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# Molecular Crystals and Liquid Crystals

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## A Pure Disc-Like Molecule with Cholesteric Properties

J. Malthete  $^{\rm a}$  , C. Destrade  $^{\rm b}$   $^{\rm a}$  , Nguyen Huu Tinh  $^{\rm b}$   $^{\rm a}$  & J. Jacques  $^{\rm a}$ 

<sup>a</sup> Laboratoire de Chimie des Interactions Moléculaires COLLEGE DE FRANCE, 75235, PARIS, Cedex, FRANCE

<sup>b</sup> Centre de Recherche Paul Pascal, Domaine Universitaire, 33405, TALENCE, Cedex, FRANCE Version of record first published: 20 Apr 2011.

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#### A PURE DISC-LIKE MOLECULE WITH CHOLESTERIC PROPERTIES

J. MALTHETE, C. DESTRADE  $^{(\star)}$ , NGUYEN HUU TINH  $^{(\star)}$  and J. JACQUES

Laboratoire de Chimie des Interactions Moléculaires COLLEGE DE FRANCE, 75235 PARIS Cedex FRANCE

Centre de Recherche Paul Pascal, Domaine Universitaire, 33405 TALENCE Cedex FRANCE
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#### Abstract

A cholesterogenic disc-like derivative of triphenylene is described. The  $N_D^{\star}$  phase exhibits oily streaks from 192.5 to 246.5°C. That is the first evidence of a cholesteric phase in a pure disc-like molecule.

#### Introduction

Recently, we have shown that an optically active disc-like molecule, when dissolved in the N<sub>D</sub> nematic phase (N<sub>D</sub>: a nematic phase built up with disc-like molecule ) of 2,3,6,7,10,11-hexa(4-n-heptyloxy)benzoyloxytriphenylene, induced a torsion. This result suggested us to build up a pure chiral disc-like molecule with such a cholesteric behaviour.

We describe in the present letter the first example of a pure disc-like compound which exhibits "cholesteric" properties : (+)-2,3,6,7,10,11-hexa-[S-(4-methy1)-4-n-hexyloxybenzoyloxy] triphenylene 1 displays a twisted  $N_D$  (or  $N_D^{\bigstar}$ ) phase in the range of  $\widetilde{1}92.5\,^{\circ}\text{C}$  (K+ND) to 246.5°C (ND+1).

Experimental

This compound 1 was prepared by reaction of the S-(+)-(4-methyl)-4-n-hexyloxybenzoyl chloride 2 with 2,3,6,7,10,11-hexahydroxytriphenylene in anhydrous pyridine and was purified by thin layer chromatography on silicagel (using 40% acetone/hexane mixture as an eluant) and recrystallized from ethanol/diethylether mixture.

The acid chloride 2 was prepared from S-(-)-2-methylbutano1, the p-toluenesulfonate of which was condensed with sodium diethyl malonate to give S-(+)-4-methylhexanoic acid 4. 4 was reduced (LiAlH<sub>4</sub> in boiling THF) and the resulting alcohol was condensed, via its p-toluenesulfonate, with the di-sodium salt of 4-hydroxybenzoic acid in DMF (2 h, reflux). Saponification afforded S-(+)-(4-methyl)-4-n-hexyloxybenzoic acid 3, which was purified by crystallization from aqueous ethanol: K 116 S<sub>C</sub> 126 I;  $[\alpha]_{578}^{25}$ : +9.0° (c $^{\sim}$ 1, CHCl<sub>3</sub>). The reaction of 3 with thionyl chloride afforded 2.

Transition temperatures of 1 were determined by calorimetry using a DSC2 (Perkin Elmer). The textures were observed with a polarising microscope equipped with a heating and cooling stage (Mettler FP5).

$$C_2H_5$$
  $C_2H_5$   $C$ 

Results

This compound 1 is mesogenic. The cholesteric range is K 192.5 Nx 246.5  $\widetilde{I}$ . Microscopic observations of optical textures of this fluid phase shown oily streaks (Fig. 1) similar to those observed from a 50/50 mixture in weight of C<sub>7</sub>OHBT(\*) and (+)-2,3,6,7,10,11-hexa-[S-(3-methyl)-nonanoyloxy] triphenylene.

