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.

The support by CNR and CNRS through an International Scientific Project (ERA 895) is acknowledged.

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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, $\beta = 81·4\ (1)°$, $V = 745·9\ (3)$ Å^3, $Z = 4$, $D_x = 3·02$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 82·5$ 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, $3·0 < \theta < 70·0°$; 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: $\Delta\rho \le 0·25$ 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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Discussion. The final positional parameters and equivalent isotropic *B* values are given in Table 1. Table 2 reports bond distances and angles and selected torsion angles.

The conformation of the molecule (*cf.* Fig. 1) is all-*trans* and therefore completely different from the *trans–gauche* arrangement found in ethylene bis(*p*-chlorobenzoate) (Pérez & Brisse, 1975). Hence, moving the chlorine atom from the *para* to the *meta* position induces a conformational shift towards another stable state similar to that found for the unchlorinated derivative (Pérez & Brisse, 1976). The dihedral angle between the aromatic ring and the carboxylic moiety [3·9 (2)°] is practically equal to that found in ethylene dibenzoate (~3°). The C—Cl bond [1·735 (4) Å] falls within the range found in comparable systems, though the C(8)—C(8′) bond [1·484 (7) Å] is rather short for a single C_{sp^3}—C_{sp^3} distance. This fact is not surprising and has often been found in analogous derivatives (Pérez & Brisse, 1975, 1976, 1977*a*,*b*; Bocelli & Grenier-Loustalot, 1982*a*,*b*). The mean C—C distance of the aromatic ring [1·379 (3) Å] is exactly the same as the value computed from 78 C_{sp^2}—C_{sp^2} distances by Brisse & Sygusch (1974). A small enlargement of the angle C(2)—C(1)—C(6) [122·8 (4)°] is probably due to the presence of the chlorine atom which is out of the ring plane by 0·022 Å. The angle O(2)—C(8)—C(8′) [105·6 (3)°] is smaller than the accepted value but is comparable with those found in the already quoted derivatives.

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

	x	y	z	B_{eq}* (Å^2)
Cl	5534 (2)	3991 (1)	−145 (2)	5·12 (2)
O(1)	9857 (5)	628 (2)	−2108 (6)	5·54 (6)
O(2)	6555 (5)	895 (2)	−559 (5)	4·24 (5)
C(1)	7585 (7)	3321 (2)	−1021 (7)	3·62 (4)
C(2)	9580 (8)	3620 (2)	−1814 (8)	4·57 (4)
C(3)	11220 (8)	3087 (3)	−2488 (9)	4·89 (4)
C(4)	10855 (7)	2272 (3)	−2369 (8)	4·25 (4)
C(5)	8818 (6)	1989 (2)	−1555 (7)	3·46 (4)
C(6)	7154 (7)	2516 (2)	−874 (7)	3·39 (4)
C(7)	8506 (8)	1106 (3)	−1467 (9)	4·01 (3)
C(8)	6148 (8)	46 (2)	−475 (11)	4·88 (3)

* According to Hamilton (1959).

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

Cl—C(1)	1·735 (4)	C(2)—C(3)	1·380 (7)
C(1)—C(7)	1·196 (6)	C(3)—C(4)	1·382 (7)
O(2)—C(7)	1·328 (6)	C(4)—C(5)	1·386 (6)
O(2)—C(8)	1·441 (5)	C(5)—C(6)	1·385 (5)
C(1)—C(2)	1·369 (6)	C(5)—C(7)	1·489 (6)
C(1) C(6)	1·373 (5)	C(8)—C(8′)	1·484 (7)
C(7)—O(2)—C(8)	114·9 (4)	C(4)—C(5)—C(6)	120·5 (4)
Cl—C(1)—C(6)	118·9 (4)	C(6)—C(5)—C(7)	122·2 (4)
Cl—C(1)—C(2)	118·3 (3)	C(1)—C(6)—C(5)	118·2 (5)
C(2)—C(1)—C(6)	122·8 (4)	O(2)—C(7)—C(5)	112·7 (4)
C(1)—C(2)—C(3)	118·3 (4)	O(1)—C(7)—C(5)	124·6 (5)
C(2)—C(3)—C(4)	120·8 (5)	O(1)—C(7)—O(2)	122·7 (5)
C(3)—C(4)—C(5)	119·4 (5)	O(2)—C(8)—C(8′)	105·6 (3)
C(4)—C(5)—C(7)	117·3 (4)		
C(4)—C(5)—C(7)—O(2)	175·9 (4)	C(5)—C(7)—O(2)—C(8)	178·5 (4)
C(4)—C(5)—C(7)—O(1)	−2·9 (8)	O(1)—C(7)—O(2)—C(8)	−2·7 (7)
C(6)—C(5)—C(7)—O(2)	−4·7 (7)	C(7)—O(2)—C(8)—C(8′)	−176·2 (4)
C(6)—C(5)—C(7)—O(1)	176·5 (5)		

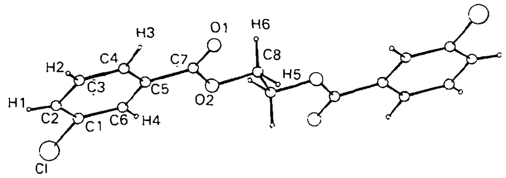

Fig. 1. Projection of the molecule on (100).

