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Stereochemical Studies of Oligomers. XIV.* Structures of 2,4-Pentanediyl Bis(*p*-chlorobenzoate) and of 1,3-Butanediyl Bis(*p*-chlorobenzoate) (PEDP and BUDP), $C_{19}H_{18}Cl_2O_4$ and $C_{18}H_{16}Cl_2O_4$

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Abstract. Ni-filtered Cu $K\alpha$ radiation, $\bar{\lambda} = 1{\cdot}5418$ Å, room temperature. PEDP: $M_r = 381{\cdot}3$, monoclinic, $P2_1/n$, $a = 15{\cdot}274$ (3), $b = 11{\cdot}099$ (2), $c = 11{\cdot}559$ (3) Å, $\beta = 106{\cdot}24$ (4)°, $V = 1881{\cdot}4$ (8) Å^3, $Z = 4$, $D_x = 1{\cdot}35$ g cm^{-3}, $\mu = 33{\cdot}25$ cm^{-1}, $F(000) = 792$, $R = 0{\cdot}048$ for 2573 observed reflections. BUDP: $M_r = 367{\cdot}2$, monoclinic, $P2_1/n$, $a = 16{\cdot}935$ (3), $b = 6{\cdot}045$ (2), $c = 16{\cdot}887$ (3) Å, $\beta = 95{\cdot}01$ (3)°, $V = 1722{\cdot}2$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}42$ g cm^{-3}, $\mu = 36{\cdot}13$ cm^{-1}, $F(000) = 760$, $R = 0{\cdot}054$ for 2049 observed reflections. The conformation of the propanediyl segment of the chain is *gauche–gauche* in the two compounds. The values of the remaining torsion angles cause in PEDP a rotation of one aromatic ring with respect to the other with a resulting *endo* conformation of the molecule. The two carboxylic groups are tilted by 8·3 (1), 13·1 (1)° and 3·6 (1), 6·7 (1)° with respect to their phenyl rings in the two compounds, respectively.

Introduction. The interest in conformational changes of this series of compounds has now focused on the presence of chlorine atoms on the aromatic rings or methyl groups on the aliphatic chain. In earlier papers we gave analyses of compounds similar to PEDP but without chlorines (Bocelli & Grenier-Loustalot, 1983*a*) or with chlorine atoms in *meta* positions (Bocelli & Grenier-Loustalot, 1983*b*), while Pérez & Brisse (1977, 1976) have reported two structures: one without methyl groups and chlorines and a second without methyls and with *p*-chlorines.

The two structures here reported constitute another step towards the final goal to have, for all compounds of this series, the crystal structures of all the possible derivatives with or without a methylated chain and with or without halogens.

Experimental. For both compounds: crystallographic system and cell parameters by least squares (30 reflections with θ range 13·1–69·5°, 26 with θ range 12·2–20·1° for PEDP and BUDP, respectively); Siemens AED single-crystal diffractometer equipped with a General Automation Jumbo 220 computer; intensities collected with modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) procedure; data corrected for Lorentz and polarization effects but not for absorption; one standard reflection monitored every 50 counts; atomic scattering factors of *SHELX* (Sheldrick, 1976).†

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

† Lists of structure factors, anisotropic thermal parameters and H coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39655 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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PEDP: colourless prismatic crystal, 0·11 × 0·23 × 0·38 mm, recrystallized from methanol; $2 < \theta < 70°$; check reflection displayed only statistical fluctuation throughout data collection; 3945 collected reflections, h −18/17, k 0/13, l 0/13; 3623 unique, $R_{\rm int} = 0·0174$; 2573 $[I > 2\sigma(I)]$ considered observed and included in the refinement; direct methods with *SHELX* (Sheldrick, 1976), block-matrix anisotropic least squares minimizing $\sum w(\Delta F)^2$, H atoms (from ΔF map) isotropic; final $R = 0·048$, $R_w = 0·051$ with $w = 1·0/(\sigma^2 F_o + 0·0034 F_o^2)$; $\Delta\rho_{\rm max} = 0·17$ e Å^{-3}, $(\Delta/\sigma)_{\rm max} = 0·79$.

