Refinement

Refinement on F
$R = 0.0486$
$wR = 0.0556$
$S = 1.60$
1364 reflections
226 parameters
H atoms: see below
$w = 1/[\sigma^2(F) + 0.0002F^2]$
$(\Delta/\sigma)_{\max} = 0.007$
$\Delta\rho_{\max} = 0.160$ e Å^{-3}
$\Delta\rho_{\min} = -0.230$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.4307 (6)	0.7354 (3)	0.7698 (3)	0.054 (2)
C2	0.2578 (6)	0.6907 (3)	0.6608 (3)	0.059 (2)
C3	0.2778 (5)	0.5633 (3)	0.5850 (3)	0.045 (2)
C4	0.3237 (5)	0.3358 (3)	0.5972 (3)	0.052 (2)
C5	0.3882 (6)	0.2513 (3)	0.6651 (4)	0.055 (2)
C6	0.4775 (6)	0.2916 (4)	0.7842 (4)	0.059 (2)
C7	0.5025 (6)	0.4204 (4)	0.8386 (3)	0.056 (2)
C8	0.4356 (5)	0.5089 (4)	0.7733 (3)	0.049 (2)
C9	0.3485 (5)	0.4678 (3)	0.6516 (3)	0.042 (2)
C10	0.6193 (6)	0.7679 (4)	0.7365 (4)	0.075 (2)
C11	0.3884 (8)	0.8456 (4)	0.8597 (4)	0.096 (2)
C12	0.1011 (5)	0.5796 (4)	0.2959 (3)	0.051 (2)
C13	0.0532 (5)	0.6868 (4)	0.2399 (3)	0.050 (2)
C14	−0.0258 (6)	0.6526 (4)	0.1182 (3)	0.067 (2)
C15	−0.0701 (7)	0.7458 (6)	0.0568 (4)	0.090 (3)
C16	−0.0386 (7)	0.8731 (6)	0.1169 (5)	0.091 (3)
C17	0.0376 (6)	0.9088 (4)	0.2380 (4)	0.078 (2)
C18	0.0838 (5)	0.8147 (4)	0.2995 (3)	0.058 (2)
O1	0.4583 (4)	0.6332 (2)	0.8352 (2)	0.061 (1)
O2	0.2716 (7)	0.0795 (3)	0.5016 (3)	0.138 (2)
O3	0.4050 (5)	0.0370 (3)	0.6661 (3)	0.109 (2)
O4	0.1695 (3)	0.6249 (2)	0.4180 (2)	0.054 (1)
O5	0.0849 (4)	0.4682 (3)	0.2460 (2)	0.068 (1)
N1	0.2352 (4)	0.5235 (3)	0.4715 (3)	0.050 (1)
N2	0.3518 (6)	0.1115 (3)	0.6054 (4)	0.082 (2)

Table 2. *Selected geometric parameters* (Å, °)

C1—C2	1.519 (5)	C8—O1	1.348 (4)
C1—O1	1.471 (5)	C12—C13	1.470 (6)
C2—C3	1.498 (5)	C12—O4	1.384 (4)
C3—C9	1.474 (6)	C12—O5	1.191 (5)
C3—N1	1.279 (5)	O4—N1	1.429 (4)
C8—C9	1.393 (5)		
C2—C1—O1	110.7 (3)	C13—C12—O4	110.6 (3)
C1—C2—C3	111.3 (3)	C13—C12—O5	126.3 (3)
C2—C3—C9	114.7 (3)	O4—C12—O5	123.1 (4)
C2—C3—N1	129.7 (4)	C1—O1—C8	119.0 (3)
C9—C3—N1	115.5 (3)	C12—O4—N1	110.8 (3)
C9—C8—O1	123.2 (4)	C3—N1—O4	109.8 (3)
C3—C9—C8	119.5 (3)		

H atoms were introduced in calculated positions and given fixed isotropic U values.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1093). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4-Octyloxybiphenyl-4′-yl 4-Methoxybenzoate

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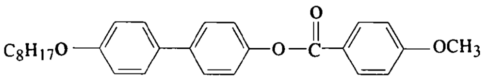
Abstract

The title compound, $C_{28}H_{32}O_4$, has the phase sequence crystal–nematic–isotropic liquid. The crystal has an 'imbricated' structure, *i.e.* a half-and-half overlapping of molecules. Molecular long axes are oriented in one direction, resulting in a polar structure.

Comment

Liquid crystalline behaviour is influenced by molecular structure, such as the direction of polar groups, as well as the kind of polar groups. For example, the title compound, (I), has the phase sequence crystal–nematic–isotropic liquid, while 4-methoxyphenyl 4′-octyloxybiphenyl-4-carboxylate, in which the direction of the ester linkage is inverted, has the phase sequence crystal–smectic A–nematic–isotropic liquid (Takeda, Sakurai, Takenaka, Miyake, Doi & Kusabayashi, 1990). To clarify the intermolecular interaction controlling the phase sequences, the crystal structure analyses have been attempted for both compounds. This paper describes the crystal structure of compound (I).



