Acta Cryst. (1984). C**40**, 1391–1394

Stereochemical Studies of Oligomers. X.* The Structure of 1,6-Hexanediyl Bis[*o*-(*m*- and *p*-)chlorobenzoates] (HDDO, HDDM and HDDP), $C_{20}H_{20}Cl_2O_4$

By Gabriele Bocelli

Centro di Studio per la Strutturistica Diffrattometrica del CNR, Via M. D'Azeglio 85, 43100 *Parma, Italy*

and Marie Florence Grenier-Loustalot

Institut Universitaire de Recherche Scientifique, Avenue Philippon, 64000 *Pau, France*

(*Received* 25 *May* 1983; *accepted* 5 *March* 1984)

Abstract. $M_r = 395{\cdot}28$, Ni-filtered Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, room temperature. HDDO: monoclinic, $P2_1/c$, $a = 8{\cdot}145$ (2), $b = 20{\cdot}359$ (3), $c = 12{\cdot}000$ (2) Å, $\beta = 103{\cdot}99$ (2)°, $V = 1930{\cdot}9$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}36$ g cm^{-3}, $\mu = 32{\cdot}58$ cm^{-1}, $F(000) = 824$, $R = 0{\cdot}060$ for 2882 observed reflections. HDDM: triclinic, $P\bar{1}$, $a = 8{\cdot}354$ (1), $b = 7{\cdot}365$ (1), $c = 8{\cdot}431$ (2) Å, $\alpha = 71{\cdot}21$ (2), $\beta = 97{\cdot}48$ (2), $\gamma = 95{\cdot}84$ (3)°, $V = 485{\cdot}9$ (2) Å^3, $Z = 1$, $D_x = 1{\cdot}35$ g cm^{-3}, $\mu = 32{\cdot}4$ cm^{-1}, $F(000) = 206$, $R = 0{\cdot}045$ for 1251 observed reflections. HDDP: triclinic, $P1$, $a = 10{\cdot}483$ (2), $b = 7{\cdot}760$ (2), $c = 6{\cdot}746$ (2) Å, $\alpha = 107{\cdot}77$ (2), $\beta = 98{\cdot}32$ (2), $\gamma = 70{\cdot}43$ (3)°, $V = 491{\cdot}9$ (2) Å^3, $Z = 1$, $D_x = 1{\cdot}33$ g cm^{-3}, $\mu = 31{\cdot}97$ cm^{-1}, $F(000) = 206$, $R = 0{\cdot}060$ for 1455 observed reflections. An all-*trans* conformation characterizes the aliphatic chain of the three compounds. The tilt of the carboxylic groups with respect to the aromatic rings is greatly increased in the *ortho* derivative. All bond lengths and angles conform with those of similar compounds; the bond distances in the aliphatic chain are slightly shorter than the ideal value, as expected.

Introduction. These structures were determined as part of systematic studies on the influence of substituents on the conformation of oligomers which may be considered as 'model compounds' of polymers (Brisse, Marchessault & Pérez, 1980). In the analogous butane derivatives (Bocelli & Grenier-Loustalot, 1984) we note that the position of the chlorine atoms on the phenyl rings seems to influence the mutal rotation of fragments of the molecule while it remains difficult to rationalize the conformational changes of the aliphatic chains. In order to analyse whether the conformational variations observed in the butane derivatives remain the same when the aliphatic chain is longer, the crystal structures of the title compounds were analysed and the results are reported in this paper.

Experimental. For all compounds: Siemens AED single-crystal diffractometer, General Automation Jumbo 220 computer, cell parameters obtained from least-squares fit of 23–27 reflections automatically centred on the diffractometer with programs written by Belletti, Ugozzoli, Cantoni & Pasquinelli (1979), 1 check reflection measured every 50 counts, profile analysis following the Lehmann & Larsen (1974) notation modified by Belletti *et al.* (1979), Lp corrections, absorption ignored.