BUDP: crystals from acetone solution as colourless prisms, specimen 0·15 × 0·44 × 0·92 mm; $3 < \theta < 70°$; periodically monitored reflection with no significant change in intensity; 3429 collected reflections, h −20/20, k 0/7, l 0/20; 3339 unique, $R_{\rm int} = 0·067$; 2058 observed $[I > 2\sigma(I)]$; direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), block-matrix anisotropic least squares minimizing $\sum w(\Delta F)^2$, H atoms (from ΔF map) isotropic; final $R = 0·054$, $R_w = 0·063$ with $w = 0·0658/(\sigma^2 F_o + 0·06637 F_o^2)$; $\Delta\rho_{\rm max} = 0·37$ e Å^{-3}; $(\Delta/\sigma)_{\rm max} = 0·51$. All calculations on a Gould SEL 32/77 computer.

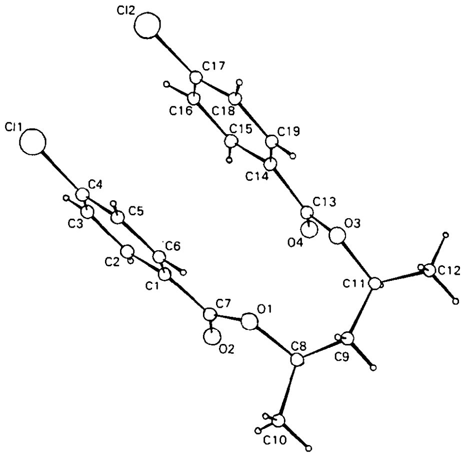

Fig. 1. Projection of PEDP along **c**.

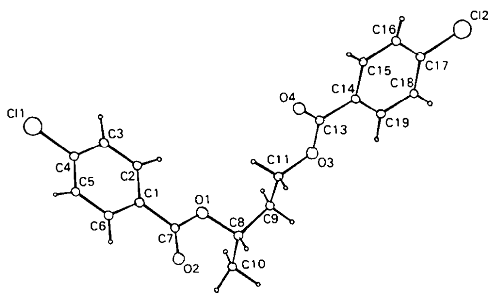

Fig. 2. Projection of BUDP along **c**.

Discussion. Atomic coordinates for both compounds, illustrated in Figs. 1 and 2, respectively, are in Table 1.

The aromatic rings are planar within the errors and their geometrical parameters (Table 2) are normal, with values 1·379 (3), 1·382 (5) Å, 119·9 (6), 120·1 (5)° and 1·383 (4), 1·380 (5) Å, 120·0 (6), 120·0 (4)° for their respective C—C and C—C—C mean values. As always observed in the preceding similar compounds, an enlargement of the internal angles at the chlorine atoms with respect to the adjacent ones, caused by inductive and resonance effects, is present.

The different conformation of the chains in the two derivatives determines a different rotation of the carboxylic moieties with respect to their rings, which are tilted by 3·6 (1), 6·7 (1)° in BUDP and 8·3 (1), 13·1 (1)° in PEDP.

Table 1. *Fractional atomic coordinates* (×10^4) *with* U_{eq} *values* (Å^2) *for* PEDP *and* BUDP

