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

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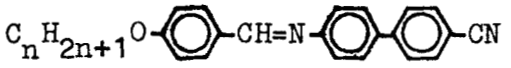
Abstract: A few members of the homologous 4-n-alkyloxy-benzylidene-amino-4'-cyanobiphenyls were synthesized. The C_7-compound exhibits a reentrant nematic phase and at lower temperatures smectic B and smectic E phases.

We have synthesized five members of the homologous 4-n-alkyloxy-benzylidene-amino-4'-cyanobiphenyls in analogy to the 4-cyanobiphenyl 4'-n-alkyloxybenzoates [1]. The substances were obtained by condensation of 4-n-alkyloxybenzaldehydes with 4-amino-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



n	cr	S_E	S_B	N_{re}	S_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	.

cr : solid crystal

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

N : nematic

N_{re} : reentrant nematic

is. : isotropic

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

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

In table 2 the transition enthalpies of the C_7 derivative measured with a differential scann-

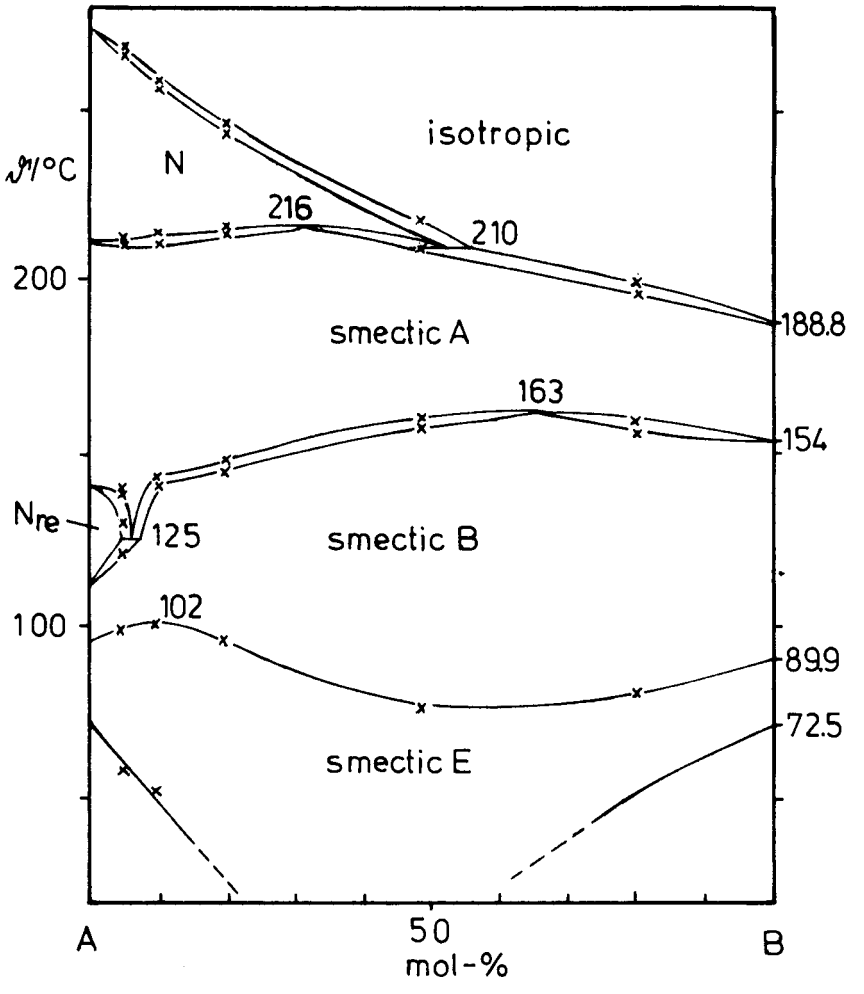
ing calorimeter (DSC 2, Perkin-Elmer) are listed. It is remarkable that the enthalpy for the transition $N_{re} \rightarrow S_A$ is very low compared with the enthalpy of the $S_A \rightarrow N$ transition or with the corresponding values of other substances with reentrant nematic phases [2,3].