HDDO: colourless prismatic crystals from diethyl ether solution, 0·06 × 0·18 × 0·20 mm, $2 \leq \theta \leq 70°$, $-9 \leq h \leq 9$, $0 \leq k \leq 24$, $0 \leq l \leq 14$, 1 check reflection showing only statistical fluctuations in intensity, 4047 reflections collected, 3721 independent ($R_{\rm int} = 0{\cdot}024$), 2887 with $I \geq 2\sigma(I)$ considered observed, direct methods (*SHELX*; Sheldrick, 1976), refinement with anisotropic thermal parameters for non-H atoms, hydrogens found in ΔF map and refined isotropically, 299 parameters refined, final $R = 0{\cdot}060$ for 2882 reflections, five eliminated because of probable extinction, $R_w = 0{\cdot}072$, $w = 1{\cdot}0/(\sigma^2 F + 0{\cdot}00562 F^2)$, $\sum w(\Delta F)^2$ minimized, $(\Delta/\sigma)_{\rm max} = 0{\cdot}96$, $\Delta\rho_{\rm max} = 0{\cdot}39$ e Å^{-3}.

HDDM: colourless prisms by slow evaporation at room temperature of acetone solution, 0·06 × 0·011 × 0·36 mm, 1 check reflection < 0·6% average fluctuation during data collection, 1771 unique reflections $2 \leq \theta \leq 70°$, $-10 \leq h \leq 10$, $-8 \leq k \leq 8$, $0 \leq l \leq 9$, 1251 considered observed at the $2\sigma(I)$ level, structure determined by direct methods (*SHELX*; Sheldrick, 1976) in centrosymmetric space group $P\bar{1}$ which was chosen on the basis of E statistics and confirmed by structure refinement, all non-H atoms refined isotropically then anisotropically by full-matrix least squares, H (from Δ map) included in last cycles with isotropic thermal parameters, final $R = 0{\cdot}045$, $R_w = 0{\cdot}047$ for observed data, $w = 1{\cdot}0/(\sigma^2 F + 0{\cdot}05028 F^2)$, $\sum w(\Delta F)^2$ minimized, parameters refined 158, $(\Delta/\sigma)_{\rm max} = 0{\cdot}57$ (for heavy atoms), $\Delta\rho_{\rm max} = 0{\cdot}047$ e Å^{-3}.

---

* Part IX: Bocelli & Grenier-Loustalot (1984).

0108-2701/84/081391-04$01.50

HDDP: crystal cut from a larger one, $0{\cdot}2 \times 0{\cdot}7 \times 0{\cdot}11$ mm, $2 \le \theta \le 70°$, $-12 \le h \le 12$, $-8 \le k \le 9$, $0 \le l \le 7$, 1 check reflection showing only statistical intensity fluctuations, 1727 reflections collected, 1455 considered observed having $I \ge 2\sigma(I)$, structure solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1974), anisotropic block-matrix least squares, H (from ΔF synthesis) isotropic, aromatic rings refined as rigid bodies, final $R = 0{\cdot}060$, $R_w = 0{\cdot}064$, $w = 1{\cdot}0303/(\sigma^2 F + 0{\cdot}0023 F^2)$, refinement calculations with *SHELX* (Sheldrick, 1976), parameters refined 224 and 68 in the two last blocks, $(\Delta/\sigma)_{\max} = 0{\cdot}086$, $\Delta\rho_{\max} = 0{\cdot}43$ e Å^{-3}.

All scattering factors from *SHELX*; all the calculations were performed on a Gould-SEL 32/77 computer, those with *MULTAN* on a CDC Cyber 76 computer of the CINECA, Casalecchio (Bologna).*

Discussion. Atomic coordinates are given in Table 1. Bond lengths and internal angles (Table 2) for the aromatic rings in HDDO and HDDM are normal, being 1·387 (5), 1·387 (3) Å and 120·0 (4), 120·0 (5)°; average 1·383 (3) Å and 120·0 (6)°. Views of the molecules are shown in Figs. 1, 2 and 3.

The C—Cl distances in HDDO and HDDP show a little shortening with respect to the value reported by Domenicano, Vaciago & Coulson (1975) [1·7404 (11) Å] but are in good agreement with the values found in preceding papers of this series. The chlorine atoms are out of plane of the phenyl groups by 0·012 (2), 0·046 (2) Å in HDDO, 0·040 (1) Å in HDDM and 0·051 (2), 0·031 (3) Å in HDDP. The deformation of the aromatic-ring skeletons from the ideal D_{6h} geometry, caused by the inductive and resonance effects of chlorines (Domenicano *et al.*, 1975; Domenicano, Mazzeo & Vaciago, 1976; Domenicano & Murray-Rust, 1979), is still present and causes an enlargement of the internal angles at the chlorines with respect to the adjacent ones.