	x	y	z	$U_{\rm eq}$*
PEDP				
Cl(1)	−2703 (1)	2024 (1)	7317 (1)	6·15
Cl(2)	−869 (1)	4462 (1)	8390 (1)	6·83
O(1)	751 (1)	−1736 (2)	8046 (1)	4·64
O(2)	154 (1)	−2056 (2)	6060 (2)	6·58
O(3)	2149 (1)	72 (2)	8908 (1)	4·45
O(4)	1707 (1)	206 (2)	6892 (1)	5·05
C(1)	−604 (2)	−731 (2)	7091 (2)	4·25
C(2)	−1198 (1)	−266 (3)	6062 (2)	5·45
C(3)	−1839 (2)	573 (3)	6112 (2)	5·58
C(4)	−1914 (2)	929 (2)	7230 (2)	4·68
C(5)	−1349 (2)	459 (2)	8276 (2)	4·87
C(6)	−690 (2)	−371 (2)	8202 (2)	4·53
C(7)	116 (2)	−1580 (2)	6990 (2)	4·76
C(8)	1517 (2)	−2544 (2)	8076 (3)	4·76
C(9)	2315 (2)	−2109 (3)	9077 (3)	4·95
C(10)	1214 (4)	−3821 (3)	8274 (6)	7·68
C(11)	2751 (2)	−936 (3)	8879 (2)	4·69
C(12)	3622 (3)	−660 (4)	9862 (4)	6·93
C(13)	1669 (2)	561 (2)	7867 (2)	3·89
C(14)	1088 (1)	1565 (2)	8027 (2)	3·67
C(15)	457 (2)	2039 (2)	7020 (2)	4·37
C(16)	−136 (2)	2926 (2)	7134 (2)	4·80
C(17)	−96 (2)	3366 (2)	8258 (2)	4·51
C(18)	524 (2)	2923 (2)	9277 (3)	5·07
C(19)	1111 (2)	2027 (2)	9163 (2)	4·46
BUDP				
Cl(1)	2202 (1)	6668 (2)	6520 (1)	6·19
Cl(2)	6522 (1)	11600 (2)	5923 (1)	7·21
O(1)	1194 (1)	1634 (4)	5992 (1)	3·96
O(2)	704 (1)	−914 (4)	6786 (2)	5·03
O(3)	3441 (1)	4867 (4)	6212 (1)	4·08
O(4)	3197 (1)	7368 (4)	7141 (1)	4·93
C(1)	−62 (2)	2279 (6)	6428 (2)	3·64
C(2)	−98 (2)	4333 (6)	6057 (2)	4·08
C(3)	−760 (2)	5663 (6)	6069 (2)	4·47
C(4)	−1378 (2)	4933 (6)	6482 (2)	4·27
C(5)	−1364 (2)	2927 (6)	6853 (2)	4·50
C(6)	−703 (2)	1584 (7)	6830 (2)	4·38
C(7)	640 (2)	816 (6)	6427 (2)	3·80
C(8)	1922 (2)	335 (5)	5958 (2)	3·85
C(9)	2565 (2)	1945 (6)	5779 (2)	3·91
C(10)	1782 (2)	−1435 (7)	5337 (3)	5·24
C(11)	2760 (2)	3586 (6)	6429 (2)	4·09
C(13)	3606 (2)	6693 (5)	6642 (2)	3·56
C(14)	4334 (2)	7848 (6)	6435 (2)	3·49
C(15)	4509 (2)	9893 (6)	6793 (2)	4·03
C(16)	5179 (2)	11027 (6)	6630 (2)	4·53
C(17)	5678 (2)	10132 (6)	6116 (2)	4·54
C(18)	5527 (2)	8114 (7)	5766 (2)	4·97
C(19)	4848 (2)	6981 (6)	5920 (2)	4·19

* According to Hamilton (1959).

