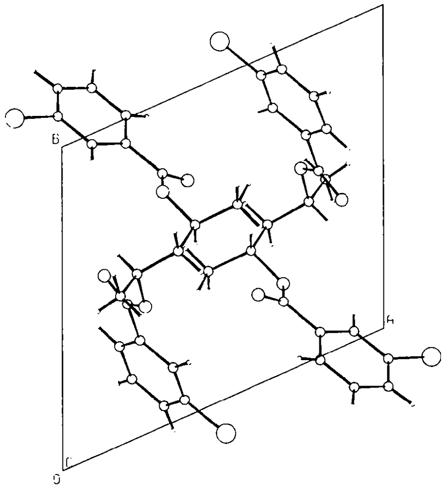

Fig. 2. Projection of the crystal structure along **c**.

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. $M_r = 367·2$, orthorhombic, *Pbca*, $a = 13·257$ (2), $b = 17·069$ (2), $c = 7·506$ (1) Å, $V = 1698·5$ (4) Å^3, $Z = 8$, $D_x = 2·87$ g cm^{-3}, Cu *K*α, $\lambda = 1·5418$ Å, $\mu = 73·3$ cm^{-1}, $F(000) = 1529$, $T = 298$ K, $R = 0·067$ 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, $\theta_{max} = 70·0°$, Siemens AED single-crystal diffractometer (nickel-filtered Cu *K*α), 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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modification of the Lehmann & Larsen (1974) procedure with a program which records the outline of the peak along the θ circle (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979); 1638 independent reflections, $0 \leq h \leq 16$, $0 \leq k \leq 20$, $0 \leq l \leq 9$, corrected for Lorentz, and polarization, not for absorption; structure solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1974), refined by full-matrix least squares with *SHELX* (Sheldrick, 1976); $\sum w(\Delta F)^2$ minimized; anisotropic refinement of heavy atoms, hydrogens found in ΔF map refined isotropically, refinement with 1285 reflections with $I > 2\sigma(I)$ until Δ/σ for each parameter $< 0{\cdot}7$, $R = 0{\cdot}067$ (observed reflections only), $R_w = 0{\cdot}082$, $w = 6{\cdot}106/[\sigma^2(F) + 0{\cdot}033\,F^2]$; final difference map: $\Delta\rho < 0{\cdot}35$ e Å^{-3};* scattering factors of *SHELX* used. Calculations performed on the CDC Cyber 76 computer of the Centro di Calcolo Interuniversitario dell'Italia Nord-Orientale, Casalecchio (Bologna) and on a Gould SEL 32/77 computer.

Discussion. Final positional and equivalent isotropic thermal parameters are listed in Table 1. The molecule is shown in Fig. 1 with its arbitrary numbering. Bond distances and angles are reported in Table 2.

The conformation of the studied molecule exhibits striking differences with respect to the all-*trans* arrangement found in ethylene dibenzoate (Pérez & Brisse, 1976) or to the *trans–gauche* form displayed by ethylene bis(*p*-chlorobenzoate) (Pérez & Brisse, 1975). In the present study, a conformation quite similar to the one trapped in the unchlorinated derivative (Bocelli & Grenier-Loustalot, 1983) is observed. For both cases, the conformation about C(7)—O(2)—C(8)—C(8′) [−145·2 (2)°] is significantly shifted away from a *trans* arrangement. Such an alteration is not exhibited by the conformation about the C(8)—C(8′) bond which is *trans* (from symmetry requirements) in both compounds. Further comparison between the chlorinated and the unchlorinated compounds points towards a non-negligible influence of the halogen atom on the relative orientation of the phenyl ring with respect to the C(2), C(7), O(1), O(2) group [23·7 (1)° in the present case *versus* 9·1 (1)° and 21·0 (1)° in the unchlorinated molecule]. Furthermore, the same conformational analogy between the two compounds is found in the orientation of C(2), C(7), O(1), O(2) with respect to C(9)—C(8)—C(8′)—C(9′) [78·0 (1) (present case) and 75·2 (1) and 77·3 (1)°]. The methyl groups are in a *trans* arrangement with respect to the C(8)—C(8′) bond. Finally, whereas in the present compound the two phenyl rings are parallel (see Fig. 2), in the unchlorinated one the dihedral angle formed by the two aromatic ring planes was 39·61 (4)°.