The geometry of the carboxylic moieties, which are planar within error, conforms to the notation of Borthwick (1980), the angles opposite the C=O bonds being significantly smaller than the two adjacent ones. One of these groups in HDDO [C(15), C(14), O(3), O(4)] shows a more marked displacement from planarity [maximum out-of-plane distance = 0·02 (3) Å].

The C_{sp^3}—C_{sp^3} bond distances of the aliphatic chains are generally shorter than the expected value [1·537 (5) Å; Sutton, 1965] but in the range previously observed in a large number of analogous derivatives (Bocelli & Grenier-Loustalot, 1984, and papers quoted therein).

Table 1. *Fractional atomic coordinates* (×10⁴) *with* B_{eq} *values* (Å²) *for HDDO, HDDM and HDDP*

	x	y	z	B^*_{eq}
HDDO				
Cl(1)	3841 (1)	3840·5 (5)	41 (1)	7·42
Cl(2)	11377 (1)	9303·4 (4)	10178 (1)	6·38
O(1)	4163 (2)	5535 (1)	2212 (1)	4·36
O(2)	3698 (4)	4460 (1)	2265 (2)	6·66
O(3)	10024 (2)	7549 (1)	8162 (1)	4·63
O(4)	10895 (4)	8563 (1)	7964 (2)	7·33
C(1)	2683 (3)	5049 (1)	501 (2)	3·90
C(2)	1755 (4)	5614 (1)	166 (2)	4·60
C(3)	849 (4)	5700 (2)	−974 (3)	5·55
C(4)	909 (4)	5210 (2)	−1757 (2)	6·05
C(5)	1824 (5)	4652 (2)	−1445 (2)	5·74
C(6)	2721 (4)	4565 (1)	−302 (2)	4·69
C(7)	3562 (3)	4967 (1)	1745 (2)	4·13
C(8)	4980 (5)	5513 (1)	3431 (2)	6·38
C(9)	5663 (4)	6184 (1)	3777 (2)	4·36
C(10)	6644 (4)	6200 (1)	5032 (2)	6·66
C(11)	7336 (4)	6873 (1)	5401 (2)	4·63
C(12)	8355 (4)	6893 (1)	6656 (2)	7·33
C(13)	9026 (4)	7568 (1)	6972 (2)	3·90
C(14)	10872 (3)	8089 (1)	8548 (2)	4·60
C(15)	11875 (3)	8009 (1)	9763 (2)	5·55
C(16)	12525 (4)	7397 (1)	10135 (2)	6·05
C(17)	13492 (4)	7302 (2)	11248 (2)	5·74
C(18)	13790 (4)	7826 (2)	12000 (2)	4·69
C(19)	13147 (4)	8434 (2)	11655 (2)	4·13
C(20)	12185 (3)	5829 (1)	10539 (2)	5·04
HDDM				
Cl	1079 (1)	−8477 (1)	13215 (1)	6·06
O(1)	3340 (2)	−320 (2)	8528 (2)	4·17
O(2)	4095 (3)	−3124 (3)	8485 (3)	6·48
C(1)	2244 (3)	−3135 (3)	10399 (3)	3·35
C(2)	2192 (3)	−5124 (3)	11102 (3)	3·75
C(3)	1154 (3)	−5984 (3)	12303 (3)	3·98
C(4)	157 (3)	−4935 (4)	12821 (3)	4·48
C(5)	253 (3)	−2964 (4)	12133 (3)	4·50
C(6)	1294 (3)	−2046 (3)	10932 (3)	3·82
C(7)	3331 (3)	−2224 (3)	9053 (3)	3·86
C(8)	4359 (3)	682 (4)	7196 (3)	4·46
C(9)	4070 (3)	2766 (3)	6649 (3)	3·97
C(10)	5132 (3)	3946 (3)	5291 (3)	3·97
HDDP				
Cl(1)	30	−48	2518	6·08
Cl(2)	19597 (2)	−12549 (3)	16210 (3)	5·72
O(1)	6245 (5)	−4673 (9)	5520 (9)	4·65
O(2)	6390 (7)	−6017 (11)	2069 (11)	6·18
O(3)	13427 (6)	−7839 (9)	13332 (10)	4·52
O(4)	13300 (7)	−6536 (11)	16734 (11)	6·32
C(1)	4327 (2)	−3658 (4)	3379 (4)	3·71
C(2)	3624 (2)	−2398 (4)	5120 (4)	4·42
C(3)	2297 (2)	−1254 (4)	4874 (4)	4·99
C(4)	1673 (2)	−1371 (4)	2886 (4)	4·15
C(5)	2376 (2)	−2631 (4)	1145 (4)	4·70
C(6)	3703 (2)	−3774 (4)	1391 (4)	4·45
C(7)	5723 (8)	−4891 (15)	3541 (18)	4·53
C(8)	7593 (8)	−5914 (14)	5736 (15)	5·72
C(9)	8043 (8)	−5328 (15)	8023 (16)	4·65
C(10)	9551 (10)	−6545 (12)	8291 (15)	6·18
C(11)	10129 (7)	−5963 (14)	10522 (12)	4·52
C(12)	11552 (9)	−7168 (12)	10805 (15)	6·32
C(13)	12023 (8)	−6643 (15)	13053 (16)	3·71
C(14)	13868 (10)	−7609 (13)	15266 (16)	4·42
C(15)	15282 (3)	−8948 (5)	15457 (6)	4·99
C(16)	15998 (3)	−10187 (5)	13718 (6)	4·15
C(17)	17332 (3)	−11306 (5)	13972 (6)	4·70
C(18)	17949 (3)	−11186 (5)	15964 (6)	4·45
C(19)	17234 (3)	−9947 (5)	17702 (6)	4·53
C(20)	15900 (3)	−8828 (5)	17449 (6)	4·46

