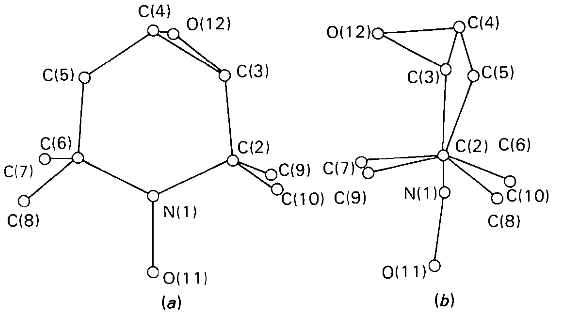

Fig. 2. La numérotation choisie pour désigner les atomes de l'unité asymétrique est précisée sur cette figure. Molécule tanépoxy *A* (les atomes sont représentés par des sphères de rayon arbitraire). (*a*) Projection sur le plan C(2)—N(1)—C(6). (*b*) Projection perpendiculaire à (*a*).

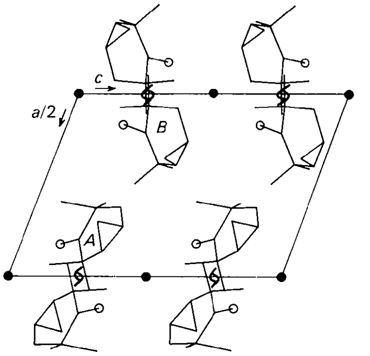

Fig. 3. Projection de la structure sur le plan (010). o: oxygènes de la liaison N—O.

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Stereochemical Studies of Oligomers. VI.* 2,4-Pentanediyl Bis(*m*-chlorobenzoate), $C_{19}H_{18}Cl_2O_4$

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Abstract. $M_r = 381\cdot3$, triclinic, $P\bar{1}$, $a = 11\cdot115\,(3)$, $b = 10\cdot743\,(2)$, $c = 9\cdot124\,(2)$ Å, $\alpha = 109\cdot1\,(3)$, $\beta = 97\cdot2\,(2)$, $\gamma = 65\cdot2°$, $V = 934\cdot5$ Å^3, $Z = 2$, $D_x = 1\cdot36$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5418$ Å, $\mu = 33\cdot48$ cm^{-1}, $F(000) = 396$, $T = 298$ K, $R = 0\cdot053$ for 1953 observed reflections. The propylenic fragment of the chain shows the *gauche–gauche* conformation and the two phenyl rings are on opposite sides with respect to the chain.

Introduction. In preceding papers (Bocelli & Grenier-Loustalot, 1982, 1983 *a,b*) we have analyzed oligomers in which the aliphatic chain was influenced by pendant methyls. In the present case we are interested in studying the conformational changes of the chain as a result of halogen sustituents on the aromatic rings.

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* Part V: Bocelli & Grenier-Loustalot (1983*b*).

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Experimental. Crystals recrystallized at room temperature from acetone, crystal 0·11 × 0·17 × 0·29 mm, Siemens AED single-crystal diffractometer (nickel-filtered Cu *Kα*), General Automation Jumbo 220 computer, 25 reflections used for measuring lattice parameters, $\theta_{\max} = 70°$, no absorption correction, 3491 collected reflections, $-13 \leq h \leq 12$, $-11 \leq k \leq 10$, $0 \leq l \leq 9$, 1953 observed $[I > 2\sigma(I)]$ used in refinement, 1 standard reflection measured every 50, no significant variation, $R_{\rm int} = 0{\cdot}011$; direct methods [*SHELX* (Sheldrick, 1976), which was employed for all the subsequent calculations]; H atoms mainly from ΔF map, rest in calculated positions; full-matrix least squares, $\sum w(\Delta F)^2$ minimized, H atoms isotropic, rest anisotropic, $R = 0{\cdot}053$, $R_w = \sum ||F_o| - |F_c|| w^{1/2}/\sum |F_o| w^{1/2} = 0{\cdot}054$, $w = 1{\cdot}0/(\sigma^2 F_o + 0{\cdot}0027 F_o^2)$; final ΔF map featurless, $\Delta\rho_{\max} = 0{\cdot}29$ e Å^{-3}, $(\Delta/\sigma)_{\max}$ in last cycle = 0·776; scattering factors of *SHELX* used.* All calculations performed on a Gould SEL 32/77 computer.

Discussion. Table 1 reports the final atomic coordinates. Bond distances and angles are given in Table 2. A drawing of the molecule with atom numbering is in Fig. 1.

The conformation of the propylenic fragment of the aliphatic chain exhibits an all-*gauche* conformation, hence the presence of two chlorine atoms does not induce conformational changes of this part of the molecule compared with that found for 2,4-pentanediyl dibenzoate (Bocelli & Grenier-Loustalot, 1983*b*). The chlorines modify, on the contrary, the C(7)—O(1)—C(8)—C(10) and C(13)—O(3)—C(11)—C(10) torsion angles [−158·0 (10) and −113·7 (11)° in the present compound *versus* −100·1 (5) and −157·0 (5)° in the unchlorinated derivative]. These variations affect the conformation of the molecule as a whole, which exhibits an *endo* arrangement with quasi-overlapping of the two aromatic rings in 2,4-pentanediyl dibenzoate, whereas, in the compound under study, the rings lie on opposite sides with respect to the chain. The mean aromatic ring planes subtend an angle of 117·5 (4)°.

