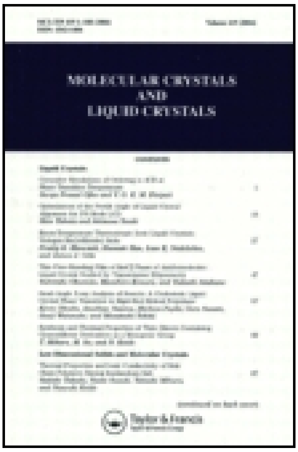

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A Smectic F to Isotropic Liquid Phase Transition in *n*O.*m* Compounds

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The search for the hexatic smectic F phase in *N*(*p*-n-alkoxybenzylidene)-*p*-n-alkylanilines, has revealed that *N*(*p*-n-decyloxybenzylidene)-*p*-n-tetradecylaniline, 10O.14, exhibits a direct isotropic liquid to smectic F phase transition with a phase sequence variant FG, while both the compounds 10O.12 and 10O.16 exhibit the AFG phase sequence variant. This is the first observation of an isotropic liquid to smectic F phase transition in nO.*m* compounds.

INTRODUCTION

The smectic F phase is hexatic in nature. The hexagonally close packed molecules are tilted towards the edge of the matrix and the inplane positional correlation of the molecules is short range. However, this smectic phase possesses long range bond orientational ordering. The formation of the smectic F phase on direct cooling of an isotropic liquid phase was reported[1] first for esters of the type *p*-n-alkynoloxy *p*-n-octyloxybiphenyl with n = 9, 11, 13. The search for the smectic F phase in the *N*(*p*-n-alkoxybenzylidene)-*p*-n-alkylanilines, *n*O.*m*, series amongst homologues with $n = 10$ and $m = 12$, 14 and 16, has shown that 10O.14 is the first compound in the series to exhibit formation of the smectic F phase directly on cooling the isotropic liquid. The other two compounds, with $m = 12$ and 16 also exhibit the smectic F phase. The experimental results of thermal microscopy, DSC and miscibility studies of the compounds are presented.

Preliminary studies by thermal microscopy (TM) on *N*(*p*-n-decyloxybenzylidene)-*p*-n-alkylanilines with $m = 12$, 14 and 16 revealed that the compounds with $m = 12$ and 16 exhibit three enantiotropic liquid crystalline phases, while the compound *N*(*p*-n-decyloxybenzylidene)-*p*-n-tetradecylaniline, 10O.14, exhibits only two enantiotropic smectic phases. The transition temperatures are presented in Table I. Differential scanning calorimetry (DSC) confirmed these results and the enthalpy values are presented in Table I.

TABLE I

Transition temperatures (°C) from TM and DSC, and enthalpy values (Joules/mole)

	S_1	S_2	S_3	S_4		
10O.*m* I	A	C	F	G	K	Ref.
$m = 6$	87.5	78.5	78.0	74.0	mp < 20[a]	3
	86.2	—	76.9	74.3	—[b]	
	8591	—	4946	—	—[c]	
$m = 8$	89.8	84.8	82.2	81.8	43.0	3
	89.1	—	82.3	—	42.3	
	8385	—	5251	—	42635	
$m = 10$	89.0	86.1	85.0	84.8	55.0	3
	88.8	—	84.1	—	53.0	
	9289	—	5770	—	49640	
$m = 12$	85.0		83.0	70.5	57.5	
	86.7		83.7	—	56.1	
	5607		3326	—	34443	
$m = 14$			83.5	82.2	68.5	
			81.5		64.9	
			15400		52350	
$m = 16$	85.8		84.5	83.5	58.0	
	84.7		83.3	—	56.6	
	7400		4364	—	24680	

[a] Values from TM (°C).
[b] Values from DSC (°C).
[c] Enthalpy (ΔH) in Joules/mole.

THERMAL MICROSCOPIC STUDIES

a) 10O.12 and 10O.16

On cooling from the isotropic liquid, a mesomorphic phase S_1 appeared in the form of bâtonnets at 85.0°C for 10O.12 and at 85.8°C for 10O.16, and these coalesced into a focal-conic fan texture. This phase also exhibited the homeotropic or pseudo-isotropic texture indicating that the phase is orthogonal. These observations indicate that the phase may be smectic A. Further cooling produced the second phase S_3 which exhibited both broken focal conic fan and schlieren-mosaic textures formed from the focal conic fan and the homeotropic textures of the S_1 phase, at 83.0°C and 84.5°C respectively. These textures are similar to those observed for the compounds 9O.4[2], 9O.6[3] and 9O.8[3]. Therefore the S_3 phase may be smectic F. Subsequent cooling leads to a S_4 phase which forms with slight changes in the texture. The breakages in the S_3 texture changed to the form of large dark patches and these breakages or patches have a more chunky appearance; the texture is similar to that observed for the smectic G phase.[2]

b) 10O.14

On cooling the isotropic liquid, two distinct types of optical texture, *viz.*, the focal conic texture with the appearance of spherulites and lancets, and the droplet texture

