_B$ transition interface. However, the varying dT_i/dP values might be due to the different experimental conditions used to determine the density and the enthalpy values. The small but noticeable increase of density in the smectic F phase coming from the smectic C phase is due to the greater positional order of the molecules in the hexagons of the smectic F phase.

3.4. Smectic F–smectic G transition

In the literature very few compounds are known to exhibit the $S_F S_G$ transition. The $nO.m$ series 5O.5, 5O.6, 7O.6, 9O.4, and 10O.6 in addition to 9O.6 and 9O.8 exhibit this transition [32]. The reported [34] transition enthalpies associated with the $S_F S_G$ transition are small. The $S_F S_G$ transition is accompanied by small density jumps ($\Delta\rho/\rho \times 100 = 0.015$ for 9O.6 and 0.003 for 9O.8) and relatively small volume expansion coefficient values ($15.06 \times 10^{-4} \text{C}^{-1}$ for 9O.6 and $30.22 \times 10^{-4} \text{C}^{-1}$ for 9O.8) indicating the continuous second order character for the $S_F S_G$ transition. It is noticed that the density jumps observed for the $S_C S_F$ transition are less than the reported [33] values for the $S_F S_G$ transition in the compound 7O.6. However, no density jump is observed [38] in the compound 5O.6 for this transition. The higher density slope in the smectic G phase indicates the densely packed and orthogonally arranged hexagons that suffer cooperative librational degrees of freedom in the smectic F phase while growing into a smectic G phase.

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