TABLE 2 Transition Enthalpies (J mol^{-1}) of 4-n-Heptyloxy-benzylidene-amino-4'-cyanobiphenyl

transition	transition temperature/°C	ΔH/J mol^{-1}
cr I - cr II	65	23 500
cr II - S_E	73	4 500
S_E - S_B	95	395
S_B - N_{re}	111	3 400
N_{re} - S_A	140	4 - 5
S_A - N	212	37
N - is	273	1 000

On microscopic observation the S_B phase appears as a homeotropic texture, a fan-shaped texture or a typical mosaic texture depending on the experimental conditions. The S_E phase often occurs as a fan-shaped texture or a paramorphic mosaic texture.

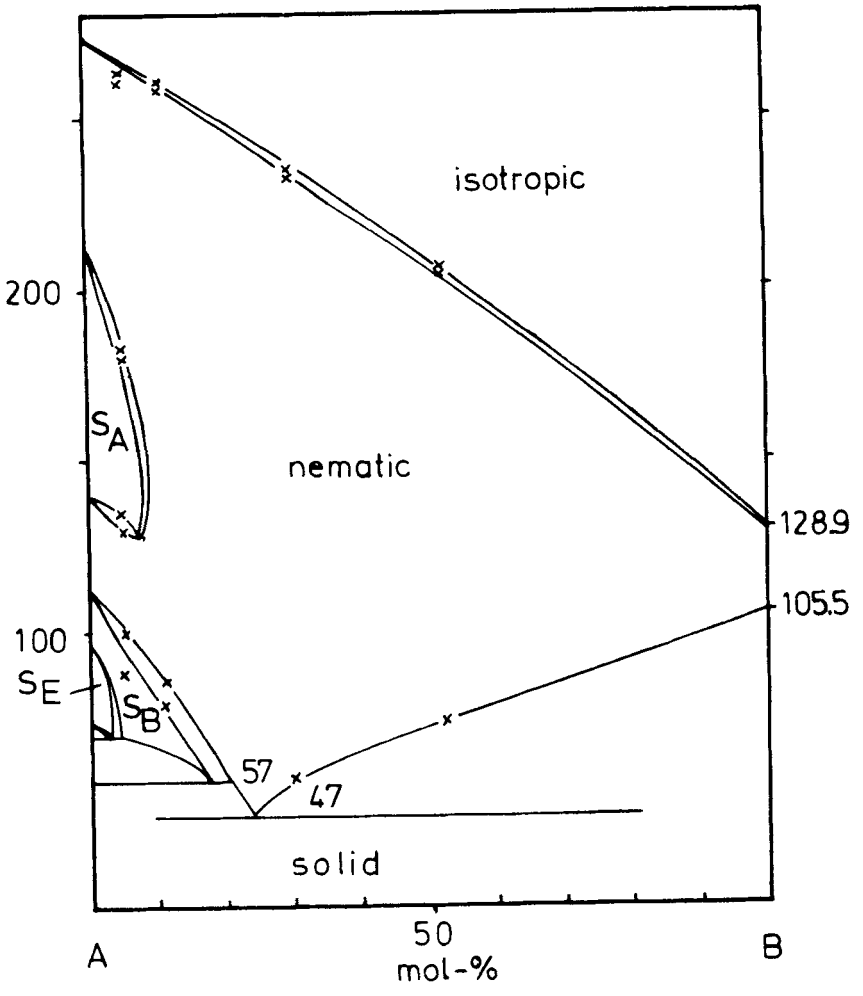
The identification of the low temperature smectic phases was carried out by miscibility in-



A: $H_{15}C_7O$-⟨⟩-CH=N-⟨⟩-⟨⟩-CN

B: ⟨⟩-⟨⟩-CH=N-⟨⟩-CH=CH-$COOC_{10}H_{21}$

FIGURE 1



A: $H_{15}C_7O$-⟨⟩-CH=N-⟨⟩-⟨⟩-CN

B: H_5C_2O-⟨⟩-CH=N-⟨⟩-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_{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 [6] is completely miscible with the nematic high-temperature phase as well as with the reentrant nematic phase.

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