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Reentrant nematic phase with the new pentamorphism $n_{S_a} S_c n_{re} S_{are}$ in a mixture

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REENTRANT NEMATIC PHASE WITH THE NEW PENTAMORPHISM $N_{SA} S_C N_{re} S_{Are}$ IN A MIXTURE

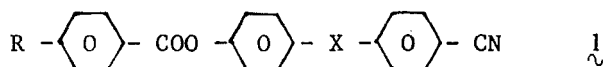
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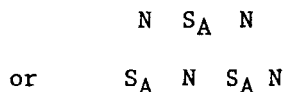
Abstract : Three homologues of the new series 4 - cyano-
 benzylidene - 4' - [4''-alkoxybenzoyloxy] aniline are
 presented. One of them exhibits a reentrant nematic and
 a reentrant smectic A phases. A diagram of state with
 4 - cyanobenzylidene - 4' - [4''-decyloxybenzoyloxy] ani-
 line and 4 - [4''-decyloxybenzoyloxy] benzylidene - 4' -
 cyanoaniline has been studied. In the system studied,
 the following new phase sequence was found with decrea-
 sing temperature $N_{SA} S_C N_{SA}$.

Introduction : From the discovery of the reentrant ne-
 matic phase by P.E. Cladis^{1,2}, at first in binary mixture,
 then for a pure compound at high pressure^{3,4}, at last by
 F. Hardouin et al.^{5,6} in a pure substance at atmospheric
 pressure, numerous synthesis have been performed and a lot
 of new homologous series have been published. These substan-
 ces belong to the general formula⁶⁻¹⁷



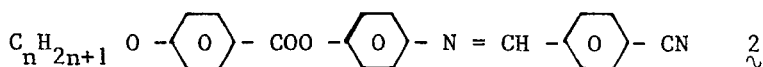
with $X = - \text{CH} = \text{CH} -$, $- \text{C} \equiv \text{C} -$, single bond, $- \text{N} = \text{N} -$,
 $- \text{CH} = \text{N} -$, $- \text{COO} -$, $- \text{OCO} -$

More often than not the reentrant phenomenon was obser-
 ved with the aliphatic chain varying from C_8 to C_{10} and the
 observed sequences are with increasing temperature :



Moreover Weissflog et al.¹¹, in the case of 1 with $X = -CH=N-$, reported another sequence : $N \sim S_C \sim S_A N$ with the decyloxy derivative. At last, Pelzl et al.¹⁷ in recent paper reported the new sequence $S_C N S_A N$ in a binary mixture. We present in this paper three homologues of the new series : 4 - cyanobenzylidene - 4' - [4'' - alkoxybenzoyloxy] aniline $\tilde{2}$. One of them exhibits a reentrant nematic and a reentrant S_A phases. In the diagram of state between 1 and 4 - cyanobenzylidene - 4' - [4'' - decyloxybenzoyloxy] aniline the following new phase sequence was found with decreasing temperature $N S_A S_C N S_A$.

Results and discussion : The substances were synthesized by reaction between 4 - alkoxybenzoyl chloride with suitable phenate. They were purified by recrystallization from ethanol. The three studied derivatives are



with $n = 10 \rightarrow 12$

The mesophases of these compounds, listed in the following Table were examined by differential scanning calorimetry (Dupont 990) and by observation with a polarizing microscope equipped with a programmable heating stage (Mettler FP 5).

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

n	K	S_C	S_A	N	S_A	N	I
10	. 104	-	. (79)	. (94)	. 245	. 254	.
11	. 88	. (79)	-	-	. 247	. 249	.
12	. 91	. (70)	-	-	. 247	-	.

K : crystal phase ; S_A , S_C : smectic A, C phases ; N : nematic phase ; I : isotropic phase ; . : the phase exists ; - : the phase is not observed ; () : monotropic transition.