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

	PEDP	BUDP		PEDP	BUDP
Cl(1)–C(4)	1·734 (3)	1·751 (4)	Cl(2)–C(17)	1·731 (3)	1·737 (4)
O(1)–C(7)	1·341 (3)	1·336 (4)	O(3)–C(13)	1·337 (3)	1·337 (4)
O(1)–C(8)	1·467 (3)	1·467 (4)	O(3)–C(11)	1·455 (4)	1·462 (4)
O(2)–C(7)	1·214 (3)	1·209 (5)	O(4)–C(13)	1·211 (3)	1·208 (4)
C(1)–C(2)	1·379 (3)	1·390 (5)	C(14)–C(15)	1·390 (3)	1·397 (5)
C(1)–C(6)	1·386 (4)	1·394 (5)	C(14)–C(19)	1·401 (3)	1·386 (5)
C(1)–C(7)	1·477 (4)	1·482 (5)	C(13)–C(14)	1·468 (3)	1·485 (5)
C(2)–C(3)	1·364 (4)	1·381 (5)	C(15)–C(16)	1·369 (4)	1·374 (5)
C(3)–C(4)	1·386 (4)	1·380 (5)	C(16)–C(17)	1·373 (3)	1·374 (5)
C(4)–C(5)	1·377 (3)	1·364 (5)	C(17)–C(18)	1·379 (4)	1·369 (5)
C(5)–C(6)	1·385 (4)	1·386 (5)	C(18)–C(19)	1·370 (4)	1·383 (5)
C(8)–C(9)	1·506 (4)	1·511 (5)	C(9)–C(11)	1·509 (5)	1·495 (5)
C(8)–C(10)	1·528 (5)	1·502 (6)	C(11)–C(12)	1·520 (5)	
C(7)–O(1)–C(8)	118·0 (2)	117·2 (2)	C(11)–O(3)–C(13)	118·9 (2)	115·7 (2)
C(6)–C(1)–C(7)	121·5 (2)	118·7 (3)	C(19)–C(14)–C(13)	122·6 (2)	123·4 (3)
C(2)–C(1)–C(7)	119·7 (2)	122·4 (3)	C(15)–C(14)–C(13)	118·9 (2)	117·6 (3)
C(2)–C(1)–C(6)	118·2 (2)	118·9 (3)	C(15)–C(14)–C(19)	118·4 (2)	119·0 (3)
C(1)–C(2)–C(3)	121·7 (2)	121·1 (3)	C(14)–C(15)–C(16)	120·9 (2)	120·2 (3)
C(2)–C(3)–C(4)	118·8 (3)	118·3 (3)	C(15)–C(16)–C(17)	119·4 (2)	119·6 (3)
Cl(1)–C(4)–C(3)	119·6 (2)	118·0 (3)	Cl(2)–C(17)–C(16)	118·8 (2)	118·6 (3)
Cl(1)–C(4)–C(5)	119·3 (2)	119·8 (3)	Cl(2)–C(17)–C(18)	119·8 (2)	120·0 (3)
C(3)–C(4)–C(5)	121·1 (3)	122·2 (3)	C(16)–C(17)–C(18)	121·3 (2)	121·4 (3)
C(4)–C(5)–C(6)	119·1 (2)	119·3 (3)	C(17)–C(18)–C(19)	119·2 (3)	119·1 (3)
C(1)–C(6)–C(5)	120·5 (2)	120·1 (3)	C(14)–C(19)–C(18)	120·7 (2)	120·6 (3)
O(2)–C(7)–C(1)	124·7 (2)	123·5 (3)	O(4)–C(13)–C(14)	123·5 (2)	123·0 (3)
O(1)–C(7)–C(1)	112·0 (2)	112·4 (3)	O(3)–C(13)–C(14)	113·2 (2)	113·5 (3)
O(1)–C(7)–O(2)	123·3 (2)	124·0 (3)	O(3)–C(13)–O(4)	123·3 (2)	123·5 (3)
O(1)–C(8)–C(9)	106·9 (2)	106·6 (3)	O(3)–C(11)–C(9)	110·6 (3)	107·2 (3)
O(1)–C(8)–C(10)	107·3 (3)	109·1 (3)	O(3)–C(11)–C(12)	104·8 (3)	
C(9)–C(8)–C(10)	113·3 (3)	113·1 (3)	C(9)–C(11)–C(12)	113·1 (3)	
C(2)–C(1)–C(7)–O(1)	166·1 (2)	−4·1 (5)	C(15)–C(14)–C(13)–O(3)	−170·8 (2)	173·5 (3)
C(6)–C(1)–C(7)–O(1)	−12·8 (4)	177·2 (3)	C(19)–C(14)–C(13)–O(3)	5·9 (3)	−7·7 (5)
C(2)–C(1)–C(7)–O(2)	−11·6 (4)	175·3 (4)	C(15)–C(14)–C(13)–O(4)	8·8 (4)	173·5 (3)
C(6)–C(1)–C(7)–O(2)	169·5 (3)	−3·4 (5)	C(19)–C(14)–C(13)–O(4)	−174·5 (3)	173·6 (3)
C(1)–C(7)–O(1)–C(8)	−179·1 (2)	179·1 (3)	C(14)–C(13)–O(3)–C(11)	179·8 (2)	176·0 (3)
O(2)–C(7)–O(1)–C(8)	−1·4 (4)	−0·3 (5)	O(4)–C(13)–O(3)–C(11)	0·1 (4)	−5·2 (4)
C(7)–O(1)–C(8)–C(9)	151·9 (2)	−154·8 (3)	C(13)–O(3)–C(11)–C(9)	−101·5 (3)	167·5 (3)
O(1)–C(8)–C(9)–C(11)	−70·7 (3)	65·5 (4)	O(3)–C(11)–C(9)–C(8)	70·6 (3)	174·1 (3)
C(10)–C(8)–C(9)–C(11)	171·2 (3)	−174·7 (3)	C(12)–C(11)–C(9)–C(8)	−172·4 (3)	

In these derivatives too, the angles opposite to the C=O bonds are significantly smaller than the two adjacent ones.

The C_{sp^3}—C_{sp^3} distances are generally shorter than the value suggested by Sutton (1965) [1·537 (5) Å] and one of them in BUDP [C(9)—C(11) = 1·495 (5) Å] shows a marked shortening, which has often been observed in this type of molecular fragment (Bocelli & Grenier-Loustalot, 1984*a*, and papers quoted therein).

The methyl groups in both compounds are all practically equatorial with respect to the chain, the angle between the methyl-bond directions and the normal to the C(8)—C(9)—C(11) plane being 98·0 (3) and 97·0° in PEPD and 85·1 (3)° in BUDP.

