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## A New Liquid Crystalline Substance Exhibiting A Reextrant Nematic Phase and Two Low-Temperature Smectic Phases

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A NEW LIQUID CRYSTALLINE SUBSTANCE EXHIBITING A REENTRANT NEMATIC PHASE AND TWO LOW-TEMPE-RATURE SMECTIC PHASES

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Abstract: A few members of the homologous 4-n-alkyloxy-benzylidene-amino-4'-cyanobiphenyls were synthesized. The C<sub>7</sub>-compound exhibits a reentrant nematic phase and at lower temperatures smectic B and smectic E phases.

We have synthesized five members of the homologous 4-n-alkyloxy-benzylidene-amino-4'-cyanobiphe-nyls in analogy to the 4-cyanobiphenyl 4'-n-alkyloxy-benzoates <sup>1</sup>. The substances were obtained by condensation of 4-n-alkyloxybenzaldehydes with 4-ami-no-4'-cyanobiphenyl. The products were purified by recrystallization from ethanol or amylalcohol.

The transition temperatures determined with a polarizing microscope are given in table 1.

The  $C_5$ -compound shows  $S_E$ - $S_B$ -N polymorphism.

TABLE 1  $C_nH_{2n+1}O-O-CH=N-O-CN$ 

n	cr	S	E		$s_{\mathrm{B}}$		Nre		$S_{\mathbf{A}}$		N		is.
5	•	119	•	(76)	•		***		_	-	299		•
6	•	92	-		•	111	_		-		280		
7	•	73	•	95	•	111	•	140	•	212	•	273	•
8	•	68	•	100	-		-		•	243	•	267	
9	•	76	•	99	_		_		•	249	•	258	<u>.</u>

cr : solid crystal

 $S_A$ ,  $S_B$ ,  $S_E$ : smectic A, B or C

N : nematic

N<sub>re</sub> : reentrant nematic phases.

is. : isotropic

The numbers are the transition temperatures (°C). Brackets denote monotropic

In the next homologue ( ${\rm C_6}$ ) we could not detect a  ${\rm S_E}$  phase, although the  ${\rm S_B}$  phase was supercooled to room temperature. In the  ${\rm C_7}$ -compound, below the temperature range of the reentrant nematic ( ${\rm N_{re}}$ ) phase, two smectic low temperature phases -  ${\rm S_E}$  and  ${\rm S_B}$  - could be observed. In the  ${\rm C_8}$  and  ${\rm C_9}$  homologues, the  ${\rm S_B}$  phase and the  ${\rm N_{re}}$  phase disappear and a polymorphism  ${\rm S_E}$ ,  ${\rm S_A}$ ,  ${\rm N}$  occurs.

In table 2 the transition enthalpies of the  ${\tt C}_7$  derivative measured with a differential scann-

ing calorimeter (DSC 2, Perkin-Elmer) are listed. It is remarkable that the enthalpy for the transition  $N_{re} \rightarrow S_A$  is very low compared with the enthalpy of the  $S_A \rightarrow N$  transition or with the corresponding values of other substances with reentrant nematic phases 2,3.

TABLE 2 Transition Enthalpies (J mol<sup>-1</sup>) of 4-n-Heptyloxy-benzylidene-amino-4'-cyanobiphenyl

transit	tion	transition temperature,	transition <u>AH/J mol-1</u> temperature/ <sup>O</sup> C				
cr I -	- cr	<i>-</i>	23 500				
cr II -	- S <sub>E</sub>	73	4 500				
s <sub>E</sub> -	- s <sub>B</sub>	95	395				
$s_{\mathrm{B}}$ .	- N <sub>re</sub>	111	3 400				
$^{ exttt{N}}_{ exttt{re}}$	- S <sub>A</sub>	140	4 - 5				
S <sub>A</sub>	- N	212	37				
N -	- is	273	1 000				

On microscopic observation the  $\mathbf{S}_{\mathbf{B}}$  phase appears as a homeotropic texture, a fan-shaped texture or a typical mosaic texture depending on the experimental conditions. The  $\mathbf{S}_{\mathbf{E}}$  phase often occurs as a fan-shaped texture or a paramorphic mosaic texture.

The identification of the low temperature smectic phases was carried out by miscibility in-

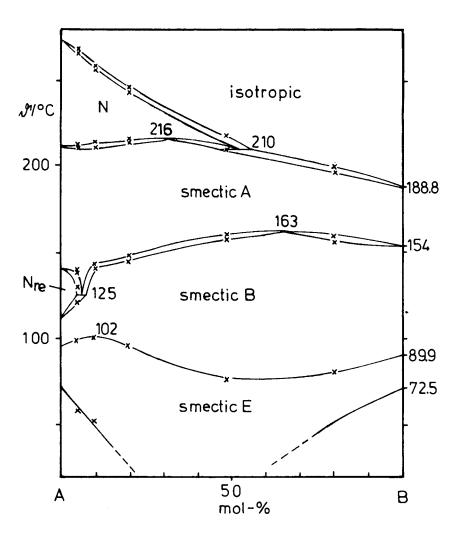
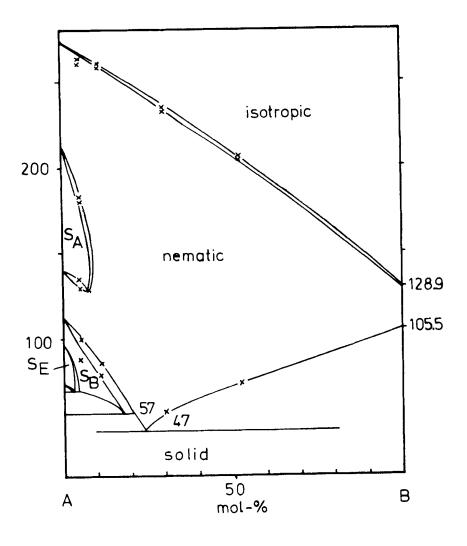


FIGURE 1



A: 
$$H_{15}C_{7}O \longleftrightarrow CH = N \longleftrightarrow CO \longleftrightarrow CN$$
B:  $H_{5}C_{2}O \longleftrightarrow CH = N \longleftrightarrow CN$ 

FIGURE 2

vestigations with suitable reference substances. The diagrams of state were determined by the contact method, 4 completed by the study of singular concentrations.

Figure 1 shows that the two smectic low temperature phases of 4-n-heptyloxy-benzylidene-amino-4'-cyanobiphenyl are completely miscible with the  $S_E$  and  $S_B$  phases of n-decyl 4-[4-phenyl-benzylidene-amino]-cinnamate  $^5$ . The  $N_{\rm re}$  phase exists only in a small concentration range. The eutectic temperature and also the melting points of the mixtures could not be determined because the samples did not crystallize.

As shown in figure 2 the nematic phase of ethoxybenzylidene-amino-4'-cyanoaniline is completely miscible with the nematic high-temperature phase as well as with the reentrant nematic phase.

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