* According to Hamilton (1959).

Like the analogous unhalogenated derivative (Pérez & Brisse, 1977*a*), the aliphatic chain shows torsion angles near to 180° so the conformations are all-*trans*. This conformational behaviour is different from that

---

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates for all 3 compounds have been deposited with the British Library Division as Supplementary Publication No. SUP 39305 (37 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

	HDDO	HDDM	HDDP		HDDO	HDDP
Cl(1)–C(6)	1·730 (2)			Cl(2)–C(20)	1·722 (2)	
Cl–C(3)		1·745 (2)				
Cl(1)–C(4)			1·712 (2)	Cl(2)–C(18)		1·712 (3)
O(1)–C(7)	1·326 (3)	1·327 (3)	1·350 (13)	O(3)–C(13)	1·463 (3)	1·475 (10)
O(1)–C(8)	1·454 (3)	1·455 (3)	1·435 (9)	O(3)–C(14)	1·321 (3)	1·299 (12)
O(2)–C(7)	1·197 (3)	1·203 (4)	1·225 (12)	O(4)–C(14)	1·195 (3)	1·168 (11)
C(1)–C(2)	1·382 (3)	1·391 (3)	1·395 (3)	C(11)–C(12)	1·534 (3)	1·493 (11)
C(1)–C(6)	1·384 (3)	1·389 (4)	1·395 (4)	C(12)–C(13)	1·494 (3)	1·500 (14)
C(1)–C(7)	1·500 (3)	1·489 (4)	1·466 (8)	C(14)–C(15)	1·498 (3)	1·513 (9)
C(2)–C(3)	1·400 (4)	1·372 (4)	1·395 (3)	C(15)–C(16)	1·385 (3)	1·395 (5)
C(3)–C(4)	1·379 (5)	1·387 (4)	1·395 (4)	C(15)–C(20)	1·392 (3)	1·395 (5)
C(4)–C(5)	1·361 (5)	1·377 (4)	1·395 (3)	C(16)–C(17)	1·390 (3)	1·395 (4)
C(5)–C(6)	1·401 (3)	1·381 (3)	1·395 (3)	C(17)–C(18)	1·380 (5)	1·395 (5)
C(8)–C(9)	1·496 (3)	1·489 (4)	1·521 (14)	C(18)–C(19)	1·369 (5)	1·395 (5)
C(9)–C(10)	1·525 (3)	1·523 (3)	1·564 (12)	C(19)–C(20)	1·392 (3)	1·395 (4)
C(10)–C(11)	1·507 (3)		1·530 (12)			
C(10)–C(10′)		1·500 (3)				
C(7)–O(1)–C(8)	115·5 (2)	116·2 (2)	114·7 (7)	C(13)–O(3)–C(14)	116·1 (2)	113·8 (8)
C(6)–C(1)–C(7)	121·9 (2)	121·4 (2)	117·6 (5)	C(10)–C(11)–C(12)	112·8 (2)	112·3 (7)
C(2)–C(1)–C(7)	118·6 (2)	117·9 (2)	122·5 (5)	C(11)–C(12)–C(13)	110·6 (2)	109·9 (8)
C(2)–C(1)–C(6)	119·5 (2)	120·6 (2)	120·0 (3)	O(3)–C(13)–C(12)	107·8 (2)	109·9 (8)
C(1)–C(2)–C(3)	120·7 (2)	118·4 (2)	120·0 (3)	O(3)–C(14)–O(4)	123·2 (2)	127·3 (10)
C(2)–C(3)–C(4)	118·6 (3)	122·1 (2)	120·0 (3)	O(4)–C(14)–C(15)	125·2 (2)	121·4 (9)
