

F. H atoms in idealized positions [$C-H = 0.96 \text{ \AA}$, $U(H) = 1.2 \times U_{iso}(C)$]. All non-H atoms [with the exception of $C(5)$] refined with anisotropic thermal parameters. Difficulty in refining $C(5)$ anisotropically due to lack of absorption correction; necessary data unfortunately not collected before crystal removed from diffractometer. At convergence [$(\Delta/\sigma)_{max} = 0.009$, $(\Delta/\sigma)_{mean} = 0.002$ for the last three cycles] $R = 0.077$, $wR = 0.090$, $S = 1.20$, slope of normal probability plot = 1.07, $(\Delta\rho)_{max} = 0.70$, $(\Delta\rho)_{min} = -0.65 \text{ e \AA}^{-3}$. Neutral-atom scattering factors and anomalous-dispersion corrections used (*International Tables for X-ray Crystallography*, 1974, Vol. IV); all calculations performed on a Data General Eclipse S/140 computer using *SHELXTL* program library (Sheldrick, 1983). Table 1 gives atomic coordinates, and Table 2 gives bond lengths and angles.* Fig. 1

* Lists of anisotropic thermal parameters, H-atom coordinates and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54037 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

shows the structure of (1), as well as the numbering scheme used.

Related literature. The structure of *O,O'*-dimethylaspirochlorine has been previously studied (Sakata, Maruyama, Uzawa, Sakurai, Lu & Clardy, 1987). The structure of (1) and *O,O'*-dimethylaspirochlorine are similar with the exception of different substituents on the main three-ring system. In the structure of (1), a very close $C(2)-H(2)\cdots O(1)^i$ contact exists linking molecules of (1) into dimers [$C(2)-O(1)^i$, 3.126, $H(2)-O(1)^i$, 2.255 \AA].

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Mesogenic 4-Methoxyphenyl 4-Hexyloxybenzoate

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Abstract. $C_{20}H_{24}O_4$, $M_r = 328.4$, monoclinic, $P2_1/c$, $a = 16.211$ (4), $b = 5.457$ (2), $c = 21.688$ (5) \AA, $\beta = 109.41$ (1)°, $V = 1809.23 \text{ \AA}^3$, $Z = 4$, $D_x = 1.205 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$, $\mu = 0.48 \text{ cm}^{-1}$, $F(000) = 704$, $T = 298 \text{ K}$, $R = 0.0480$ for 2094 independent reflections with $|F_o| > 2\sigma(F_o)$. The compound is fully extended with an all-*trans* conformation of the hexyloxy group. The phenyl ring of the benzoate group forms an interplanar angle of 66.1° with the phenyl ring of the methoxyphenyl group. The dihedral angle between the best planes through the hexyloxy group and the attached phenyl group is 3.1°. In the benzoate group the dihedral angle

between the best planes through the phenyl group and carboxylate group is 7.4°. In the crystalline state the molecules form layers parallel to (102).

Experimental. The title compound was prepared by Professor Finkelmann, Freiburg. Colorless prismatic crystals were obtained by evaporation of an acetone–water solution; dimensions 0.22 × 0.27 × 30 mm. Stoe–Siemens AED2 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan, cell parameters by least squares on 58 reflections ($13.1 \leq 2\theta \leq 43.8^\circ$). Analytical absorption correction, maximum and minimum transmission factors 0.766 and 0.862. Total of 4584 reflections ($3 < 2\theta < 45^\circ$) measured in the range $0 < h < 17$, $-5 < k < 5$, $-23 < l < 22$. Standard reflections 011, 006, $\bar{5}04$ measured every

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Table 1. Atomic parameters and equivalent isotropic temperature factors

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor multiplied by 10^3 .

	x	y	z	$U_{eq}(\text{\AA}^2)$
C(1)	0.5905 (2)	-0.2472 (6)	0.5560 (2)	120 (3)
C(2)	0.6741 (2)	-0.3347 (5)	0.5489 (1)	93 (2)
C(3)	0.7076 (1)	-0.1713 (4)	0.5064 (1)	67 (2)
C(4)	0.7922 (1)	-0.2568 (4)	0.4973 (1)	59 (1)
C(5)	0.8177 (1)	-0.0942 (4)	0.4498 (1)	59 (1)
C(6)	0.8997 (1)	-0.1760 (4)	0.4379 (1)	57 (1)
C(7)	0.9864 (1)	-0.0299 (4)	0.3739 (1)	49 (1)
C(8)	0.9959 (1)	0.1483 (4)	0.3308 (1)	52 (1)
C(9)	1.0638 (1)	0.1336 (4)	0.3059 (1)	50 (1)
C(10)	1.1235 (1)	-0.0585 (3)	0.3241 (1)	45 (1)
C(11)	1.1133 (1)	-0.2339 (4)	0.3673 (1)	56 (1)
C(12)	1.0451 (1)	-0.2228 (4)	0.3921 (1)	55 (1)
C(13)	1.1977 (1)	-0.0836 (4)	0.2999 (1)	53 (1)
C(14)	1.2640 (1)	0.0819 (4)	0.2270 (1)	51 (1)
C(15)	1.3233 (1)	0.2685 (4)	0.2439 (1)	58 (1)
C(16)	1.3901 (1)	0.2788 (4)	0.2169 (1)	56 (1)
C(17)	1.3952 (1)	0.0996 (4)	0.1732 (1)	50 (1)
C(18)	1.3341 (1)	-0.0877 (4)	0.1564 (1)	54 (1)
C(19)	1.2681 (1)	-0.0971 (4)	0.1832 (1)	55 (1)
C(20)	1.5193 (2)	0.2893 (5)	0.1562 (1)	78 (2)
O(1)	0.9177 (1)	0.0047 (3)	0.3958 (1)	66 (1)
O(2)	1.2557 (1)	-0.2320 (3)	0.3186 (1)	85 (1)
O(3)	1.1945 (1)	0.0825 (3)	0.2526 (1)	62 (1)
O(4)	1.4585 (1)	0.0900 (3)	0.1443 (1)	67 (1)

