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A New Series 4(4'-Cyanobenzoyloxy) Benzylidene-4-Alkoxyaniline with a Smectic Phase Made of Ribbons

Nguyen Huu Tinh^a, F. Hardouin^a & C. Destrade^a

^a Centre de Recherche Paul Pazcal, Domaine Universitaire, 33405, TALENCE, Cédex, FRANCE

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A NEW SERIES 4(4'-CYANOBENZOYLOXY) BENZYLIDENE-4-ALKOXYANILINE WITH A SMECTIC PHASE MADE OF RIBBONS

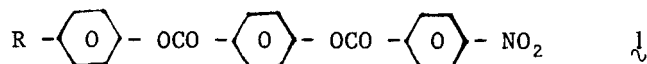
NGUYEN HUU TINH, F. HARDOUIN, C. DESTRADE

Centre de Recherche Paul Pazcal,
 Domaine Universitaire, 33405 TALENCE Cédex, FRANCE

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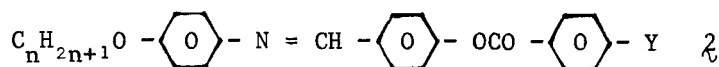
Abstract : A homologous series of 4-(4'-cyanobenzoyl-oxy)benzylidene-4-alkoxyaniline have been synthesized. Four derivatives with a long alkoxy chain from $C_8H_{17}O-$ to $C_{11}H_{23}O-$ exhibit a smectic phase made of ribbons (S_C) recently discovered.

Introduction : The new smectic phase made of ribbons S_C , corresponding to a two-dimensional oblique lattice with a short range liquid-like order, was recently discovered in the nitro series^{1,2}.



with $R = C_8H_{17}-$, $C_9H_{19}O-$ or $C_{10}H_{21}O-$

We present new homologous series with cyano compounds which exhibit the same behavior



with $n = 4 \rightarrow 12$

y : a) NO_2
 b) CN

Results and discussion : The substances were synthesized by condensation of 4(4'-cyano or nitrobenzoyloxy) benzaldehyde and 4-alkoxyaniline in ethanolic solution. They were purified by repeated recrystallization from ethanol. Phase transitions were studied both by polarizing microscopy (equipped with a Mettler PF5 heating stage) and Differential Scanning Calorimetry (Dupont 990). The transition temperatures and types of mesophases are given in Table.

Table : Transition temperatures ($^{\circ}\text{C}$) of compounds 2

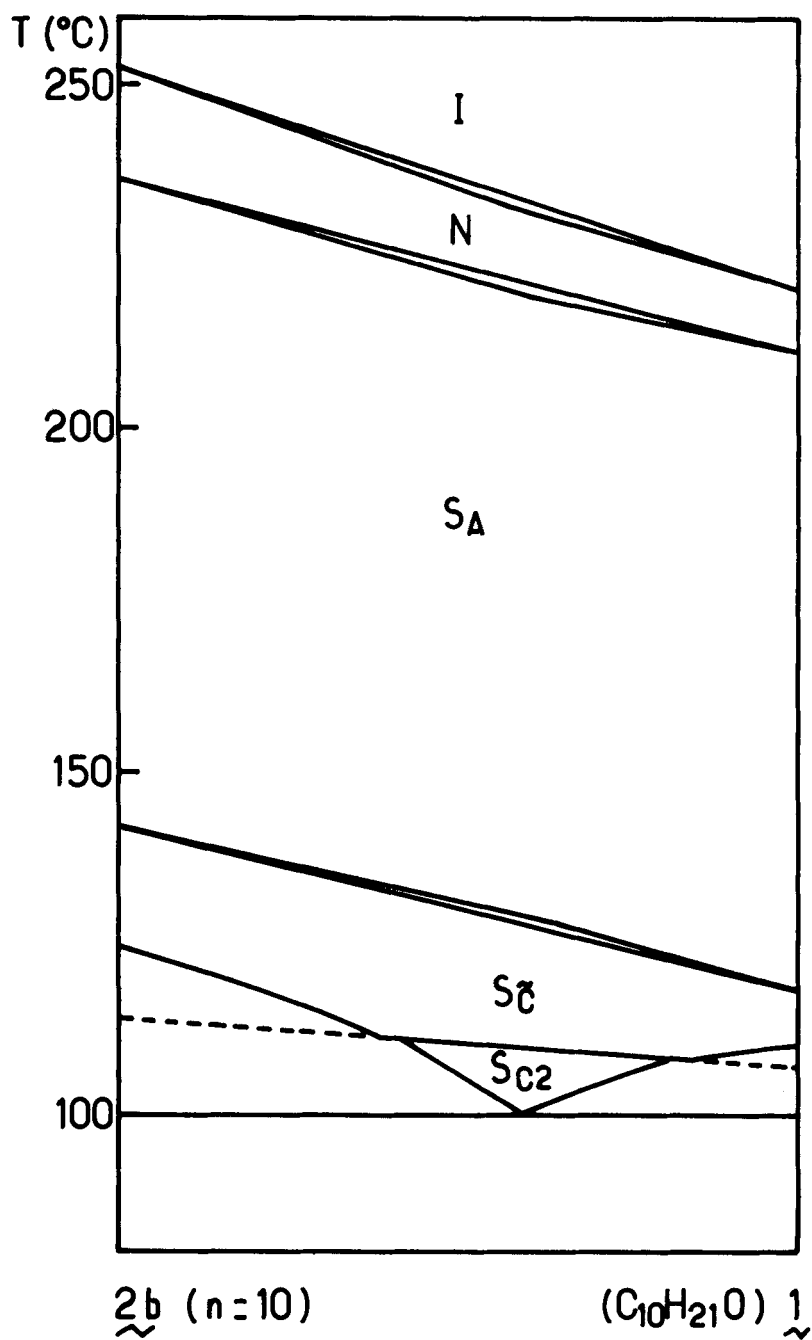
n	Y	K	S_{C_2}	$S_{\tilde{C}}$	S_A	N	I
4	NO_2	. 140	-	-	. 154	. 281	.
5	"	. 123	-	-	. 165	. 267	.
6	"	. 124.5	-	-	. 173	. 263	.
7	"	. 114	-	-	. 180	. 254	.
8	"	. 121	-	-	. 196	. 252	.
9	"	. 109	-	-	. 227	. 246	.
10	"	. 112	-	-	. 235	. 242	.
11	"	. 110	-	-	. 239	. 240	.
12	"	. 112	-	-	. 237	-	.
4	CN	. 128	-	-	. 179	. 296	.
5	"	. 122.5	-	-	. 181	. 287	.
6	"	. 120	-	-	. 181.5	. 279	.
7	"	. 114	-	(.)*	. 178	. 271	.
8	"	. 122	-	(.119)	. 172	. 262	.
9	"	. 125	-	.154	. 212	. 257	.
10	"	. 124	(.114)	.142	. 236	. 252	.
11	"	. 126	(.125)	.131	. 239	. 246	.
12	"	. 124.5	.129	-	. 240.5	. 242	.