(I)

The molecular structure of (I) with the atomic numbering scheme is shown in Fig. 1. The almost coplanar biphenyl moiety with a dihedral angle of 2.6 (2)° between the rings and the paraffin chain with an all-*trans* conformation lie approximately in a plane. The methoxybenzoate moiety forms another plane and so the molecule is composed of two approximate planes, which make a dihedral angle of 70.64 (7)° with one another.

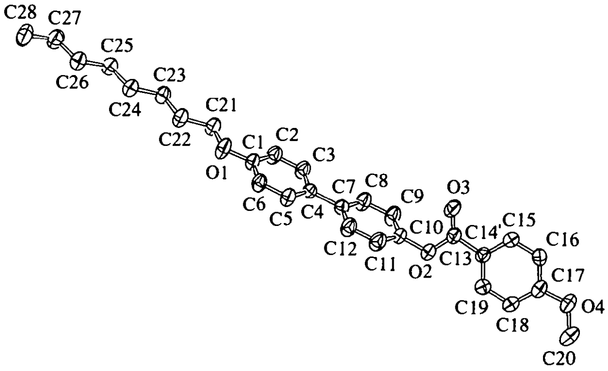


Fig. 1. The molecular structure of (I) shown with 50% probability ellipsoids. H atoms have been omitted for clarity.

The crystal structure viewed along the *b* axis is shown in Fig. 2. All the molecules are arranged in a parallel fashion. Lateral overlapping of core moieties is rather small, resulting in an 'imbricated' structure, *i.e.* a half-and-half overlapping of molecules. It should also be noted that the heads and tails of all the molecules are lined up in one direction. It was considered that the electron-donating methoxy group enhances the dipole moment of the carbonyl group of the ester linkage and the total dipole moment was calculated to be 2.95 D for a similar compound, 4-methoxybiphenyl-4′-yl 4-methoxybenzoate (Takeda, Sakurai, Takenaka, Miyake, Doi & Kusabayashi, 1990). The components of the dipole moment along the *b* axis are cancelled due to the *c*-glide symmetry. However, the components parallel to the *ac* plane are not cancelled but accumulated throughout the crystal, resulting in a highly polar structure.

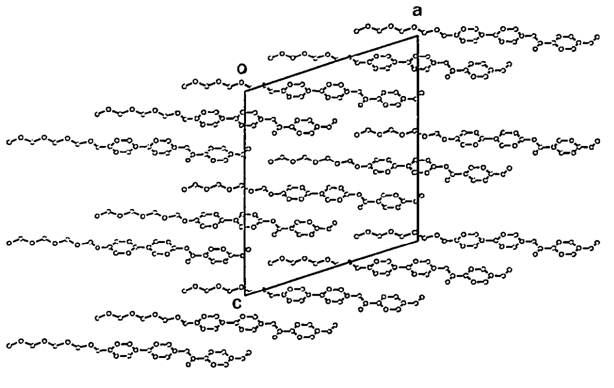


Fig. 2. The crystal structure viewed along the *b* axis.

Differential scanning calorimetric (DSC) measurement, however, shows no anomaly until the crystal melts to the nematic, with a single sharp endothermic peak (ΔH = 38 kJ mol^{-1}) suggesting that the crystal is in the thermodynamically most stable state.

Experimental

The title compound was synthesized by a conventional method (Takeda, Sakurai, Takenaka, Miyake, Doi & Kusabayashi, 1990). Single crystals were obtained from a toluene–methanol solution at room temperature.

Crystal data

$C_{28}H_{32}O_4$
M_r = 432.558
Monoclinic
Cc
a = 20.190 (4) Å
b = 5.388 (2) Å
c = 22.728 (3) Å
β = 107.502 (11)°
V = 2358.0 (11) Å^3
Z = 4
D_x = 1.218 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 20 reflections
θ = 27.5–28.5°
μ = 0.637 mm^{-1}
T = 293 (2) K
Plate
0.50 × 0.40 × 0.05 mm
Colourless

Data collection

Rigaku AFC-7*R* diffractometer
$2\theta-\omega$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min}$ = 0.752, $T_{\max}$ = 1.000
4047 measured reflections
1746 independent reflections

1709 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int}$ = 0.02
$\theta_{\max}$ = 60°
$h = 0 \rightarrow 22$
$k = 0 \rightarrow 6$
$l = -25 \rightarrow 24$
3 standard reflections monitored every 150 reflections intensity decay: 0.45%

