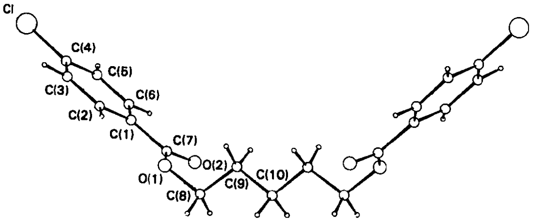

Fig. 1. Numbering of the molecule.

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, $\beta = 95{\cdot}04\ (2)°$, $V = 1109{\cdot}8\ (5)$ 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 $I \geq 2\sigma(I)$, $2 \leq \theta \leq 60°$, $-26 \leq h \leq 26$, $0 \leq k \leq 5$, $0 \leq l \leq 10$, 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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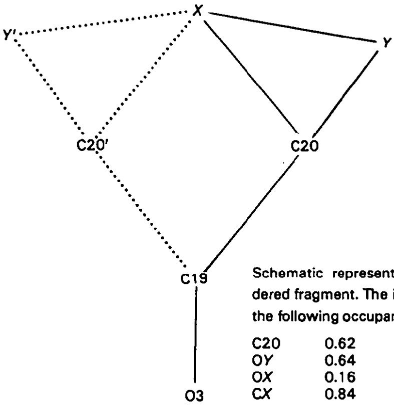
factors for the non-H atoms by full-matrix least squares using *SHELX* (Sheldrick, 1976). The atoms of the disordered epoxide ring were considered to be C in site *X* and O in *Y* and *Y′* (occupancy = 0·5) (see scheme) until the refinement decreased to about $R = 0{\cdot}09$. Refinement of the occupancies of C20 and C20′ gave values of 0·64 and 0·36 respectively. Further refinement showed an alternation of C and O atoms present only in site *Y′* (occupancy 0·16 and 0·20 respectively), while in the first orientation the site *Y* is occupied only by O (occupancy 0·64). A ΔF map computed after the refinement of O and C in *X* (filling coefficient 0·16 and 0·84 respectively) revealed practically all H atoms (three were placed in their theoretical positions) and all refined isotropically (those of the disordered fragment with the occupancies of respective C atoms). The refinement (eight reflections with high ΔF value and low θ omitted) reduced R to 0·062 and wR to 0·065 with $w = 1{\cdot}6211[\sigma^2(F) + 0{\cdot}000998F^2]$, $(\Delta/\sigma)_{\max} = 0{\cdot}23$, $\Delta\rho = 0{\cdot}20\ \text{e Å}^{-3}$, scattering factors those of *SHELX*. The final atomic coordinates of the molecule, which is illustrated in Fig. 1, are in Table 1.* Bond distances and valence angles are given in Table 2. All the calculations were performed on a Gould SEL 32/77 computer.



Schematic representation of the disordered fragment. The indicated sites show the following occupancies:

C20	0.62	C20′	0.36
O*Y*	0.64	O*Y′*	0.20
O*X*	0.16	C*Y′*	0.16
C*X*	0.84		

Related literature. The geometrical parameters of the epoxide rings differ from values reported for 17 rings of this type by Foces-Foces, Cano & García-Blanco (1977).

The authors thank the CNR and CNRS which supported this work through an International Scientific Project.

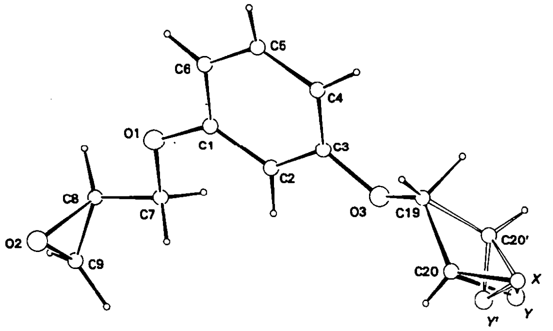


Fig. 1. Projection of the compound. The H atoms of the disordered fragment have been omitted.

