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Stereochemical Studies of Oligomers. XIX.* 1,5-Pentanediyl Bis(*p*-chlorobenzoate)

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Abstract. $C_{19}H_{18}Cl_2O_4$, $M_r = 381{\cdot}3$, monoclinic, $C2/c$, $a = 32{\cdot}991$ (3), $b = 4{\cdot}654$ (2), $c = 11{\cdot}920$ (2) Å, $\beta = 92{\cdot}20$ (4)°, $V = 1828{\cdot}9$ (9) Å^3, $Z = 4$, $D_x = 1{\cdot}38$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 34{\cdot}21$ cm^{-1}, $F(000) = 792$, room temperature, $R = 0{\cdot}051$ for 1333 unique observed reflections. The conformation of the aliphatic chain of the molecule is all-*trans* while the conformation around the C—O single bond is approximately *gauche* [torsion angle C(7)—O(1)—C(8)—C(9) = 88·3 (3)°] so the two phenyl-ring planes form a dihedral angle of 99·9 (1)°. The carboxylic group is tilted by 2·6 (1)° with respect to the aromatic ring.

Experimental. Crystals obtained from an ethanol–petroleum ether 1:1 mixture. Intensities measured on a Siemens AED single-crystal diffractometer equipped with a General Automation Jumbo 220 computer using Ni-filtered Cu $K\alpha$ radiation and a crystal of about 0·25 × 0·35 × 0·60 mm. Cell parameters from least-squares fit of 25 reflections automatically well centred on the diffractometer. The intensity data evaluated from a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) method. One standard reflection every 50 counts without significant variation. Lp correction, absorption ignored.

Until the end of the second layer all reflections collected; thereafter, for the higher layers, those with $h + k$ odd omitted. In total 2507 reflections measured, 1778 unique ($R_{\rm int} = 0{\cdot}037$), 1333 with $I \geq 2\sigma(I)$ considered observed, $2 \leq \theta \leq 70°$, $-40 \leq h \leq 40$, $0 \leq k \leq 5$, $0 \leq l \leq 14$. Structure solved by direct methods with *MULTAN*80 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement by full-matrix least squares using *SHELX* (Sheldrick, 1976), $\sum w(\Delta F)^2$ minimized, H atoms, found in a ΔF map, refined isotropically, others anisotropically, 150 variables in total, final $R = 0{\cdot}051$ and $wR = 0{\cdot}058$, $w = 1{\cdot}5754/[\sigma^2(F) + 0{\cdot}0210F^2]$. At the end of the refinement $(\Delta/\sigma)_{\max} = 0{\cdot}68$ and $(\Delta\rho)_{\max} = 0{\cdot}38$ e Å^{-3}.

All the calculations were performed on an AT IBM personal computer using the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreetti, 1987). Scattering factors in *SHELX*.

Table 1 reports the positional parameters of the heavy atoms, and Table 2 gives bond distances and angles of the molecule, which is illustrated in Fig. 1†

Table 1. *Atomic fractional coordinates* (×10^4) *and* U_{eq}(×10^4 Å^2)

	x	y	z	$U_{\rm eq}$*
Cl	2706 (1)	11588 (1)	3474 (1)	674 (3)
O(1)	3998 (1)	2408 (4)	6004 (1)	495 (5)
O(2)	4277 (1)	2647 (6)	4329 (2)	801 (7)
C(1)	3681 (1)	5367 (4)	4627 (1)	425 (6)
C(2)	3389 (1)	6252 (5)	5373 (2)	476 (6)
C(3)	3089 (1)	8175 (5)	5027 (2)	507 (6)
C(4)	3079 (1)	9184 (4)	3931 (2)	474 (6)
C(5)	3364 (1)	8305 (5)	3181 (2)	558 (7)
C(6)	3660 (1)	6393 (5)	3536 (2)	528 (7)
C(7)	4016 (1)	3344 (5)	4949 (2)	480 (7)
C(8)	4328 (1)	555 (5)	6428 (2)	515 (6)
C(9)	4669 (1)	2360 (5)	6930 (2)	527 (7)
C(10)	5000	492 (7)	7500	472 (8)

* Hamilton (1959).