C(3)–C(4)–C(5)	121·6 (3)	118·5 (3)	120·0 (3)	O(3)–C(14)–C(15)	111·5 (2)	111·3 (8)
C(4)–C(5)–C(6)	119·8 (3)	121·1 (3)	120·0 (3)	C(14)–C(15)–C(20)	122·4 (2)	117·7 (5)
C(1)–C(6)–C(5)	119·9 (2)	119·3 (2)	120·0 (3)	C(14)–C(15)–C(16)	119·2 (2)	122·2 (5)
Cl(1)–C(6)–C(5)	117·5 (2)			C(16)–C(15)–C(20)	118·4 (2)	120·0 (4)
Cl(1)–C(6)–C(1)	122·7 (2)			C(15)–C(16)–C(17)	121·2 (2)	120·0 (4)
Cl–C(3)–C(2)		119·2 (2)		C(16)–C(17)–C(18)	119·3 (3)	120·0 (4)
Cl–C(3)–C(4)		118·7 (2)		C(17)–C(18)–C(19)	120·5 (3)	120·0 (4)
Cl(1)–C(4)–C(3)			121·6 (2)	C(18)–C(19)–C(20)	120·1 (3)	120·0 (4)
Cl(1)–C(4)–C(5)			118·4 (2)	C(15)–C(20)–C(19)	120·5 (2)	120·0 (4)
O(2)–C(7)–C(1)	125·2 (2)	123·4 (2)	125·3 (10)	Cl(2)–C(20)–C(19)	117·5 (2)	
O(1)–C(7)–C(1)	111·2 (2)	112·8 (2)	113·4 (8)	Cl(2)–C(20)–C(15)	122·1 (2)	
O(1)–C(7)–O(2)	123·6 (2)	123·8 (3)	121·4 (10)	Cl(2)–C(17)–C(18)		118·6 (3)
O(1)–C(8)–C(9)	107·5 (2)	108·3 (2)	106·6 (8)	Cl(2)–C(18)–C(19)		121·3 (3)
C(8)–C(9)–C(10)	111·4 (2)	112·1 (2)	108·2 (8)			
C(9)–C(10)–C(11)	112·3 (2)		112·1 (8)			
C(9)–C(10)–C(10′)		113·4 (2)				
C(2)–C(1)–C(7)–O(1)	−36·8 (3)	−177·1 (2)	−0·2 (11)	C(10)–C(11)–C(12)–C(13)	179·4 (2)	−175·2 (8)
C(6)–C(1)–C(7)–O(1)	146·0 (3)	3·5 (4)	179·6 (6)	C(11)–C(12)–C(13)–O(3)	−178·3 (2)	179·8 (8)
C(2)–C(1)–C(7)–O(2)	142·4 (3)	4·1 (4)	−179·7 (8)	C(12)–C(13)–O(3)–C(14)	172·8 (2)	−173·5 (8)
C(6)–C(1)–C(7)–O(2)	−34·8 (4)	−175·2 (3)	0·1 (13)	C(13)–O(3)–C(14)–C(15)	−178·8 (2)	176·9 (7)
C(1)–C(7)–O(1)–C(8)	177·6 (2)	−179·3 (2)	177·7 (7)	C(13)–O(3)–C(14)–O(4)	−2·8 (4)	−3·2 (15)
O(2)–C(7)–O(1)–C(8)	−1·6 (4)	−0·5 (4)	−2·7 (14)	O(3)–C(14)–C(15)–C(16)	32·2 (3)	2·5 (10)
C(7)–O(1)–C(8)–C(9)	176·5 (2)	173·8 (2)	174·8 (8)	O(3)–C(14)–C(15)–C(20)	−148·0 (2)	178·5 (6)
O(1)–C(8)–C(9)–C(10)	−176·5 (2)	178·1 (2)	−175·7 (7)	O(4)–C(14)–C(15)–C(16)	−143·7 (3)	−177·4 (8)
C(8)–C(9)–C(10)–C(11)	−179·7 (2)		176·1 (8)	O(4)–C(14)–C(15)–C(20)	36·2 (4)	−1·5 (12)
C(8)–C(9)–C(10)–C(10′)		179·9 (2)				
C(9)–C(10)–C(10′)–C(9′)		180·0 (2)				
C(9)–C(10)–C(11)–C(12)	−178·9 (2)		177·9 (8)			