This  $N_D^{\star}$  phase is entirely miscible with the  $N_D$  nematic one of the C<sub>6</sub>OHBT derivative (Fig. 2). In this contact, one can see the oily streaks of  $\frac{1}{1}$  overgrow the  $N_D$  nematic

phase of C60HBT.

Let us point out that the existence of cholesteric properties seems to be very dependent to the structure of the chiral chain attached to the benzoic acid. We have observed that optically active compounds 1 with R = S-(+) n-C<sub>6</sub>H<sub>13</sub>-CH(CH<sub>3</sub>)(CH<sub>2</sub>)<sub>2</sub>O-CO- and S-(+) n-C<sub>4</sub>H<sub>5</sub>CH(CH<sub>3</sub>)-CH<sub>2</sub>-O exhibit respectively only a rectangular columnar phase  $D_{rd}$ , and none mesomorphic propertie. This behaviour recalls the hard dependence of the classical nematic stability toward the ramifications<sup>5</sup>.

<sup>(\*)</sup>C<sub>7</sub> OHBT is 2,3,6,7,10,11-hexa(4-n-heptyloxy)benzoyloxy-triphenylene.

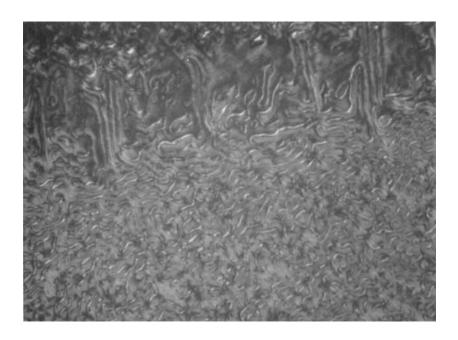


Fig. 1 : Optical texture observed from derivative 1 at 190°C : oily streaks.

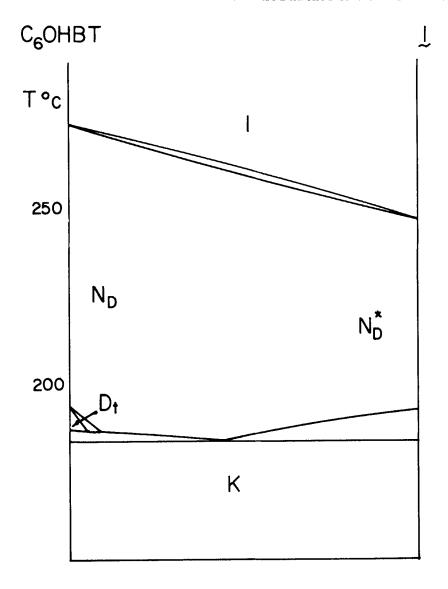


Fig. 2 : Isobaric phase diagram between  $C_6^{OHBT}$  and 1.

### J. MALTHETE, C. DESTRADE, N. H. TINH and J. JACQUES

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- 3 [ $\alpha$ ]  $^{25}$  = +8.6° ( $c^{\circ}$ 2, CHCl $_3$ ); IR (nujol) 1740-1610-1510-1250-1170 cm $^{-1}$ ; H NMR (CDCl $_3$ , TMS)  $\delta$ 0.7...1.8 (m, 78H),  $\delta$ 3.85 (t, 12H), 6.55 and 7.80 (2d, 24H),  $\delta$ 8.25 (s, 6H); Anal. Calcd for C  $_{102}^{1}$ H  $_{120}^{1}$ O  $_{18}^{1}$ C, 74.97; H, 7.40; Found: C, 75.25; H, 7.46.
- This  $N_D^{\bigstar}$  phase is right-handed with a comparatively large pitch (about 30  $\mu m$  at  $\sim$  200°C). We are grateful to Prof. J. Billard for these preliminary measurements.
- J. Malthête, J. Billard, J. Canceill, J. Gabard et J. Jacques, <u>J. Physique Colloq.</u> 37 C3, 1 (1976)