Table 2. *Bond distances* (Å) *and angles* (°)

Cl—C(4)	1·735 (4)	C(2)—C(3)	1·385 (4)
O(1)—C(7)	1·334 (3)	C(3)—C(4)	1·387 (3)
O(1)—C(8)	1·464 (4)	C(4)—C(5)	1·384 (4)
O(2)—C(7)	1·201 (4)	C(5)—C(6)	1·376 (4)
C(1)—C(2)	1·398 (4)	C(8)—C(9)	1·509 (4)
C(1)—C(6)	1·385 (3)	C(9)—C(10)	1·534 (3)
C(1)—C(7)	1·491 (4)		

C(7)—O(1)—C(8)	117·3 (2)	Cl—C(4)—C(5)	118·6 (1)
C(6)—C(1)—C(7)	118·0 (1)	C(4)—C(5)—C(6)	119·0 (2)
C(2)—C(1)—C(7)	123·0 (1)	C(1)—C(6)—C(5)	121·4 (2)
C(2)—C(1)—C(6)	119·0 (2)	O(2)—C(7)—C(1)	123·7 (2)
C(1)—C(2)—C(3)	120·2 (2)	O(1)—C(7)—C(1)	112·9 (2)
C(2)—C(3)—C(4)	119·3 (2)	O(1)—C(7)—O(2)	123·4 (2)
Cl—C(4)—C(3)	120·3 (2)	O(1)—C(8)—C(9)	110·0 (2)
C(3)—C(4)—C(5)	121·1 (2)	C(8)—C(9)—C(10)	111·6 (2)

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* Part XVIII: Bocelli & Grenier-Loustalot (1986).

† Lists of structure factors, H-atom coordinates, thermal parameters and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43697 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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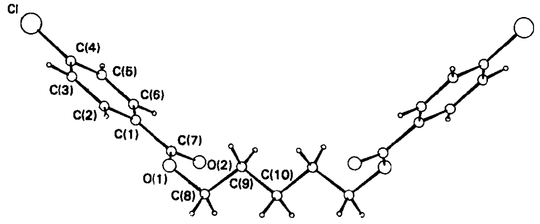

Fig. 1. Numbering of the molecule.

Related literature. An all-*trans* conformation of the chain and a displacement from this arrangement around the C—O single bond was previously observed in the analogous derivative with four carbon atoms in the aliphatic chain (Bocelli & Grenier-Loustalot, 1984).

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Stereochemical Studies of Oligomers. XX.* 2,2′-[1,3-Phenylenebis(oxymethylene)]bisoxirane

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Abstract. $C_{12}H_{14}O_4$, $M_r = 222{\cdot}2$, monoclinic, $P2_1/a$, $a = 23{\cdot}277\ (3)$, $b = 4{\cdot}909\ (2)$, $c = 9{\cdot}750\ (2)$ Å, $\beta = 95{\cdot}04\ (2)^\circ$, $V = 1109{\cdot}8\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}33$ g cm^{-3}, $F(000) = 472$, $\lambda(\text{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 7{\cdot}90$ cm^{-1}, room temperature. The structure was solved by direct methods and refined to $R = 0{\cdot}062$ for 1051 observed reflections. The compound shows a disorder in one of the terminal epoxide rings consisting of two alternate orientations (with filling coefficients of 0·64 and 0·36 respectively) of the ring with one atom in common. The first of these two orientations is fixed with a C atom in the common position, while in the second one the atom which occupies the common position is alternately O and C. The aromatic ring is planar to within 0·004 (5) Å with mean bond lengths and angles of 1·379 (4) Å and 120·0 (5)°. The oxygen atoms bonded to the phenyl ring lie 0·064 (3) and 0·035 (3) Å out of this plane. There are no significant intermolecular contacts.

Experimental. Data collected on a Siemens AED single-crystal diffractometer equipped with a General Automation Jumbo 220 computer, Ni-filtered Cu $K\alpha$ radiation, colorless prism of about 0·04 × 0·05 × 0·13 mm, lattice parameters from 26 reflections automatically well centred on the diffractometer. Intensities evaluated from a profile analysis following a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) method. A reflection monitored every 50 counts indicated only statistical variation in intensity. Lp correction, absorption ignored. 2095 reflections collected, 1814 unique ($R_{\text{int}} = 0{\cdot}019$), 1051 observed $I \geq 2\sigma(I)$, $2 \leq \theta \leq 60^\circ$, $-26 \leq h \leq 26$, $0 \leq k \leq 5$, $0 \leq l \leq 10$, the structure was solved by direct methods and refined first isotropically and then with anisotropic temperature

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* Part XIX: Bocelli & Grenier-Loustalot (1987).

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