

Kanji Kubo* and Akira Mori

Institute of Advanced Material Study, 86,
Kyushu University, Kasuga-koen, Kasuga,
Fukuoka 816-8580, JapanCorrespondence e-mail:
kubo-k@cm.kyushu-u.ac.jp

Key indicators

Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.071
 wR factor = 0.303
Data-to-parameter ratio = 18.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4-Cyanophenyl 4-*n*-decanyloxybenzoate

The title compound, $\text{C}_{24}\text{H}_{29}\text{NO}_3$, has a phase sequence of crystals–smectic A–nematic–isotropic liquid. The molecular length of the compound is 25.323 (9) Å. Intermolecular contacts between two CN groups, between a carbonyloxy and a CN group, and between two carbonyloxy groups are observed in the crystal state.

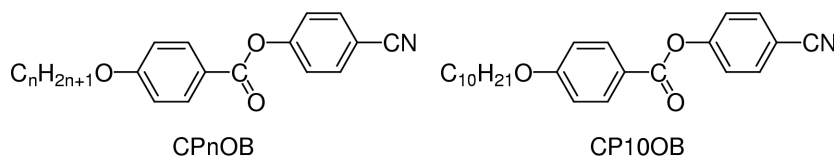
Received 23 February 2001

Accepted 5 March 2001

Online 9 March 2001

Comment

Mesomorphic sequences of 4-cyanophenyl 4-*n*-alkoxybenzoates (CP*n*OB) are dependent on the length of the alkoxy chains, *i.e.* crystals–nematic–isotropic for $n = 5$ –8, crystals–smectic A–nematic–isotropic for $n = 9$ –11, and crystals–smectic A–isotropic for $n = 12$ (Vill, 2000). The crystal structures of CP*n*OB with some alkyl chains ($n = 4, 5, 6, 7, 8, 12$) have been reported (Baumeister *et al.*, 1981; Ibrahim *et al.*, 1995; Iki & Hori, 1995; Kubo & Mori, 2001). We now report the structure of 4-cyanophenyl 4-*n*-decanyloxybenzoate (CP10OB), aimed at elucidating the relationships between mesomorphic properties and molecular packings of CP*n*OB in the crystal lattice.



The intersection angle between the least-squares planes *A* (defined by C2–C7) and *B* (defined by C9–C14) of CP10OB is 49.6 (2)°, which is similar to those [49.2 and 50.5 (1)°] of CP8OB (Iki & Hori, 1995) and CP12OB (Kubo & Mori, 2001), while that between the least-squares planes *B* and *C* (defined by O1/O2/C8) is 8.3 (3)°, which is similar to those (8.3 and 7.9°) of CP8OB and CP12OB. The paraffin chains have all-*trans* conformations and the molecular length of the compound is 25.323 (9) Å for the N1–C24 distance.

Intermolecular close contact between CN groups of a pair of antiparallel molecules is observed in the crystal lattice. The distance for C1–N1ⁱ [symmetry code: (i) 1 – *x*, 1 – *y*, –1 – *z*] is 3.50 (7) Å, which is similar to those (3.486 and 3.514 Å) of CP8OB (Iki & Hori, 1995) and CP12OB (Kubo & Mori, 2001), and shorter than those (3.829 and 3.602 Å) of CP4OB (Ibrahim *et al.*, 1995) and CP6OB (Iki & Hori, 1995). Carbonyloxy groups of another pair of antiparallel molecules are also closely arranged. The distances for O1–O1ⁱⁱ [symmetry code: (ii) 2 – *x*, 1 – *y*, –*z*], O1–O2ⁱⁱ and O1–C8ⁱⁱ are 3.403 (6), 3.578 (5) and 3.508 (7) Å, respectively. In addition, close contacts between a CN and a carbonyloxy group of

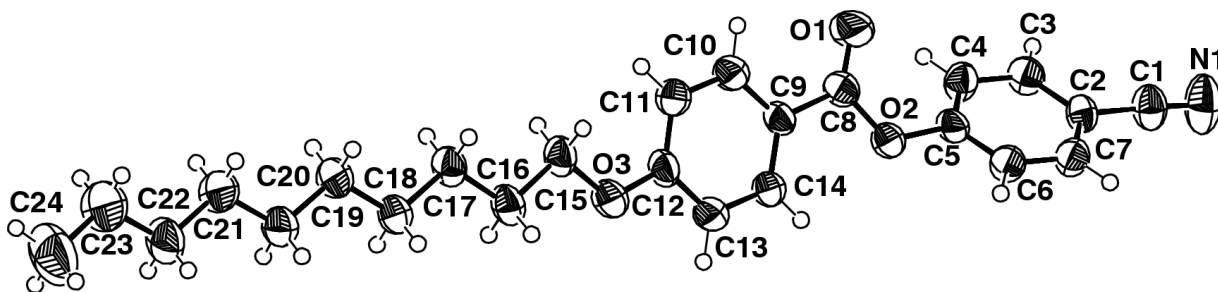


Figure 1
The molecular structure of CP10OB showing 50% probability displacement ellipsoids.