The compound $n = 10$ exhibits the tetramorphism : $N S_A N S_A$. On cooling the isotropic liquid of this compound, 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 another schlieren or paramorphic fan shaped texture is observed : on

cooling this reentrant nematic phase, the SA phase shows a focal conic texture. The reentrant nematic phase disappears in the two derivatives n = 11 and 12 but the monotropic SC phase is observed.

At first the identification of the N SA N SA sequence of the decyloxy derivative has been performed by the miscibility method with the same sequence of the well known nonyloxy derivative of 1 with $X = CH = CH^7$ (Fig. 1). The SC phase of the undecyloxy derivative was miscible with those of 4 - [4'' - decyloxy cinnamoyloxy] benzylidene - 4' - cyanoaniline [K 100 (N 70) (SC 87) SA 257 N 267 I]¹⁸ (Fig. 2).

The diagram of state of binary system between the decyloxy derivative of 1 ($X = CH = N$) and the decyloxy derivative of 2 has been studied by means of the contact method and by the investigations of singular concentrations. Fig. 3 shows the following new phase sequence with decreasing temperature : N SA SC N SA with two concentrations (68.7 mol % 2 and 31.3 mol % 1 and 69.2 mol % 2 and 30.8 mol % 1) ; for example, with the first one, the transition temperatures are: K 98.5 (SA 61.2) (N 86.5) (SC 87.5) SA 242.5 N 251.5 I. The new sequence N SA SC N SA found in mixtures of these new series shows another example of the great variance of reentrant polymorphism discovered or to be discovered with strong polar molecules.

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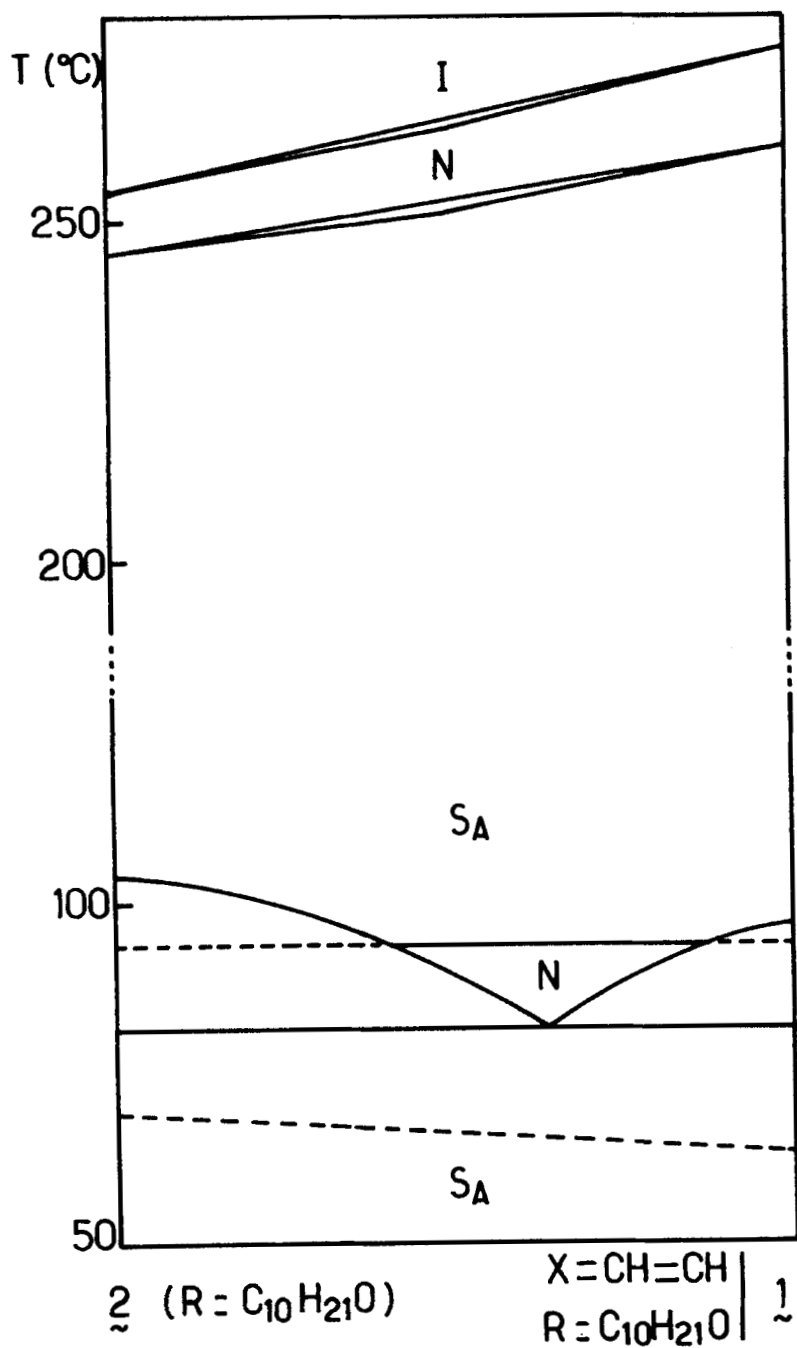


