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4-Cyanophenyl 4-*n*-dodecanyloxybenzoate

Kanji Kubo* and Akira Mori

Institute of Advanced Material Study, 86, Kyushu University, Kasuga-koen, Kasuga, Fukuoka 816-8580, Japan

Correspondence e-mail: kubo-k@cm.kyushu-u.ac.jp

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.059
wR factor = 0.193
Data-to-parameter ratio = 21.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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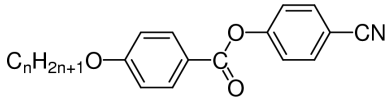
The title compound, $C_{26}H_{33}NO_3$, has a phase sequence of crystal–smectic A–isotropic liquid. The molecular length of the compound is 27.8 Å and the paraffin chain has an all-*trans* conformation. Intermolecular contacts between two CN groups, between a carbonyloxy and a CN group, and between two carbonyloxy groups are observed in the crystal state.

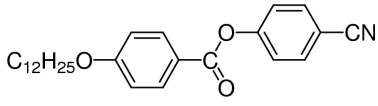
Comment

Mesomorphic sequences of 4-cyanophenyl 4-*n*-alkoxybenzoates (CP*n*OB) are dependent on the length of the alkoxy chains, *i.e.* crystal–nematic–isotropic for n = 5–8, crystal–smectic A–nematic–isotropic for n = 9–11, and crystal–smectic A–isotropic for n = 12 (Vill, 2000). Although the crystal structures of CP*n*OB with shorter alkyl chains (n = 5–8) have been reported (Baumeister *et al.*, 1981; Iki & Hori, 1995), those with longer alkyl chains have not been elucidated. For CP5OB, the crystal has a non-parallel arrangement of molecular long axes (Baumeister *et al.*, 1981). In the crystals of CP6OB and CP8OB (Iki & Hori, 1995), close contacts between CN groups of a pair of molecules are observed, though in the latter crystal two carbonyloxy groups between another pair of molecules are also closely arranged. For CP7OB (Iki & Hori, 1995), a CN and a carbonyloxy group come close to each other between a pair of molecules. These compounds are enantiotropic nematogens. We now report the structure of 4-cyanophenyl 4-*n*-dodecanyloxybenzoate (CP12OB), which has a smectic A phase, with the aim of contributing to a deeper understanding of the relationships between mesomorphic properties and molecular packings in the lattice.

The intersection angle between the least-squares planes *A* (defined by C2–C7) and *B* (defined by C9–C14) is 50.5 (1)°, which is similar to that (49.2°) reported by Iki & Hori (1995), while that between the least-squares planes *B* and *C* (defined by O1, O2 and C8) is 7.9 (2)°. The paraffin chain has an all-*trans* conformation and the molecular length of the compound is 27.848 (13) Å for the N1···C26 distance.



CPnOB



CP12OB

Intermolecular close contact between CN groups of a pair of antiparallel molecules is observed in the crystal lattice. The distance for C1···N1^{i} is 3.514 (5) Å [symmetry code: (i) $-1-x, 1-y, 2-z$], which is similar to that (3.486 Å; Iki & Hori, 1995). Carbonyloxy groups of another pair of anti-

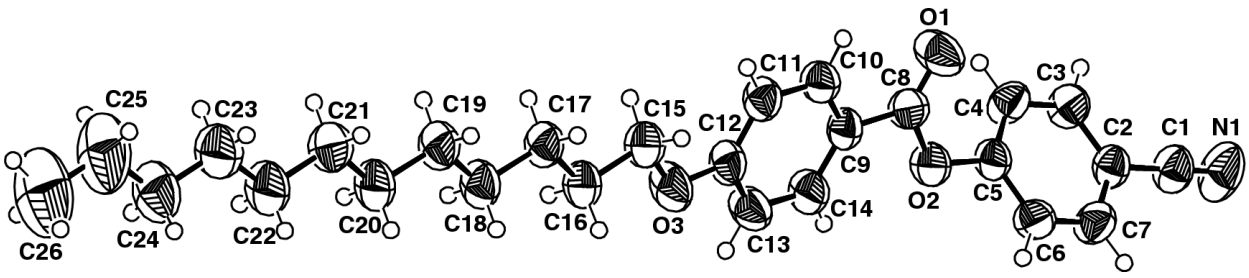


Figure 1
The molecular structure of CP12OB showing 50% probability displacement ellipsoids.

parallel molecules are also closely arranged. The distances for O1···O1^{ii}, O1···O2^{ii} and O1···C8^{ii} are 3.336 (4), 3.598 (3) and 3.460 (5) Å, respectively [symmetry code: (ii) $-x, 1-y, 1-z$]. In addition, close contacts between a CN and a carbonyloxy group of the other pair of molecules are observed as follows: the distances for C1···O1^{iii} and N1···O1^{iii} are 3.581 (4) and 3.798 (4) Å, respectively [symmetry code: (iii) $-x, 1-y, 2-z$].