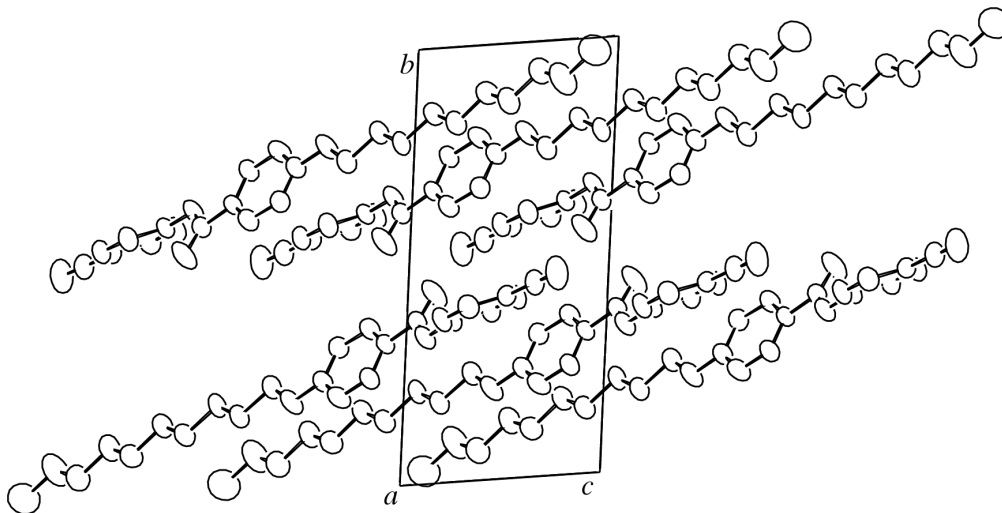


Figure 2
Packing diagram viewed down the *a* axis. H atoms have been omitted for clarity.

the other pair of molecules are observed; the distances for C1–O1ⁱⁱⁱ [symmetry code: (iii) $2 - x, 1 - y, -1 - z$] and N1–O1ⁱⁱⁱ are 3.565 (7) and 3.794 (7) Å, respectively.

The crystal of CP10OB has a distinct layer structure through infinite networks of the CN...CN interaction, which is similar to those of CP8OB and CP12OB. In conclusion, correlations between crystal structure and mesomorphic properties of CPnOB with different phases have not been found.

Experimental

The title compound (CP10OB) was prepared by esterification of 4-cyanophenol with 4-*n*-decyloxybenzoyl chloride. Single crystals of CP10OB were obtained by recrystallization from ethyl acetate.

Crystal data

$C_{24}H_{29}NO_3$	$Z = 2$
$M_r = 379.48$	$D_x = 1.162 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.9982 (17) \text{ \AA}$	Cell parameters from 25 reflections
$b = 14.850 (2) \text{ \AA}$	$\theta = 8.8\text{--}18.1^\circ$
$c = 6.7625 (6) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$\alpha = 96.864 (9)^\circ$	$T = 296 (2) \text{ K}$
$\beta = 93.694 (9)^\circ$	Prism, colorless
$\gamma = 81.945 (12)^\circ$	$0.47 \times 0.37 \times 0.34 \text{ mm}$
$V = 1084.5 (2) \text{ \AA}^3$	

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω – 2θ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.926$, $T_{\max} = 1.000$
 5095 measured reflections
 4695 independent reflections
 1517 reflections with $I > 2\sigma(I)$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.071$
 $wR(F^2) = 0.303$
 $S = 0.92$
 4695 reflections
 253 parameters

$R_{\text{int}} = 0.079$
 $\theta_{\text{max}} = 27.0^\circ$
 $h = -14 \rightarrow 14$
 $k = -18 \rightarrow 18$
 $l = 0 \rightarrow 8$
 3 standard reflections
 frequency: 120 min
 intensity decay: 0.7%

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1516P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.35 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.43 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

N1–C1	1.132 (6)	O3–C12	1.360 (5)
O1–C8	1.195 (6)	C1–C2	1.441 (7)
O2–C8	1.367 (6)	C8–C9	1.465 (6)
O2–C5	1.390 (5)		
N1–C1–C2	178.4 (7)	O2–C8–C9	110.8 (4)
C8–O2–C5–C4	–47.0 (7)	O2–C8–C9–C10	171.6 (4)
C5–O2–C8–O1	2.1 (8)	C12–O3–C15–C16	179.9 (4)
C5–O2–C8–C9	–177.5 (4)		

All H atoms were located at ideal positions and constrained with U_{iso} held fixed to $1.2U_{\text{eq}}$ of the parent atoms.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Xtal_GX* (Hall & du Boulay, 1995); software used to prepare material for publication: *SHELXL97*.

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Baumeister, U., Hartung, H., Graniec, M. & Jaskólski, M. (1981). *Mol. Cryst. Liq. Cryst.* **69**, 119–130.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN*. Enraf-Nonius, Delft, The Netherlands.
- Hall, S. R. & du Boulay, D. (1995). *Xtal_GX*. University of Western Australia, Australia.
- Iki, H. & Hori, K. (1995). *Bull. Chem. Soc. Jpn.* **68**, 1281–1288.
- Ibrahim, I. H., Paulus, H., Mokhles, M. & Haase, W. (1995). *Mol. Cryst. Liq. Cryst. Sci. Technol. Sect. A*, **258**, 185–190.
- Kubo, K. & Mori, A. (2001). *Acta Cryst.* **E57**, o113–115.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Vill, V. (2000). *LiqCryst 3.5. Database of Liquid Crystalline Compounds*. Fujitsu Kyushu System Engineering, Fukuoka, Japan.