Refinement

Refinement on F^2
$R(F) = 0.0434$
$wR(F^2) = 0.1259$
$S = 1.084$
1795 reflections
253 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0962P)^2 + 0.6339P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.136$
$\Delta\rho_{\max} = 0.191$ e Å^{-3}
$\Delta\rho_{\min} = -0.200$ e Å^{-3}
Extinction correction: see below
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.3 (3)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	−0.34829 (12)	−0.0049 (5)	0.04055 (12)	0.0639 (7)
O2	0.16313 (11)	−0.0849 (5)	0.17900 (11)	0.0532 (6)
O3	0.16988 (14)	0.2262 (6)	0.2460 (2)	0.0839 (10)
O4	0.48761 (12)	−0.0462 (6)	0.30235 (12)	0.0612 (7)
C1	−0.27720 (7)	0.0027 (4)	0.05606 (9)	0.0461 (8)
C2	−0.23853 (9)	0.1832 (4)	0.03748 (10)	0.0568 (9)
C3	−0.16643 (9)	0.1703 (4)	0.05673 (11)	0.0540 (9)
C4	−0.13300 (7)	−0.0232 (4)	0.09456 (10)	0.0406 (7)
C5	−0.17167 (9)	−0.2037 (4)	0.11314 (10)	0.0519 (9)
C6	−0.24377 (9)	−0.1907 (4)	0.09389 (10)	0.0535 (9)
C7	−0.05314 (7)	−0.0395 (4)	0.11687 (9)	0.0419 (7)
C8	−0.01358 (9)	0.1370 (4)	0.09802 (10)	0.0566 (9)
C9	0.05849 (9)	0.1251 (4)	0.11921 (12)	0.0607 (9)
C10	0.09101 (7)	−0.0635 (4)	0.15927 (12)	0.0464 (8)
C11	0.05145 (10)	−0.2400 (4)	0.17813 (12)	0.0624 (10)
C12	−0.02062 (10)	−0.2280 (4)	0.15693 (12)	0.0627 (10)
C14	0.27385 (7)	0.0263 (4)	0.24376 (8)	0.0424 (7)
C15	0.31600 (9)	0.1927 (4)	0.28519 (9)	0.0497 (8)
C16	0.38767 (9)	0.1619 (4)	0.30401 (10)	0.0509 (8)
C17	0.41719 (7)	−0.0352 (4)	0.28139 (10)	0.0478 (8)
C18	0.37505 (9)	−0.2016 (3)	0.23995 (10)	0.0472 (8)
C19	0.30338 (9)	−0.1708 (4)	0.22114 (9)	0.0461 (8)
C13	0.1984 (2)	0.0697 (7)	0.22500 (15)	0.0485 (8)
C20	0.5211 (2)	−0.2467 (9)	0.2821 (2)	0.0712 (11)
C21	−0.3894 (2)	0.1812 (7)	0.0010 (2)	0.0562 (8)
C22	−0.4633 (2)	0.1104 (8)	−0.0052 (2)	0.0570 (9)
C23	−0.5195 (2)	0.2726 (7)	−0.0469 (2)	0.0507 (8)
C24	−0.5909 (2)	0.1791 (7)	−0.0469 (2)	0.0511 (8)
C25	−0.6526 (2)	0.3181 (7)	−0.0876 (2)	0.0516 (8)
C26	−0.7206 (2)	0.2079 (7)	−0.0842 (2)	0.0564 (9)
C27	−0.7857 (2)	0.3350 (8)	−0.1231 (2)	0.0669 (10)
C28	−0.8515 (2)	0.2140 (11)	−0.1178 (3)	0.0907 (15)

In the refinement, each benzene ring was constrained to have a regular hexagon with a C—C distance of 1.39 Å. H atoms were calculated geometrically (C—H 0.96 for primary, 0.97 for secondary and 0.93 Å for aromatic) and were included in the refinement but not refined. At the final stage of refinement, five reflections ($11\bar{2}$, $20\bar{6}$, $11\bar{4}$, 002 and 111) whose large discrepancies ($F_o < F_c$) were considered to have suffered from extinction effects were omitted.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: OA1004). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris[2-(2-methoxyethoxy)phenyl]phosphine Oxide Monohydrate

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Abstract

The geometry about the P atom in the title compound, $C_{27}H_{33}O_7P.H_2O$, is virtually indistinguishable from that of triphenylphosphine oxide and several analogs. Threefold symmetry is absent, surprisingly, and each 2-methoxyethoxy side chain is unique. One is involved in an internal hydrogen bond to water, another is on the same side of the plane formed by the three phosphorus-bearing C atoms as is the hydrogen-bonding side chain, and the third is on the other side of this plane.

Comment

Tris[2,6-di-(2-methoxyethoxy)phenyl]phosphine, (1), has been studied with regard to its ionophoric capabilities (Mao, 1991). As part of that study, tris[2-(2-methoxyethoxy)phenyl]phosphine, (2), was prepared to assess the contribution of 'pre-organization' to the binding characteristics of (1). Therefore, it was of interest to explore the disposition of the side chains of (2) relative

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