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A Low Temperature Liquid Crystal Exhibiting Smectic Morphology

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I wish to report the discovery of a chemically homogeneous, stable, enantiotropic liquid crystal exhibiting smectic morphology at room temperature: 3-N-(4′-ethoxybenzilideneamino)-6-*n*-butylpyridine (EBBP).

The current surge of interest in liquid crystals is due to the electro-optic behavior of nematic materials and their use in optical displays.[1] The main advantages of these materials in such displays are extremely low power requirements at the " readout " stage, superior contrast in variable ambient light environments and low cost. Optimizing these and other important parameters has been the goal of a variety of recent investigations.[2] The desire to achieve a very low ($\ll 0\,^{\circ}C$) crystal → nematic transition while maintaining a substantial nematic temperature range is one of these goals. This has been achieved, to some degree, in mixtures[3] but no chemically homogeneous nematic below 15 °C or smectic below 40 °C has been reported.[4]

The desirability and practicality of a low temperature liquid crystal prompted the investigation of heterocyclic molecular systems. An initial departure from an all carbocyclic aromatic system utilized the following general structure:

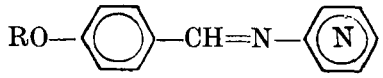
$$X—G—Y—G'—Z \qquad (1)$$

where Y may be —CH=N—, $—N{=}\overset{\uparrow}{N}—$, $—\overset{\|}{C}—$, etc.

X and Z may be carbonato, ester, alkoxy, alkyl, etc. The symbol G is used to indicate an n-membered aromatic ring. Either G and/or G' could be heteroaromatic with the heteroatom(s) occupying a

position α, β, or γ to the central linkage. The only report of a system approximating this series (1) is by Haller *et al.*[5]

The first of this series involved the following system:



(2)

It was during this investigation that the reported EBBP was discovered. EBBP was synthesized by mixing equimolar amounts of 3-amino-6-*n*-butylpyridine and *p*-anisaldehyde in toluene and refluxing about 3 hours. The water formed during the reaction was collected by azeotropic distillation. The structure was verified by ir/nmr and the purity was determined to be $> 99.5\%$ by GLC on two columns of different polarity. The intermediate 3-amino-6-butylpyridine was synthesized by a modification of an earlier procedure of Gruber.[6]

The mesomorphic phase transitions of EBBP were found to be[7]

$$\text{crystal} \underset{-10^\circ}{\overset{?}{\rightleftarrows}} \text{Sm}_{II} \underset{28^\circ}{\overset{\sim 29^\circ}{\rightleftarrows}} \text{Sm}_{I} \underset{48^\circ}{\overset{\sim 49^\circ}{\rightleftarrows}} \text{nematic} \underset{54.4^\circ}{\overset{\sim 54.5^\circ}{\rightleftarrows}} \text{isotropic}$$

by the use of an A0–20 series polarizing microscope equipped with a Mettler heating-cooling stage at magnification 65 × to 100 ×.

Carr and others[8] have reported studies of the physical properties of smectic liquid crystals including alignment owing to electric fields. These studies have necessarily required elevated temperatures because of the hitherto inaccessibility of a room temperature smectic material. EBBP, the first room temperature smectic to be reported to the author's knowledge, would obviate the need of elevated temperatures in future studies.

Work is continuing on series (2) and the completed study will be reported in a subsequent paper.

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7. The C→Sm_{11} transition was taken as that point where movement could be observed during cover slip adjustments (~10 °C); it is also possible that more than two smectic phases are present and a homeotropic nematic condition exists above 54.5 °C. A DTA study should help clarify these latter optical observations and will be discussed in a later paper.
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