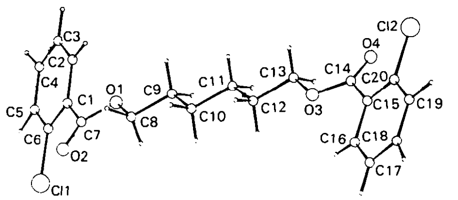

Fig. 1. Projection of HDDO on (001).

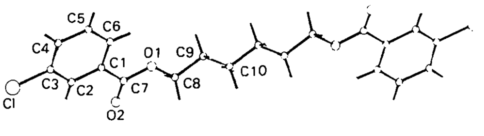

Fig. 2. Projection of HDDM on (001).

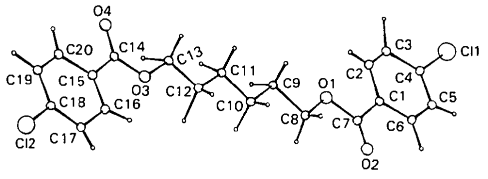

Fig. 3. Projection of HDDP on (001).

found in derivatives with butanediyl (Bocelli & Grenier-Loustalot, 1982, 1984) or ethylene chains (Pérez & Brisse, 1975, 1976*a*; Bocelli & Grenier-Loustalot, 1983) in which the presence of halogens at rings caused significant modifications in the chain conformations. It is of interest that propanediyl dibenzoate (Pérez & Brisse, 1977*b*) and its *p*-chloro derivative (Pérez & Brisse, 1976*b*) both show an all-*gauche* conformation.

The conformational uniformity of these derivatives reflects on the length of the molecules (distances between the centres of the rings) which is 15·719 Å in the unchlorinated derivative and 15·790, 15·633 and 15·717 Å in HDDO, HDDM and HDDP, respectively. The steric hindrance between the chlorines and the carboxylic groups, particularly marked in HDDO, causes a rotation of the carboxylic group with respect to the ring with consequent significant shortening of the length of the molecule. The tilt of the carboxylic moieties with respect to their rings, range 0·2 (2)–4·5 (1)° in HDDM and HDDP, increases to 36·0 (1) and 33·8 (1)° in HDDO.

The phenyl rings are parallel to each other for symmetry requirements in HDDM, quasi-parallel [1·0 (1)°] in HDDP, whilst their least-squares planes subtend a dihedral angle of 13·5 (1)° in HDDO.

The packing of the molecules in the crystals is mainly determined by van der Waals interactions.

This research has received the support of the CNR (Rome) and the CNRS (Paris) through an International Project (ERA 895).

References

Belletti, D., Ugozzoli, F., Cantoni, A. & Pasquinelli, G. (1979). *Gestione on Line di Diffrattometro a Cristallo Singolo Siemens AED con Sistema General Automation Jumbo* 220. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma. Internal Reports 1,2,3–79.

Bocelli, G. & Grenier-Loustalot, M. F. (1982). *Acta Cryst.* B**38**, 2072–2075.