Table 1. *Atomic coordinates* (×10⁴) *and isotropic* B_{eq} *values*

	x	*y*	*z*	$B_{\rm eq}^*$ (Å²)
Cl(1)	−1485 (1)	1535 (1)	3753 (2)	7·76 (3)
Cl(2)	5076 (1)	−11078 (1)	−2662 (1)	8·78 (4)
O(1)	3130 (2)	−2757 (2)	2422 (2)	4·42 (5)
O(2)	3899 (3)	−2753 (3)	272 (3)	6·70 (5)
O(3)	2570 (2)	−6064 (2)	1215 (3)	5·06 (5)
O(4)	1295 (3)	−4544 (3)	−80 (4)	8·52 (6)
C(1)	1979 (2)	−752 (4)	1528 (4)	4·18 (5)
C(2)	1984 (4)	107 (5)	690 (5)	5·34 (6)
C(3)	919 (5)	1411 (5)	811 (5)	5·90 (7)
C(4)	−138 (4)	1854 (4)	1758 (5)	5·21 (6)
C(5)	−134 (3)	1002 (4)	2581 (4)	4·60 (6)
C(6)	919 (4)	−309 (4)	2494 (4)	4·53 (6)
C(7)	3121 (3)	−2196 (4)	1315 (4)	4·64 (6)
C(8)	4127 (3)	−4216 (4)	2276 (4)	4·51 (6)
C(9)	5451 (4)	−4162 (6)	2851 (6)	6·18 (6)
C(10)	3606 (3)	−4793 (4)	3224 (5)	4·65 (6)
C(11)	2317 (4)	−4938 (4)	2707 (5)	4·91 (6)
C(12)	1802 (5)	−5397 (5)	3812 (7)	6·13 (7)
C(13)	2016 (4)	−5744 (4)	−58 (5)	5·29 (6)
C(14)	2401 (3)	−7029 (4)	−1457 (4)	4·71 (6)
C(15)	1769 (5)	−6941 (6)	−2854 (6)	6·85 (7)
C(16)	2149 (6)	−8109 (7)	−4152 (7)	7·79 (8)
C(17)	3152 (5)	−9378 (6)	−4104 (6)	6·98 (8)
C(18)	3789 (4)	−9470 (4)	−2743 (5)	5·48 (6)
C(19)	3435 (3)	−8322 (4)	−1410 (4)	4·44 (6)

* According to Hamilton (1959).

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

Cl(1)—C(5)	1·734 (6)	C(4)—C(5)	1·359 (8)
Cl(2)—C(18)	1·739 (6)	C(5)—C(6)	1·388 (6)
O(1)—C(7)	1·332 (6)	C(8)—C(9)	1·517 (6)
O(1)—C(8)	1·465 (5)	C(8)—C(10)	1·498 (7)
O(2)—C(7)	1·188 (6)	C(10)—C(11)	1·506 (6)
O(3)—C(11)	1·459 (7)	C(11)—C(12)	1·514 (9)
O(3)—C(13)	1·323 (6)	C(13)—C(14)	1·485 (8)
O(4)—C(13)	1·205 (6)	C(14)—C(15)	1·388 (7)
C(1)—C(2)	1·380 (8)	C(14)—C(19)	1·393 (6)
C(1)—C(6)	1·381 (7)	C(15)—C(16)	1·365 (9)
C(1)—C(7)	1·506 (6)	C(16)—C(17)	1·362 (9)
C(2)—C(3)	1·384 (7)	C(17)—C(18)	1·362 (7)
C(3)—C(4)	1·367 (8)	C(18)—C(19)	1·373 (7)
C(7)—O(1)—C(8)	117·0 (6)	C(8)—C(10)—C(11)	116·3 (6)
C(11)—O(3)—C(13)	120·3 (8)	O(3)—C(11)—C(10)	107·3 (7)
C(6)—C(1)—C(7)	121·2 (8)	C(10)—C(11)—C(12)	112·6 (7)
C(2)—C(1)—C(7)	118·6 (7)	O(3)—C(11)—C(12)	107·3 (7)
C(2)—C(1)—C(6)	120·2 (10)	O(3)—C(13)—O(4)	123·8 (6)
C(1)—C(2)—C(3)	119·9 (8)	O(4)—C(13)—C(14)	124·0 (7)
C(2)—C(3)—C(4)	120·4 (10)	O(3)—C(13)—C(14)	112·3 (8)
C(3)—C(4)—C(5)	119·4 (10)	C(13)—C(14)—C(19)	120·8 (6)
Cl(1)—C(5)—C(4)	119·8 (9)	C(13)—C(14)—C(15)	120·3 (9)
C(4)—C(5)—C(6)	121·8 (7)	C(15)—C(14)—C(19)	118·8 (6)
Cl(1)—C(5)—C(6)	118·4 (7)	C(14)—C(15)—C(16)	120·4 (10)
C(1)—C(6)—C(5)	118·4 (8)	C(15)—C(16)—C(17)	120·9 (7)
O(2)—C(7)—C(1)	123·3 (7)	C(16)—C(17)—C(18)	119·2 (9)
O(1)—C(7)—C(1)	111·6 (6)	Cl(2)—C(18)—C(17)	119·9 (7)
O(1)—C(7)—O(2)	125·2 (10)	C(17)—C(18)—C(19)	121·7 (10)
O(1)—C(8)—C(10)	105·7 (8)	Cl(2)—C(18)—C(19)	118·4 (6)
O(1)—C(8)—C(9)	109·8 (9)	C(14)—C(19)—C(18)	119·0 (7)
C(9)—C(8)—C(10)	112·3 (6)		