The conformation of the propanediyl chain is *gauche–gauche* in both the derivatives but a different configuration of the two molecules results from the torsion angles involving the oxygen atoms. In fact one of these angles [C(7)—O(1)—C(8)—C(9)] is practically equal in PEDP and BUDP [151·9 (2), −154·8 (3)°], while the remaining one [C(9)—C(11)—O(3)—C(13)] is different, being −101·5 (3)° in PEDP and 167·5 (3)° in BUDP. This causes in the first derivative a rotation of one ring with respect to the other and the molecule as a whole assumes an *endo* conformation with a quasi-overlapping of the two rings, which form a dihedral angle of 7·5 (1)° to each other. An identical configuration was found in the analogous derivative without chlorines (Bocelli & Grenier-Loustalot, 1983*a*). It is interesting to observe that a *gauche–gauche* conformation was found in the unmethylated derivatives without chlorines (Pérez & Brisse, 1977), with chlorines in *para* (Pérez & Brisse, 1976) and *meta* positions (Bocelli & Grenier-Loustalot, 1984*c*) and in that methylated with *meta*-chlorines (Bocelli & Grenier-Loustalot, 1983*b*). This common *gauche–gauche* conformation for all the analysed derivatives seems to contradict the conformation found in the derivatives with ethanediyl chains, in which the presence of one methyl group moves the conformation away from a *trans* arrangement while in the presence of two methyls the conformation of the chain is also influenced by the chlorine atoms on the rings.

There are no unusually close intermolecular contacts.

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Structure of a 1/1 Molecular Complex of Tylolupenol A Acetate and Tylolupenol B Acetate, $C_{32}H_{52}O_2.C_{32}H_{52}O_2$

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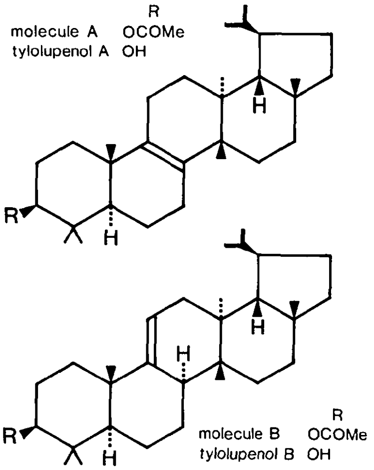
Abstract. $M_r = 937{\cdot}5$, triclinic, $P1$, $a = 13{\cdot}562\,(2)$, $b = 14{\cdot}492\,(2)$, $c = 7{\cdot}691\,(2)$ Å, $\alpha = 93{\cdot}61\,(1)$, $\beta = 90{\cdot}77\,(1)$, $\gamma = 71{\cdot}31\,(1)°$, $V = 1429{\cdot}1\,(6)$ Å^3, $Z = 1$, $D_x = 1{\cdot}089$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}50$ mm^{-1}, $F(000) = 520$, $T = 295$ K, $R = 0{\cdot}044$ for 4883 reflections. The coexistence of the two isomeric acetate molecules in the present complex suggests the presence of the corresponding isomers in tylolupenol. The molecules have the migrated lupane skeleton. There are no unusual bond distances and angles, and no intermolecular contacts shorter than the sums of the van der Waals radii.

Introduction. Xu, Wang & Zhao (1983) isolated five crystalline substances from the EtOH extract of roots of *Tylophora kerrii* Craib. Infrared, ^{13}C-NMR and mass spectral evidence suggested one of them, tylolupenol, to be a 1:1 mixture of two triterpene alcohols, isomeric with respect to the position of the double bond, which could not be separated by silica-gel column chromatography. X-ray study of its acetate revealed a 1:1 molecular complex of *D:C*-friedolup-8-en-3β-ol acetate (molecule *A*) and *D:C*-friedolup-9(11)-en-3β-ol acetate (molecule *B*). Thus, tylolupenol crystals probably consist of the corresponding alcohols, tylolupenols A and B.



Experimental. The title compounds were obtained by esterification of tylolupenol, a crystalline substance extracted from the roots of *Tylophora kerrii* Craib.

Prismatic colorless crystals grown from methanol–chloroform solution. Crystal 0·3 × 0·3 × 0·3 mm, Rigaku AFC-5UD diffractometer, graphite-monochromatized Cu $K\alpha$. Cell dimensions from 2θ angles for 25 reflections ($33° < 2\theta < 45°$). Intensities