Table 1. *Atomic fractional coordinates* (×10⁴) *and* U_{eq} *values* (Å² ×10⁴)

	x	*y*	*z*	U_{eq}
O1	4438 (1)	2882 (6)	3771 (3)	573 (12)
O2	5363 (1)	298 (7)	1975 (4)	728 (14)
O3	2601 (1)	6703 (7)	2399 (4)	782 (13)
O*Y*	1479 (4)	8078 (36)	371 (19)	812 (35)
O*Y′*	1741 (11)	8305 (86)	294 (39)	918 (110)
O*X*	1470 (3)	10941 (17)	623 (10)	1067 (35)
C1	3981 (2)	4550 (9)	3989 (5)	501 (16)
C2	3488 (2)	4752 (10)	3092 (5)	519 (17)
C3	3065 (2)	6608 (10)	3379 (5)	551 (19)
C4	3126 (2)	8221 (11)	4539 (5)	580 (20)
C5	3618 (2)	7953 (11)	5415 (6)	594 (18)
C6	4043 (2)	6162 (10)	5157 (5)	565 (19)
C7	4373 (2)	1019 (11)	2660 (6)	551 (19)
C8	4905 (2)	−680 (10)	2732 (6)	568 (18)
C9	5057 (3)	−2150 (14)	1548 (7)	743 (24)
C19	2249 (3)	9057 (14)	2320 (8)	776 (26)
C(20)	2008 (5)	9609 (25)	882 (16)	581 (42)
C20′	1711 (11)	8655 (51)	1526 (24)	971 (91)
C*X*	1470 (3)	10941 (17)	623 (10)	1067 (34)
C*Y′*	1741 (11)	8305 (86)	294 (39)	918 (110)

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

O1–C1	1·374 (5)	C1–C6	1·384 (7)
O1–C7	1·416 (6)	C2–C3	1·388 (7)
O2–C8	1·432 (6)	C3–C4	1·378 (7)
O2–C9	1·440 (8)	C4–C5	1·373 (7)
O3–C3	1·378 (5)	C5–C6	1·363 (7)
O3–C19	1·415 (8)	C7–C8	1·490 (7)
O*Y*–C20	1·491 (17)	C8–C9	1·432 (9)
O*Y*–C*X*	1·427 (20)	C19–C20	1·488 (17)
O*Y′*–C20′	1·222 (45)	C19–C20′	1·427 (25)
O*Y′*–C*X*	1·486 (41)	C20–C*X*	1·415 (14)
C1–C2	1·384 (6)	C20′–C*X*	1·504 (26)

C1–O1–C7	118·1 (3)	O2–C8–C7	116·2 (4)
C8–O2–C9	59·8 (3)	C7–C8–C9	120·5 (4)
C3–O3–C19	118·7 (4)	O2–C8–C9	60·4 (3)
C20–O*Y*–C*X*	58·0 (6)	O2–C9–C8	59·8 (3)
C20′–O*Y′*–C*X*	66·6 (21)	O3–C19–C20′	113·1 (11)
O1–C1–C6	116·2 (4)	O3–C19–C20	111·6 (27)
O1–C1–C2	123·7 (4)	O*Y*–C20–C19	117·1 (10)
C2–C1–C6	120·0 (4)	C19–C20–C*X*	120·4 (11)
C1–C2–C3	118·9 (4)	O*Y*–C20–C*X*	58·7 (7)
O3–C3–C2	114·1 (4)	O*Y′*–C20′–C19	115·5 (22)
C2–C3–C4	121·3 (4)	C19–C20′–C*X*	118·6 (18)
O3–C3–C4	124·6 (4)	O*Y′*–C20′–C*X*	65·2 (21)
C3–C4–C5	118·4 (4)	O*Y′*–C*X*–C20′	48·2 (18)
C4–C5–C6	121·7 (5)	O*Y*–C*X*–C20	63·3 (7)
C1–C6–C5	119·7 (4)		
O1–C7–C8	107·1 (4)		