K : crystal phase ; S_{C_2} : bilayered smectic C phase ;
 $S_{\tilde{C}}$: smectic phase made of ribbons ; S_A : smectic A phase ;
 N : nematic phase ; I : isotropic phase ; () : monotropic transition ; . : the phase exists ; - : the phase is not ob-

served ; * observed with rapidly cooling.

At first, we must point out that the derivatives of 2a,b (series with three phenyl rings and a polar end group) do not exhibit the reentrant phenomenon as observed in other series. If in the dibenzoate series 1 the S_C was only observed with the nitrocompounds and not observed with the cyano derivatives. On the contrary, this phase only appears in the cyano series 2b. The nitro derivatives 2a exhibit S_A and N phases. For the cyano derivatives 2b, the three first compounds ($n = 4,5,6$) also give S_A and N phases. But, in addition to S_A and N phases, the heptyloxy derivative presents a S_C phase if it is rapidly cooled. This S_C phase is enantiotropic from the nonyloxy and disappears from the dodecyloxy derivative. The bilayered S_{C_2} is observed from the decyloxy derivative. This compound exhibits the tetramorphism N S_A S_C S_{C_2} ^{1,2}. On cooling the isotropic liquid one can observe the nematic phase with a schlieren texture. Below this nematic phase the smectic A phase with focal conic and homeotropic textures appears. On further cooling, one can observe the S_C with a texture formed by polydomains in the precedent homeotropic domain and a mosaic texture. On cooling from this S_C phase, a schlieren or fan shaped texture is observed.

At first the identification of N S_A S_C S_{C_2} sequence of the decyloxy derivative of 2b has been performed by the miscibility method with same sequence of the decyloxy of 1 (Fig. 1). This sequence is corroborated by X-ray analysis of the undecyloxy derivative of 2b. Cooling down from the nematic phase in a magnetic field, the aligned sample provides a partially bilayer S_{A_d} ¹ phase with a layer spacing $d \approx 51$ Å and with an incommensurated diffuse spot corresponding approximatively to the molecular length (≈ 30 Å)³. Then a two-dimensional oblique lattice appears which corresponds to a S_C phase as elsewhere detailed in the case of nonyloxy derivative of series 1². Although the sample is not well oriented an unidimensional modulation is observed again a lower temperature. In this last smectic phase the first and second order layering reflections have similar intensities and $d \approx 56$ Å. Thus taking into account the microscopic observations of schlieren textures and by analogy with bilayer S_{A_2} . We claim that this phase is a bilayer S_{C_2} .



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