Table 2. Selected bond distances (\AA) and angles ($^\circ$)

C(1)—C(2)	1.491 (3)	C(10)—C(13)	1.469 (3)
C(2)—C(3)	1.507 (3)	C(13)—O(2)	1.205 (2)
C(3)—C(4)	1.522 (3)	C(13)—O(3)	1.358 (2)
C(4)—C(5)	1.518 (3)	O(3)—C(14)	1.412 (2)
C(5)—C(6)	1.503 (3)	C(17)—O(4)	1.369 (2)
C(6)—O(1)	1.438 (2)	O(4)—C(20)	1.433 (3)
O(1)—C(7)	1.362 (2)		
Mean C—C distances			
C(7) to C(12)	1.386 (3)	C(14) to C(19)	1.381 (3)
C(1)—C(2)—C(3)	113.5 (2)	C(10)—C(13)—O(2)	125.6 (2)
C(2)—C(3)—C(4)	115.0 (2)	C(10)—C(13)—O(3)	112.2 (2)
C(3)—C(4)—C(5)	111.7 (2)	O(2)—C(13)—O(3)	122.1 (2)
C(4)—C(5)—C(6)	113.5 (2)	C(13)—O(3)—C(14)	117.4 (2)
C(5)—C(6)—O(1)	106.4 (2)	O(3)—C(14)—C(15)	118.5 (2)
C(6)—O(1)—C(7)	119.3 (2)	O(3)—C(14)—C(19)	119.9 (2)
O(1)—C(7)—C(8)	115.0 (2)	C(16)—C(17)—O(4)	124.4 (2)
O(1)—C(7)—C(12)	124.7 (2)	C(18)—C(17)—O(4)	115.5 (2)
C(9)—C(10)—C(13)	122.9 (2)	C(17)—O(4)—C(20)	117.6 (2)
C(11)—C(10)—C(13)	118.2 (2)		

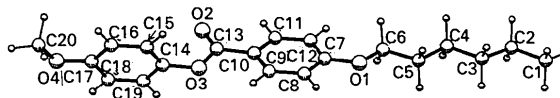


Fig. 1. Molecule projected along [010] with labeling scheme.

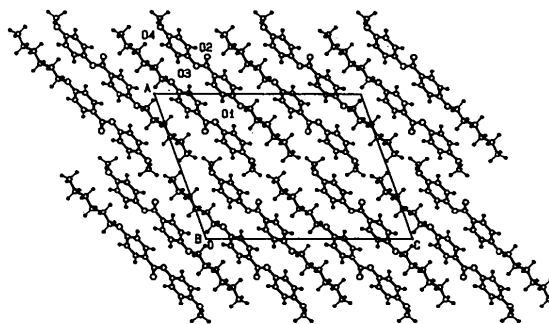


Fig. 2. Crystal structure projected along [010].

3600 s. 2094 unique reflections $|F_o| > 2\sigma(F_o)$ with $R_{int} = 0.0229$. Direct methods (*SHELX90*, Sheldrick, 1990; *SHELX76*, Sheldrick, 1976) structure solution, least-squares refinement of 224 parameters (on F), all non-H atoms anisotropic, H atoms idealized (C—H = 1.08 \AA) with $U_H = 1.1 \times U_{eq}$ of the attached C atom. $wR = 0.0455$, $w = 4.346/\sigma^2(F_o)$. Final refinement maximum shift/e.s.d. 0.002, mean < 0.001 . Final difference Fourier maximum < 0.2 , minimum $> -0.2 e \text{\AA}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters are given in Table 1* and selected bond distances and angles are in Table 2. Fig. 1 shows the molecule together with the numbering scheme and Fig. 2 shows the projection along the monoclinic b axis.

Related literature. Crystal structure determinations of the mesogenic 4'-cyanophenyl-4- n -alkyl and alkoxybenzoates are known (Baumeister, Hartung & Jaskolski, 1982; Mandal, Paul, Schenk & Goubitz, 1986) but none have been reported for 4'-methoxyphenyl analogues. The crystal structure of the mesogenic 4-butylphenyl 4'-butylbenzoyloxybenzoate was described by Haase, Paulus & Pendzialek (1983). The conformation of the mesogenic compounds in the

crystalline state is helpful for understanding the properties in the mesogenic state. A structural description of a mesogenic polysiloxane using the title compound as the side-chain fragment was given by Hotz & Strobl (1989).

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