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* Lists of structure factors, H coordinates, thermal parameters and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43732 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Stereochemical Studies of Oligomers. XXI.* 1,12-Dodecanediyl Bis(*m*-chlorobenzoate)

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Abstract. $C_{26}H_{32}Cl_2O_4$, $M_r = 479{\cdot}4$, monoclinic, $P2_1/a$, $a = 13{\cdot}239\ (2)$, $b = 4{\cdot}070\ (3)$, $c = 23{\cdot}474\ (3)$ Å, $\beta = 100{\cdot}91\ (3)°$, $V = 1242{\cdot}0\ (9)$ Å^3, $Z = 2$, $D_x = 1{\cdot}28$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 26{\cdot}15$ cm^{-1}, $F(000) = 508$, room temperature, $R = 0{\cdot}060$ for 1105 unique observed reflections. The conformation of the molecule is all-*trans*. The carboxylate groups are tilted by 6·4 (2)° with respect to the aromatic rings.

Experimental. Intensities measured on a Siemens AED single-crystal diffractometer equipped with a General Automation Jumbo 220 computer employing Ni-filtered Cu $K\alpha$ radiation and a prismatic specimen of about 0·35 × 0·40 × 0·55 mm. Lattice parameters from least-squares fit of (θ,χ,φ) angles of 24 reflections automatically centered on the diffractometer. Intensities evaluated from a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) method. The check reflection, monitored every 50 counts, did not vary significantly. Lp correction, absorption ignored.

2734 reflections measured, 2579 unique ($R_{\text{int}} = 0{\cdot}18$), 1105 with $I \geq 2\sigma(I)$ considered observed, $3 \leq \theta \leq 70°$, $-16 \leq h \leq 15$, $0 \leq k \leq 4$, $0 \leq l \leq 28$. Structure solved by direct methods with *MULTAN*80 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), $\sum w(\Delta F)^2$ minimized, H atoms found from a ΔF map and refined isotropically, other atoms refined anisotropically, 209 variables in total, final $R = 0{\cdot}060$ and $wR = 0{\cdot}068$, $w = 1{\cdot}7379/(\sigma^2F + 0{\cdot}0243F^2)$. In the last cycle of the refinement $(\Delta/\sigma)_{\max}$ was 0·26 for all atoms and $\Delta\rho = 0{\cdot}29$ e Å^{-3}.

Table 1. *Atomic fractional coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^4)

	x	y	z	U_{eq}*
Cl	720 (1)	1495 (6)	9488 (1)	1007 (9)
O(1)	250 (2)	−3765 (11)	7431 (1)	676 (14)
O(2)	1781 (3)	−5463 (13)	7277 (2)	846 (16)
C(1)	1701 (3)	−2550 (15)	8147 (2)	593 (20)
C(2)	1066 (4)	−1345 (16)	8502 (2)	654 (23)
C(3)	1506 (4)	40 (16)	9032 (2)	705 (24)
C(4)	2563 (4)	110 (21)	9213 (2)	869 (28)
C(5)	3172 (4)	−1076 (21)	8852 (3)	937 (31)
C(6)	2752 (4)	−2508 (18)	8325 (2)	762 (27)
C(7)	1272 (3)	−4098 (16)	7575 (2)	621 (23)
C(8)	−222 (4)	−5083 (25)	6864 (2)	678 (27)
C(9)	−1360 (3)	−4384 (24)	6774 (2)	639 (26)
C(10)	−1895 (4)	−5239 (24)	6153 (2)	622 (27)
C(11)	−3045 (3)	−4577 (22)	6044 (2)	591 (24)
C(12)	−3579 (3)	−5314 (24)	5422 (2)	593 (26)
C(13)	−4731 (3)	−4617 (23)	5314 (2)	595 (27)

* Hamilton (1959).

All the calculations were performed on an AT IBM personal computer using the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreetti, 1987). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

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

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

† Lists of structure factors, H-atom coordinates, anisotropic thermal parameters and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43757 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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