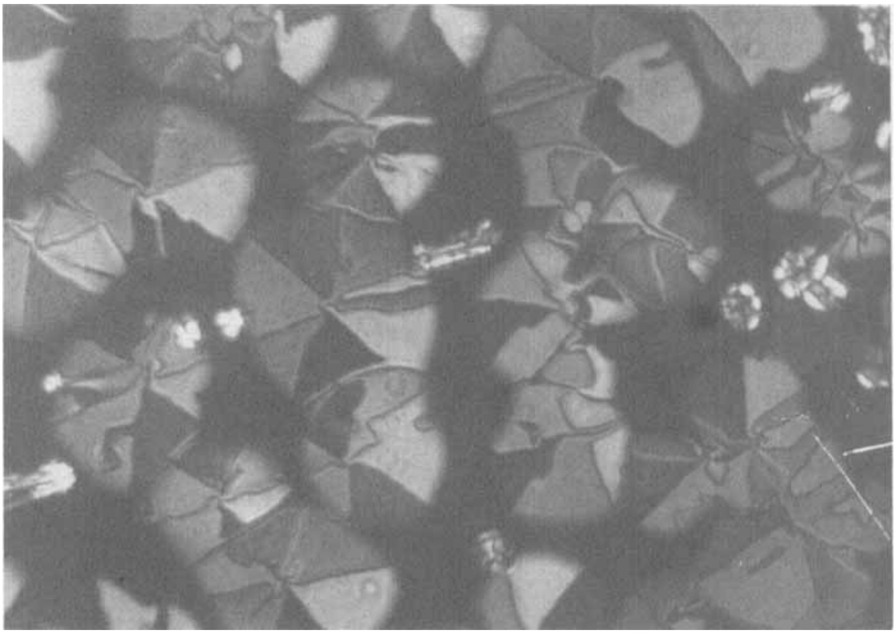

PLATE 1 See Color Plate III.

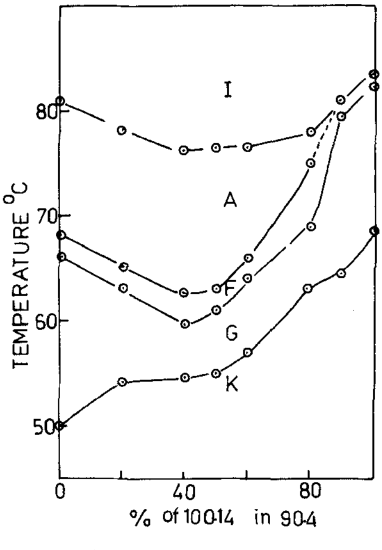

FIGURE 1 Miscibility diagram for 9O.4 + 10O.14.

leading to the normal mosaic pattern of the smectic F phase are observed at 83.5°C. The phase, as it separates out from the isotropic liquid, exhibits the texture shown in Plate 1, with spherical droplets which have a hexagonal cross of optical discontinuity centered in them. The observed textures are similar to those reported[1] for esters of the type *p*-n-alkanoloxy *p*-n-octyloxybiphenyl which exhibited the formation of the smectic F phase directly on cooling the isotropic liquid. These textural observations suggest that this phase (S_3) in 10O.14 may be smectic F. On further cooling, the S_3 phase was transformed into the S_4 phase, with a broken or striped fan texture, at 82.2°C, characterising the phase as possibly smectic G.

The smectic phases S_1, S_3 and S_4 for the compounds 10O.12 and 10O.16 and the smectic S_3 and S_4 phases of 10O.14 were further characterised by miscibility studies with *N*(*p*-n-nonyloxybenzylidene)-*p*-n-butylaniline, 9O.4, of the *n*O.*m* series, which exhibits the AFG phase sequence variant.

The smectic A, F and G phases are continuously miscible in the binary systems 9O.4 + 10O.12 and 9O.4 + 10O.16, confirming the phase variant for both compounds as AFG. The smectic F and G phases for 9O.4 are comiscible with the smectic phases of 10O.14. By increase in the concentration of the test sample 10O.14 in the binary mixture to about 90%, a direct isotropic liquid to smectic F transition is observed. The phase diagram for 9O.4 + 10O.14 is shown in Figure 1.

The miscibility studies therefore confirm that the variant FG is exhibited by the compound 10O.14.

CONCLUSIONS

Our experimental results on the 10O.*m* series confirm the presence of the smectic F phase for the compounds with $m = 6, 8, 10, 12, 14$ and 16 and that they show the phase sequence variants detailed below:

$m = 6, 8$ and 10:	10O.6, 10O.8 and 10O.10	ACFG,
$m = 12$ and 16:	10O.12 and 10O.16	AFG
$m = 14$:	10O.14	FG

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