Comparing the dipole moments between a CN group (4.0 D) and a COO group (1.8 D) (The Chemical Society of Japan, 1993), antiparallel interactions between two CN groups is expected to be dominant in the crystal lattice. The crystal of CP12OB, therefore, has a distinct layer structure through infinite networks of the CN···CN interaction.

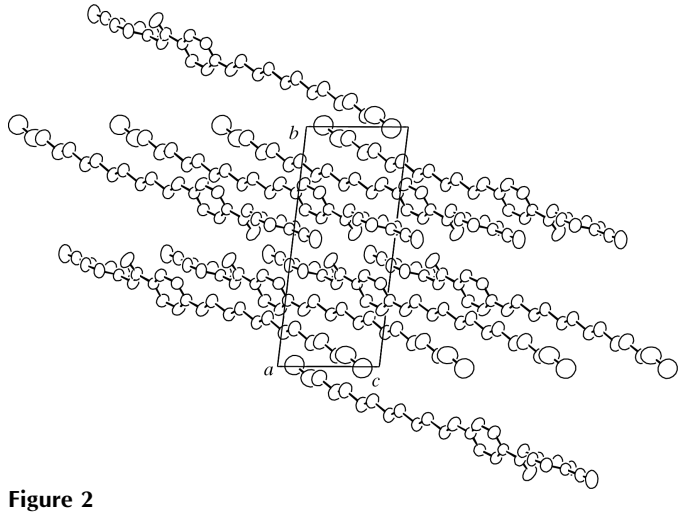


Figure 2
Packing diagram viewed down the *a* axis. H atoms have been omitted for clarity.

Experimental

The title compound (CP12OB) was prepared by esterification of 4-cyanophenol with 4-*n*-dodecanyloxybenzoyl chloride. The single crystals of CP12OB were obtained by recrystallization from ethyl acetate.

Crystal data

$C_{26}H_{33}NO_3$
M_r = 407.53
Triclinic, $P\overline{1}$
a = 11.031 (1) Å
b = 16.639 (2) Å
c = 6.813 (1) Å
α = 95.381 (10)°
β = 94.530 (7)°
γ = 104.686 (10)°
V = 1197.4 (2) Å^3
Z = 2
D_x = 1.130 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 18 reflections
θ = 9.1–18.0°
μ = 0.07 mm^{-1}
T = 296 (2) K
Prism, colorless
0.43 × 0.43 × 0.10 mm

Data collection

Enraf–Nonius FR590 diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scans (North *et al.*, 1968)
T_{min} = 0.961, T_{max} = 0.991
6224 measured reflections
5747 independent reflections
1764 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 28.0°
$h = -14 \rightarrow 14$
$k = -21 \rightarrow 21$
$l = -8 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: 5.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.193
S = 0.91
5747 reflections
272 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0080 (18)

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.133 (3)	O3—C12	1.359 (3)
O1—C8	1.188 (3)	C1—C2	1.444 (4)
O2—C8	1.371 (3)	C8—C9	1.468 (4)
O2—C5	1.400 (3)		
N1—C1—C2	179.1 (4)		
O2—C8—C9	110.5 (3)		
C8—O2—C5—C4	−48.2 (4)	O2—C8—C9—C10	172.3 (3)
C5—O2—C8—O1	2.2 (5)	C12—O3—C15—C16	179.5 (3)
C5—O2—C8—C9	−176.8 (2)		

The positional parameters of the H atoms were calculated geometrically and refined using a riding model. Their U_{iso} values were fixed to 1.2 times U_{eq} of the bonded non-H 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: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *Xtal_GX* (Hall & du Boulay, 1995); software used to prepare material for publication: *SHELXL*97.

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