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X-ray diffraction studies of the liquid crystal phases formed by certain 4'-*n*-alkylbiphenyl-4-yl 5-*n*-alkylthiophene-2-carboxylates

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X-ray diffraction studies of the liquid crystal phases formed by certain 4′-*n*-alkylbiphenyl-4-yl 5-*n*-alkylthiophene-2-carboxylates

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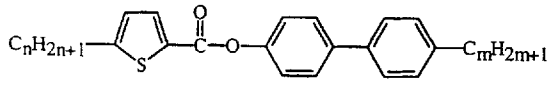
Twenty esters selected from the first ten homologues of each of the 4′-*n*-octylbiphenyl-, 4′-*n*-nonylbiphenyl- and 4′-*n*-decylbiphenyl-4-yl 5-*n*-alkylthiophene-2-carboxylates have been examined by X-ray diffraction techniques. These esters show extensive smectic polymorphism, but only tilted phases are observed, all with tilt angles of around 45°.

1. Introduction

The preparation and investigation by optical microscopy and DSC of the mesomorphic properties of ten members of each of three homologous series of 4′-*n*-alkylbiphenyl-4-yl 5-*n*-alkylthiophene-2-carboxylates, (**1**; $m=8,9$ and 10; $n=1$–10), has recently been reported [1]. The compounds show extensive smectic polymorphism which is limited to mesophases where the molecules are tilted with respect to the layer normals. In this communication we provide further confirmation of phase assignments using X-ray techniques.



(**1**)

2. Results and discussion

Structural information on all three series has been obtained by a combination of X-ray powder photographs and detailed measurements of the smectic layer spacing using an X-ray diffractometer. X-ray powder photographs from all homologues of this series have similar characteristics for a particular phase type. The S_C, S_I and S_F phases all give photographs of the expected appearance [2], a well defined inner ring and a diffuse outer ring, which becomes much sharper for the smectic crystal phases, G, J and K.

A typical plot of the smectic layer spacing as a function of temperature obtained from diffractometer results is shown for the octyl homologue of the decyl series, (**1**; $m=10$, $n=8$), in the figure. In all cases, the layer spacings are considerably less than the theoretical molecular length. Aligned photographs and diffractometer measurements from the structurally similar 4′-*n*-octyloxybiphenyl-4-yl 5-*n*-octylthiophene-2-carboxylate which is smectogenic with tilt angles of around 45° [3], suggest that there is no interdigitation between the layers. Estimated tilt angles, (see the figure), have been calculated for the compounds of series (**1**) assuming this also to be the case. The figure shows that apart from a small initial jump rather than a continuous evolution [4] at the S_C to S_I transition, changes in spacing are very small, except for a very gradual decrease seen on cooling the smectic crystal K phase. A similar decrease in spacing occurs in all the compounds as the crystallization temperature is approached.

3. Conclusions

The compounds studied have extensive smectic polymorphism, but this is limited to tilted mesophases, substantiating the optical microscopic observations on these compounds, [1]. The tilt angle shows only a small variation over the entire mesophase range. In all of the compounds it is generally possible to observe some discontinuity in the layer spacing, albeit small, at the transitions between mesophases.

4. Experimental

Investigations were carried out using filtered CuK_α radiation as described elsewhere, [5]. Sample temperatures were accurate to $\pm 0{\cdot}2$°C and absolute values of spacings are accurate to $\pm 0{\cdot}1$ Å.

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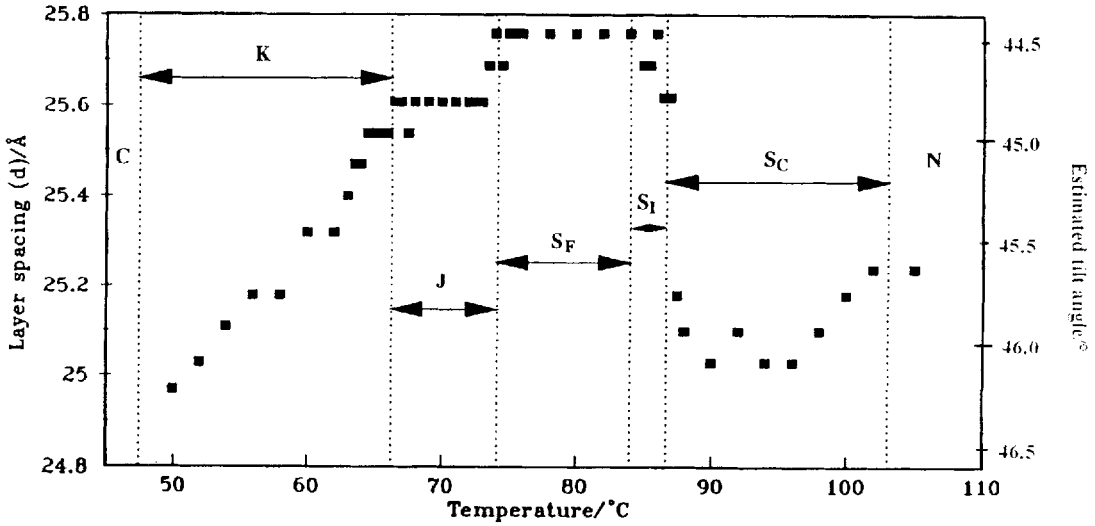

Figure 4′-*n*-Decylbiphenyl-4-yl 5-*n*-octylthiophene-2-carboxylate: temperature variation of the smectic layer spacing and estimated tilt angle.

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