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

Cl—C(3)	1·732 (6)	C(4)—C(5)	1·364 (9)
O(1)—C(7)	1·338 (5)	C(5)—C(6)	1·385 (9)
O(1)—C(8)	1·461 (6)	C(8)—C(9)	1·508 (7)
O(2)—C(7)	1·196 (7)	C(9)—C(10)	1·535 (7)
C(1)—C(2)	1·381 (8)	C(10)—C(11)	1·519 (7)
C(1)—C(6)	1·375 (6)	C(11)—C(12)	1·527 (7)
C(1)—C(7)	1·495 (7)	C(12)—C(13)	1·525 (6)
C(2)—C(3)	1·389 (7)	C(13)—C(13′)	1·543 (7)
C(3)—C(4)	1·384 (7)		
C(7)—O(1)—C(8)	115·5 (4)	O(2)—C(7)—C(1)	124·1 (4)
C(6)—C(1)—C(7)	118·0 (4)	O(1)—C(7)—C(1)	112·2 (4)
C(2)—C(1)—C(7)	121·4 (4)	O(1)—C(5)—O(2)	123·6 (4)
C(2)—C(1)—C(6)	120·6 (4)	O(1)—C(8)—C(9)	107·6 (4)
C(1)—C(2)—C(3)	119·0 (5)	C(8)—C(9)—C(10)	111·1 (4)
Cl—C(3)—C(2)	119·5 (4)	C(9)—C(10)—C(11)	112·8 (4)
C(2)—C(3)—C(4)	120·8 (5)	C(10)—C(11)—C(12)	113·2 (4)
Cl—C(3)—C(4)	119·6 (3)	C(11)—C(12)—C(13)	113·0 (4)
C(3)—C(4)—C(5)	119·0 (5)	C(12)—C(13)—C(13′)	112·8 (4)
C(4)—C(5)—C(6)	121·3 (5)	C(13)—C(13′)—C(12′)	112·8 (4)
C(1)—C(6)—C(5)	119·2 (5)		

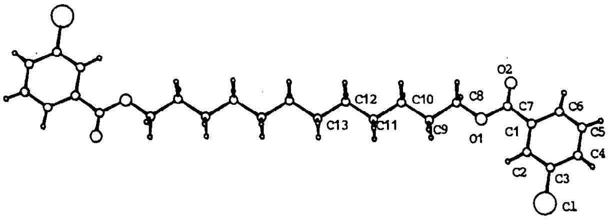

Fig. 1. Drawing of the molecule showing the atom-numbering scheme.

Related literature. The all-*trans* conformation of the chain has been widely observed in this series of derivatives (Bocelli & Grenier-Loustalot, 1986, and papers cited therein).

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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. Internal Report 1–3/79. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma.
Bocelli, G. & Grenier-Loustalot, M. F. (1986). *Acta Cryst.* C**42**, 378–379.
Bocelli, G. & Grenier-Loustalot, M. F. (1987). *Acta Cryst.* C**43**, 1221–1223.
Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* A**30**, 580–589.
Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Rizzoli, C., Sangermano, V., Calestani, G. & Andreetti, G. D. (1987). *J. Appl. Cryst.* **20**. In the press.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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Stereochemical Studies of Oligomers. XXII.* 1,8-Octanediyl Bis(*p*-chlorobenzoate)

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Abstract. $C_{22}H_{24}Cl_2O_4$, $M_r = 423·3$, triclinic, $P\bar{1}$, $a = 12·264$ (2), $b = 11·113$ (2), $c = 8·113$ (3) Å, $\alpha = 78·58$ (2), $\beta = 82·29$ (2), $\gamma = 98·40$ (3)°, $V = 1056·9$ (4) Å^3, $Z = 2$, $D_x = 1·33$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 30·08$ cm^{-1}, $F(000) = 444$, room temperature, $R = 0·039$ for 2763 unique observed reflections. The conformation of the molecule is all-*trans*. The carboxylate groups are tilted by 2·3 (1) and 3·7 (1)° with respect to the phenyl ring planes, which form a dihedral angle of 13·1 (1)° with each other.

Experimental. Intensity data collected on a Siemens AED single-crystal diffractometer equipped with a General Automation Jumbo 220 computer employing Ni-filtered Cu $K\alpha$ radiation and a specimen of about 0·2 × 0·2 × 0·6 mm. Lattice parameters from least-squares fit of (θ,χ,φ) angles of 23 reflections automatically centered on the diffractometer. Intensities

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

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evaluated from a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) method. One check reflection, monitored every 50 counts, showed no intensity change. Lp correction, absorption ignored.

3927 reflections measured, of which 2763 with $I \geq 2\sigma(I)$ used for the refinement, $3 \leq \theta \leq 70°$, $-14 \leq h \leq 14$, $-12 \leq k \leq 13$, $0 \leq l \leq 9$. Structure solved by direct methods with *SHELX*76 (Sheldrick, 1976). Refinement by block-matrix least squares using *SHELX*76, $\sum w(\Delta F)^2$ minimized, all non-H atoms anisotropic, H atoms found from a ΔF map and refined isotropically, 450 variables in total, final $R = 0{\cdot}039$ and $wR = 0{\cdot}048$, $w = 1{\cdot}0/(\sigma^2 F + 0{\cdot}0406F^2)$. In the last cycle of the refinement $(\Delta/\sigma)_{\max}$ was 0·79 for the heavy atoms and $\Delta\rho = 0{\cdot}19$ e Å^{-3}.