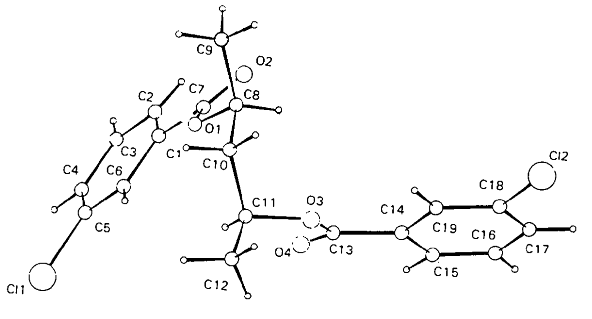

Fig. 1. Projection of the molecule.

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* Lists of structure factors, anisotropic thermal parameters, and H atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38716 (15 pp.). Copies may be obtained through The Executive Secretary International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

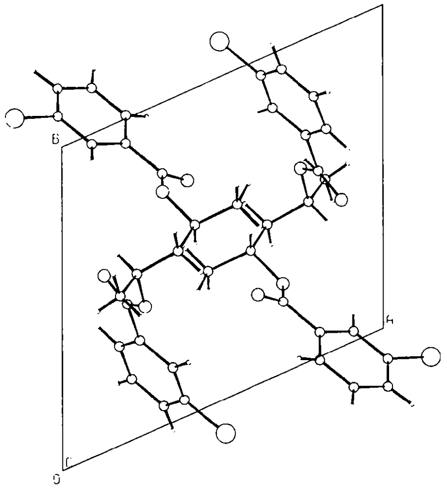

Fig. 2. Projection of the crystal structure along **c**.

A common feature in this type of derivative is the tilt between the carboxylic moieties and their respective phenyl rings [15·4 (4) and 11·4 (5)°]. The methyl groups C(9) and C(12) are in a *trans* arrangement with respect to the C(8), C(10), C(11) sequence. The geometry of the aromatic rings is normal, 1·374 (8), 1·377 (8), 0·91 (5), 0·95 (4) Å, 120·1 (5), 119·8 (5), 120·3 (7), 120·1 (15)° being the average C—C and C—H distances and the C—C—C and C—C—H mean angles for the two rings, respectively. The significant narrowing of the angles opposite to the C=O bonds with respect to the adjacent ones is in good agreement with the notation of Borthwich (1980) and was systematically observed in all the derivatives of this series. The carbon–oxygen bond lengths are in the expected range, whereas the C_{sp^3}—C_{sp^3} distances of the chain are shorter than the value reported by Sutton (1965) [1·537 (5) Å].

No unusually short intermolecular distances were observed. A projection of the crystal structure along **c** is shown in Fig. 2.

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Stereochemical Studies of Oligomers. VII.* 2,3-Butanediyl Bis(*o*-chlorobenzoate), $C_{18}H_{16}Cl_2O_4$

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Abstract. $M_r = 367{\cdot}2$, orthorhombic, *Pbca*, $a = 13{\cdot}257\ (2)$, $b = 17{\cdot}069\ (2)$, $c = 7{\cdot}506\ (1)$ Å, $V = 1698{\cdot}5\ (4)$ Å^3, $Z = 8$, $D_x = 2{\cdot}87$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 73{\cdot}3$ cm^{-1}, $F(000) = 1529$, $T = 298$ K, $R = 0{\cdot}067$ for 1285 observed reflections. The aliphatic chain is far from an all-*trans* conformation and the presence of the chlorine atom causes an increase of the tilt of the carboxylic moiety with respect to the aromatic ring.

Introduction. This structural investigation aims primarily to clarify the change in the chain conformation of the title compound caused by the presence of chlorine atoms attached to the aromatic rings.

Experimental. Transparent prismatic crystals grown by slow evaporation at room temperature from acetone solution, $\theta_{\max} = 70{\cdot}0°$, Siemens AED single-crystal diffractometer (nickel-filtered Cu $K\alpha$), crystal 0·41 × 0·63 × 0·96 mm, 17 reflections used for measuring lattice parameters, intensities obtained by a

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* Part VI: Bocelli & Grenier-Loustalot (1983).

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