Figure 1

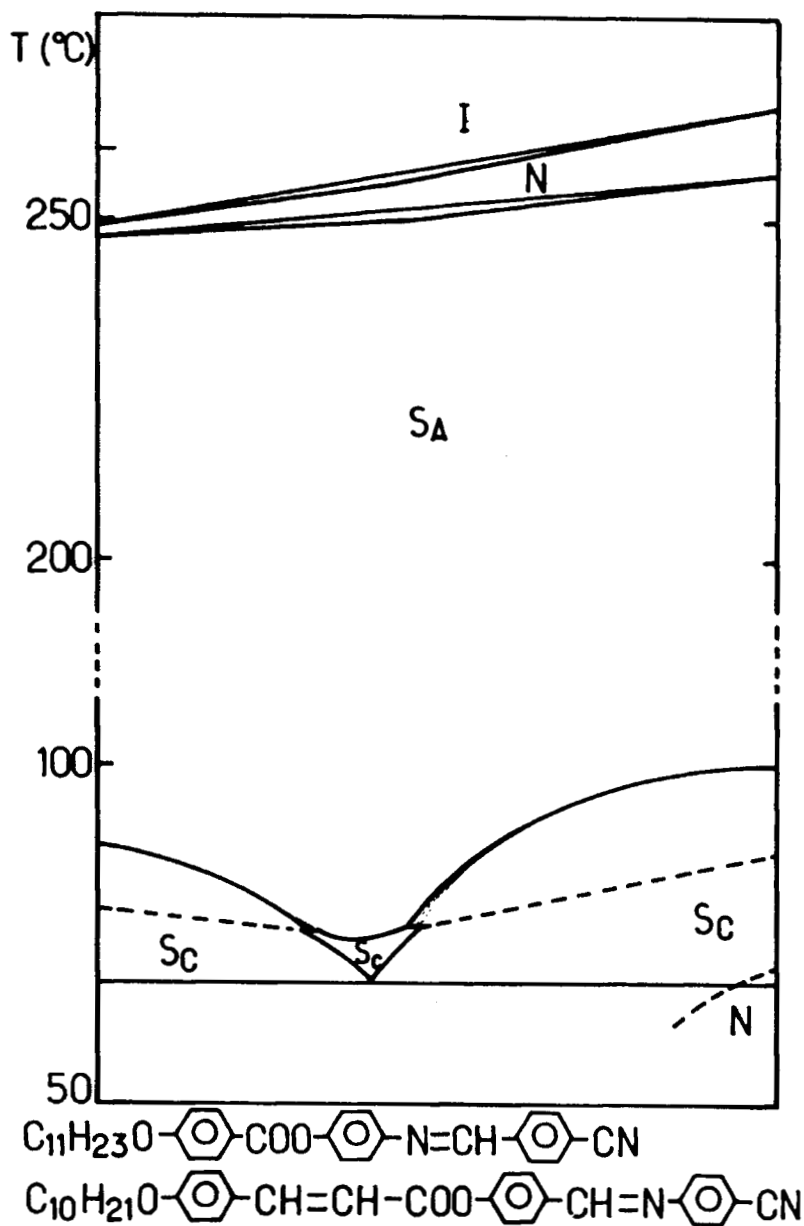


Figure 2

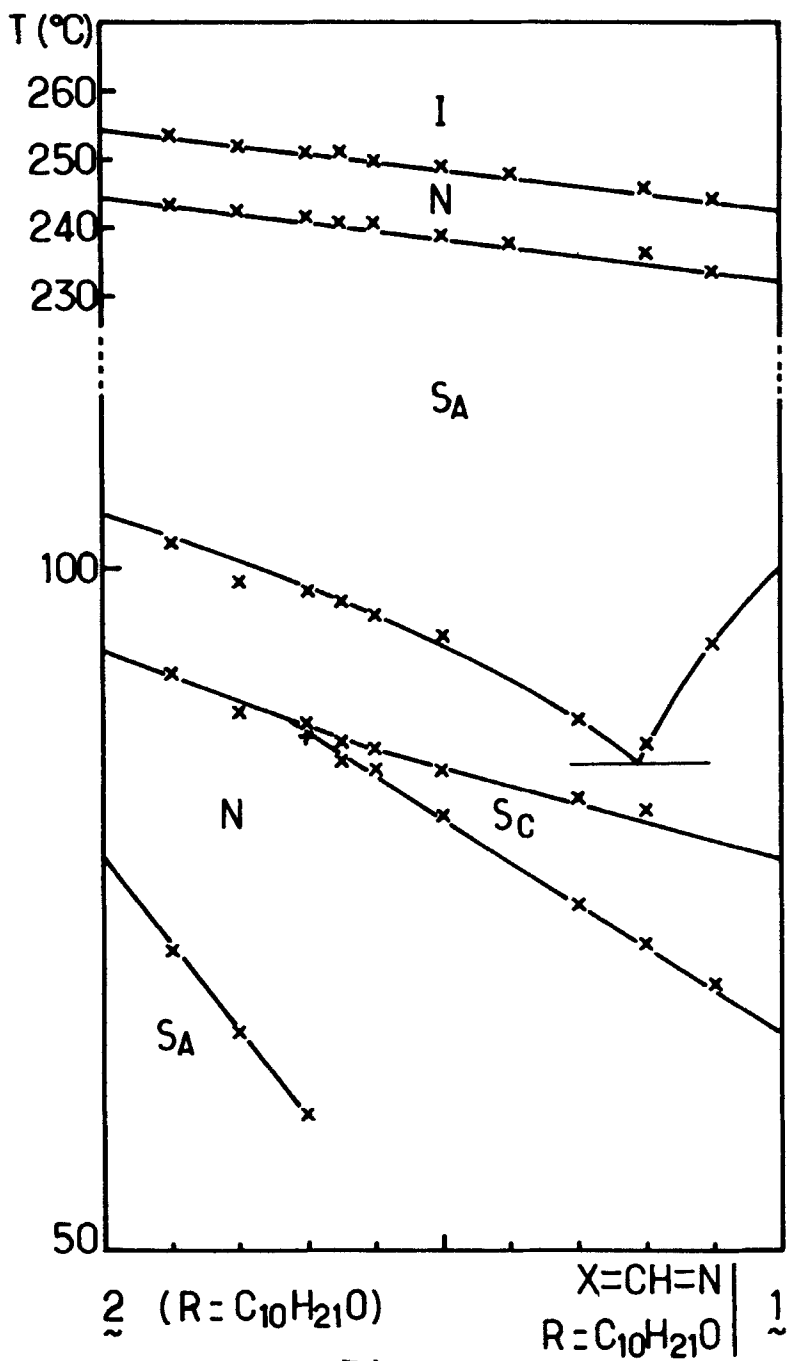


Figure 3

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