Table 1. *Fractional atomic parameters* ($\times 10^4$) *with* B_{eq} *values*

	x	y	z	B_{eq}* (Å^2)
Cl	3797 (1)	6656 (1)	7073 (1)	5·13 (2)
O(1)	2384 (2)	5539 (1)	5482 (5)	6·33 (3)
O(2)	749 (1)	5838 (1)	5623 (3)	4·17 (3)
C(1)	2776 (2)	7211 (2)	6374 (4)	3·30 (3)
C(2)	1880 (2)	6883 (1)	5750 (4)	2·99 (3)
C(3)	1109 (2)	7385 (2)	5219 (4)	3·31 (3)
C(4)	1232 (2)	8192 (2)	5302 (4)	6·33 (3)
C(5)	2118 (3)	8503 (2)	5943 (5)	4·17 (3)
C(6)	2880 (3)	8016 (2)	6481 (5)	3·30 (3)
C(7)	1729 (2)	6016 (1)	5622 (4)	2·99 (3)
C(8)	475 (2)	5000	5550 (5)	3·31 (3)
C(9)	334 (3)	4722 (2)	7448 (6)	3·90 (3)

* According to Hamilton (1959).

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

Cl—C(1)	1·733 (3)	C(2)—C(7)	1·496 (3)
O(1)—C(7)	1·195 (3)	C(3)—C(4)	1·389 (5)
O(2)—C(7)	1·334 (3)	C(4)—C(5)	1·376 (5)
O(2)—C(8)	1·477 (2)	C(5)—C(6)	1·369 (5)
C(1)—C(2)	1·394 (4)	C(8)—C(9)	1·513 (6)
C(1)—C(6)	1·383 (5)	C(8)—C(8′)	1·506 (4)
C(2)—C(3)	1·392 (4)		
C(7)—O(2)—C(8)	117·4 (1)	C(4)—C(5)—C(6)	119·9 (3)
Cl—C(1)—C(6)	116·6 (2)	C(1)—C(6)—C(5)	120·8 (3)
Cl—C(1)—C(2)	123·2 (2)	O(2)—C(7)—C(2)	110·8 (2)
C(2)—C(1)—C(6)	120·2 (3)	O(1)—C(7)—C(2)	125·6 (2)
C(1)—C(2)—C(7)	122·2 (2)	O(1)—C(7)—O(2)	123·5 (2)
C(1)—C(2)—C(3)	118·3 (2)	O(2)—C(8)—C(8′)	103·1 (1)
C(3)—C(2)—C(7)	119·5 (2)	O(2)—C(8)—C(9)	107·4 (2)
C(2)—C(3)—C(4)	120·8 (2)	C(9)—C(8)—C(8′)	114·4 (2)
C(3)—C(4)—C(5)	119·9 (3)		
Cl—C(1)—C(2)—C(7)	−0·4 (4)	C(2)—C(7)—O(2)—C(8)	−178·5 (2)
C(1)—C(2)—C(7)—O(1)	−24·5 (4)	O(1)—C(7)—O(2)—C(8)	3·6 (4)
C(3)—C(2)—C(7)—O(1)	155·0 (3)	C(7)—O(2)—C(8)—C(9)	93·7 (3)
C(1)—C(2)—C(7)—O(2)	157·6 (3)	C(7)—O(2)—C(8)—C(8′)	−145·2 (2)
C(3)—C(2)—C(7)—O(2)	−22·9 (3)		

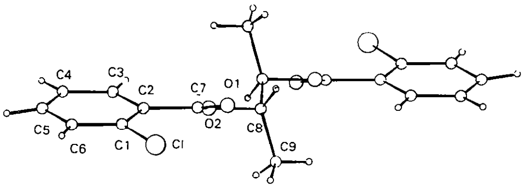

Fig. 1. Projection of the molecule.