Bocelli, G. & Grenier-Loustalot, M. F. (1983). *Acta Cryst.* C**39**, 1663–1665.

Bocelli, G. & Grenier-Loustalot, M. F. (1984). *Acta Cryst.* C**40**, 679–683.

Borthwick, P. W. (1980). *Acta Cryst.* B**36**, 628–632.

Brisse, F., Marchessault, R. H. & Pérez, S. (1980). *Preparation and Properties of Stereoregular Polymers*, edited by R. W. Lenz & F. Ciardelli, pp. 407–430. Dordrecht-Boston-London: Reidel.

Domenicano, A., Mazzeo, P. & Vaciago, A. (1976). *Tetrahedron Lett.* **13**, 1029–1032.

Domenicano, A. & Murray-Rust, P. (1979). *Tetrahedron Lett.* **24**, 2283–2286.

Domenicano, A., Vaciago, A. & Coulson, C. A. (1975). *Acta Cryst.* B**31**, 221–234.

Germain, G., Main, P. & Woolfson, M. M. (1974). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* A**30**, 580–589.

Pérez, S. & Brisse, F. (1975). *Can. J. Chem.* **53**, 3551–3556.

Pérez, S. & Brisse, F. (1976*a*). *Acta Cryst.* B**32**, 470–474.

Pérez, S. & Brisse, F. (1976*b*). *Acta Cryst.* B**32**, 1518–1521.

Pérez, S. & Brisse, F. (1977*a*). *Acta Cryst.* B**33**, 1673–1677.

Pérez, S. & Brisse, F. (1977*b*). *Acta Cryst.* B**33**, 3259–3262.

Sheldrick, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.

Sutton, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions, Suppl.* 1956–59. London: The Chemical Society.

Acta Cryst. (1984). C**40**, 1394-1397

Stereochemical Studies of Oligomers. XI.* The Structure of 1,2-Propanediyl Bis(*p*-chlorobenzoate) (PDP), $C_{17}H_{14}Cl_2O_4$, and 2,3-Butanediyl Bis(*p*-chlorobenzoate) (BDP), $C_{18}H_{16}Cl_2O_4$

By Gabriele Bocelli

Centro di Studio per la Strutturistica Diffrattometrica del CNR, Via M. D'Azeglio 85, 43100 *Parma, Italy*

and Marie Florence Grenier-Loustalot

Institut Universitaire de Recherche Scientifique, Avenue Philippon, 64000 *Pau, France*

(*Received* 15 *July* 1983; *accepted* 5 *March* 1984)

Abstract. Ni-filtered Cu $K\alpha$ radiation, $\bar{\lambda} = 1{\cdot}5418$ Å, room temperature. PDP: $M_r = 353{\cdot}2$, monoclinic, $C2/c$, $a = 19{\cdot}859\ (3)$, $b = 8{\cdot}837\ (2)$, $c = 19{\cdot}426\ (2)$ Å, $\beta = 103{\cdot}15\ (4)°$, $V = 3319{\cdot}8\ (11)$ Å^3, $Z = 8$, $D_x = 1{\cdot}41$ g cm^{-3}, $\mu = 37{\cdot}28$ cm^{-1}, $F(000) = 1456$, final $R = 0{\cdot}041$ for 1295 observed reflections. BDP: $M_r = 367{\cdot}2$, monoclinic, $P2_1/c$, $a = 14{\cdot}679\ (2)$, $b = 6{\cdot}284\ (3)$, $c = 19{\cdot}638\ (2)$ Å, $\beta = 93{\cdot}24\ (4)°$, $V = 1808{\cdot}6\ (9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}35$ g cm^{-3}, $\mu = 34{\cdot}40$ cm^{-1}, $F(000) = 760$, final $R = 0{\cdot}055$ for 2920 observed reflections. The conformation of the centre of the chain of BDP is *gauche*, while the presence of one methyl group in PDP shifts the aliphatic chain away from a *trans* arrangement. The two rings are tilted by 11·9 (2), 6·5 (2) and 6·7 (1), 0·5 (1)° with respect to their carboxylic groups in the two compounds, respectively. A significant shortening of one of the C_{sp^3}—C_{sp^3} bonds of the chain of PDP is observed.

---

* Part X: Bocelli & Grenier-Loustalot (1984).

0108-2701/84/081394-04$01.50