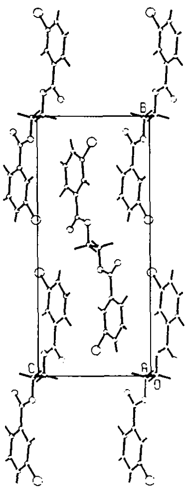

Fig. 2. Packing of the compound along **a**.

The molecules are held in the crystal mainly by van der Waals interactions. Fig. 2 shows the packing.

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2,3,4a*β*,8a*β*-Tetramethyl-4a,5,8,8a-tetrahydro-1-naphthoquin-4α-yl Benzoate,* $C_{21}H_{24}O_3$

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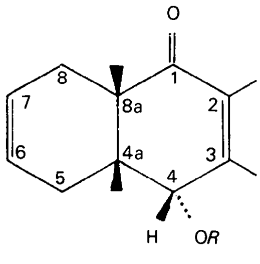
Abstract. $M_r = 324{\cdot}4$, triclinic, $P\bar{1}$, $a = 9{\cdot}6506\ (7)$, $b = 11{\cdot}0330\ (8)$, $c = 8{\cdot}5530\ (7)$ Å, $\alpha = 93{\cdot}078\ (7)$, $\beta = 91{\cdot}122\ (6)$, $\gamma = 94{\cdot}345\ (6)°$, $V = 906{\cdot}5\ (1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}189$ g cm^{-3}, $F(000) = 348$, $T = 294$ K, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}73$ cm^{-1}, $R = 0{\cdot}048$ for 2961 reflexions. The tetrahydronaphthoquinol ring system has a conformation typical for derivatives with a 4α substituent.

Introduction. The crystal structure of the benzoate has been determined in the course of a study of the solid-state photochemistry of substituted tetrahydronaphthoquinols (Secco & Trotter, 1982).



$R = C_6H_5CO$–

Experimental. CAD-4 diffractometer, crystal faces {100}, {001}, {101}, {010}, dimensions 0·4 × 0·6 × 0·7 mm, D_m not measured, monochromatized Mo $K\alpha$ radiation, cell parameters from $\sin^2\theta$ values for 21 reflections ($\theta = 5 - 19°$), intensity data for $0 < \theta \leq 27{\cdot}5°$, ω–(4/3)θ scans, ω scan angle $(0{\cdot}85 + 0{\cdot}35 \tan\theta)°$, extended by 25% in each direction for background, scan speeds 1 to 10° min^{-1}, horizontal aperture $(2{\cdot}00 + \tan\theta)$ mm, vertical aperture 4 mm, three standard reflexions, Lp but no absorption corrections; 4130 reflections, 2961 (72%) with $I \geq 3\sigma(I)$, $\sigma^2(I) = S + 2B + [0{\cdot}04(S-B)]^2$, S = scan count, B = time-corrected background. Direct methods, full-matrix least squares on F, H atoms from a difference map refined isotropically,† $w = (0{\cdot}0329 + 0{\cdot}02117|F_o| - 0{\cdot}00227|F_o|^2 + 0{\cdot}000222|F|^3)^{-1}$, final $R = 0{\cdot}048$, $R_w = 0{\cdot}069$ for 2961 reflexions with $I \geq 3\sigma(I)$, $R = 0{\cdot}066$ for all 4130 reflexions, $S = 0{\cdot}98$, mean and maximum shifts on final cycle 0·04 and 0·60σ, final difference synthesis ±0·25 e Å^{-3}. Scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). No correction for secondary extinction.

Discussion. Final atomic parameters in Table 1.‡

The conformation of the tetrahydronaphthoquinol ring system (Fig. 1) is typical of that of other molecules in the series which have 4α substituents, *i.e. anti* or *trans* to the bridgehead 4*β*-methyl groups (Greenhough & Trotter, 1981; Secco & Trotter, 1982). The ring system is twisted, with a C(4a1)—C(4a)—C(8a)—C(8a1) torsion angle of 61·7 (2)°, the corresponding value in the parent hydroxy compound being 63·0 (2)° (Greenhough & Trotter, 1980). The 4α-C_6H_5COO— substituent occupies a pseudo-equatorial position on the half-chair cyclohexenone ring. The parameters relevant to photochemical reactivity are: H1(5)···C(3) = 2·79 (2) Å, $\tau_C = 53°$, $\Delta_C = 79{\cdot}2\ (4)°$, C(2)···C(5) = 3·335 (2) Å [corresponding values in the parent tetramethyl-hydroxy compound are 2·78 (2) Å, 52°,

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* IUPAC name: 2,3,4a*β*,8a*β*-tetramethyl-1(4*H*)-oxo-4a,5,8,8a-tetrahydro-4α-naphthyl benzoate.

† Computer programs include locally written programs for data processing and locally modified versions of *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *ORFLS* (Busing, Martin & Levy, 1962) and *ORFFE* (Busing, Martin & Levy, 1964), *FORDAP* (A. Zalkin), and *ORTEP*II (Johnson, 1976).

‡ Lists of structure factors, thermal parameters, bond distances and angles involving H atoms, and a packing diagram have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38729 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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