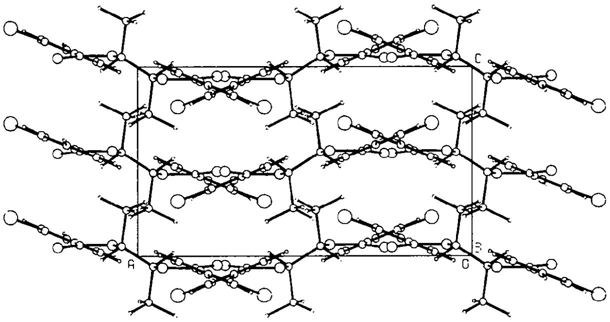

Fig. 2. Packing of the crystal on (010).

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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 38717 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The geometrical parameters of the carboxylic moiety are comparable with those found in similar derivatives, and those of the aromatic ring are normal [mean values: C—C 1·385 (4), C—H 0·91 (5) Å, C—C—C 119·9 (4), C—C—H 119·9 (7)°]. The C_{sp^3}—C_{sp^3} distances of the chain [C(8)—C(9) 1·513 (6), C(8)—C(8′) 1·506 (4) Å] show a small lengthening with respect to the values found in the two derivatives analyzed by Pérez & Brisse (1975, 1976) [1·493 (2), 1·499 (3) Å].

The packing of the molecules in the crystal, illustrated in Fig. 2, is mainly determined by van der Waals interactions.

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Stereochemical Studies of Oligomers. VIII.* Ethylene Bis(*m*-chlorobenzoate), $C_{16}H_{12}Cl_2O_4$

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Abstract. M_r = 339·17, monoclinic, $P2_1/c$, a = 6·136 (1), b = 16·724 (2), c = 7·351 (2) Å, β = 81·4 (1)°, V = 745·9 (3) Å^3, Z = 4, D_x = 3·02 g cm^{-3}, Cu $K\alpha$, $\bar{\lambda}$ = 1·5418 Å, μ = 82·5 cm^{-1}, $F(000)$ = 696, T = 298 K, R = 0·054 for 829 observed reflections. The molecule shows the *trans–trans* conformation and the usual tilt of the carboxylic moiety with respect to the aromatic plane. A shortening of the C_{sp^3}—C_{sp^3} bond of the chain is observed.

Introduction. As part of structural studies on the influence of bulky substituents on the conformation of the aliphatic chain of oligomers, we now report the crystal analysis results of a derivative which presents a chlorine atom in the *meta* position of the aromatic ring.

Experimental. Crystals recrystallized from petroleum ether, prismatic specimen, 0·3 × 0·4 × 0·7 mm; crystal system and cell dimensions automatically obtained from 24 reflections measured on a Siemens AED single-crystal diffractometer with programs written by Belletti, Ugozzoli, Cantoni & Pasquinelli (1979); a modification of the Lehmann & Larsen (1974) procedure recording the outline of the peaks along the θ circle was employed to collect data, $3{\cdot}0 < \theta < 70{\cdot}0°$; 1 standard reflection monitored every 50 measurements, no apparent decay; correction for Lorentz and polarization, not for absorption; 1584 collected reflections, $-6 \le h \le 7$, $0 \le k \le 20$, $0 \le l \le 7$, 1429 unique, $R_{\rm int}$ = 0·040, 829 with $I > 2\sigma(I)$ considered observed and used in refinement; full-matrix least squares minimizing $\sum w|\Delta F|^2$, structure solved by direct methods with *SHELX* (Sheldrick, 1976), unit weights as the weighting scheme $w^{-1} = [\sigma^2(F_o + kF_o^2)]$ with different k values gave unsatisfactory results; heavy atoms anisotropic, hydrogens (from ΔF map) isotropic, R = 0·054, R_w = 0·066; final difference synthesis: $\Delta\rho \le 0{\cdot}25$ e Å^{-3}, $(\Delta/\sigma)_{\rm max}$ = 0·62; atomic scattering factors from *SHELX*.† Calculations performed on a Gould SEL 32/77 computer.

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

† 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 38718 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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