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).

The atomic parameters are given in Table 1,* and Table 2 gives bond lengths and angles. Fig. 1 shows the molecule and the numbering scheme.

Table 1. *Atomic fractional coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^4$)

	x	y	z	U_{eq}*
Cl(1)	2563 (1)	3598 (1)	10405 (1)	737 (2)
Cl(2)	15016 (1)	20851 (1)	−8150 (1)	940 (3)
O(1)	6659 (1)	7262 (1)	4562 (2)	597 (5)
O(2)	7489 (1)	5591 (2)	5157 (2)	720 (6)
O(3)	10798 (1)	16454 (1)	−3782 (2)	593 (6)
O(4)	9858 (1)	18046 (2)	−4061 (3)	819 (8)
C(1)	3753 (2)	4340 (2)	8921 (3)	542 (7)
C(2)	4654 (2)	3735 (2)	8760 (3)	612 (8)
C(3)	5597 (2)	4330 (2)	7593 (3)	587 (9)
C(4)	5654 (2)	5520 (2)	6607 (2)	480 (7)
C(5)	4736 (2)	6104 (2)	6820 (3)	600 (8)
C(6)	3781 (2)	5521 (2)	7963 (3)	651 (9)
C(7)	6694 (2)	6100 (2)	5387 (3)	525 (8)
C(8)	7666 (2)	7930 (2)	3392 (3)	603 (9)
C(9)	7394 (2)	9130 (2)	2447 (3)	595 (8)
C(10)	8429 (2)	9976 (2)	1333 (4)	626 (9)
C(11)	8208 (2)	11264 (2)	532 (3)	603 (9)
C(12)	9239 (2)	12143 (2)	−530 (3)	587 (9)
C(13)	9024 (2)	13452 (2)	−1197 (3)	578 (9)
C(14)	10052 (2)	14355 (2)	−2247 (3)	561 (8)
C(15)	9792 (2)	15639 (2)	−2795 (4)	618 (8)
C(16)	10713 (2)	17640 (2)	−4352 (3)	561 (8)
C(17)	11785 (2)	18408 (2)	−5333 (3)	525 (8)
C(18)	12766 (2)	17915 (2)	−5539 (3)	608 (9)
C(19)	13750 (2)	18657 (2)	−6406 (3)	672 (9)
C(20)	13761 (2)	19909 (2)	−7090 (3)	611 (9)
C(21)	12795 (2)	20410 (2)	−6930 (3)	666 (10)
C(22)	11814 (2)	19663 (2)	−6059 (3)	627 (9)

* Hamilton (1959).

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

Cl(1)–C(1)	1·744 (2)	C(8)–C(9)	1·507 (3)
Cl(2)–C(20)	1·738 (2)	C(9)–C(10)	1·523 (3)
O(1)–C(7)	1·341 (3)	C(10)–C(11)	1·528 (3)
O(1)–C(8)	1·455 (2)	C(11)–C(12)	1·519 (3)
O(2)–C(7)	1·208 (3)	C(12)–C(13)	1·522 (3)
O(3)–C(15)	1·450 (3)	C(13)–C(14)	1·524 (3)
O(3)–C(16)	1·332 (3)	C(14)–C(15)	1·501 (3)
O(4)–C(16)	1·212 (3)	C(16)–C(17)	1·488 (3)
C(1)–C(2)	1·380 (4)	C(17)–C(18)	1·397 (4)
C(1)–C(6)	1·380 (3)	C(17)–C(22)	1·396 (3)
C(2)–C(3)	1·381 (3)	C(18)–C(19)	1·372 (3)
C(3)–C(4)	1·390 (3)	C(19)–C(20)	1·389 (3)
C(4)–C(5)	1·387 (4)	C(20)–C(21)	1·382 (4)
C(4)–C(7)	1·480 (3)	C(21)–C(22)	1·372 (3)
C(5)–C(6)	1·378 (3)		

C(7)–O(1)–C(8)	116·4 (2)	C(10)–C(11)–C(12)	113·9 (2)
C(15)–O(3)–C(16)	116·3 (2)	C(11)–C(12)–C(13)	113·1 (2)
Cl(1)–C(1)–C(6)	119·5 (2)	C(12)–C(13)–C(14)	114·1 (2)
Cl(1)–C(1)–C(2)	118·8 (2)	C(13)–C(14)–C(15)	111·2 (2)
C(2)–C(1)–C(6)	121·6 (3)	O(3)–C(15)–C(14)	108·7 (2)
C(1)–C(2)–C(3)	118·6 (2)	O(3)–C(16)–O(4)	123·2 (2)
C(2)–C(3)–C(4)	121·3 (3)	O(4)–C(16)–C(17)	124·2 (2)
C(3)–C(4)–C(7)	118·2 (2)	O(3)–C(16)–C(17)	112·5 (2)
C(3)–C(4)–C(5)	118·4 (2)	C(16)–C(17)–C(22)	119·0 (3)
C(5)–C(4)–C(7)	123·4 (2)	C(16)–C(17)–C(18)	122·1 (2)
C(4)–C(5)–C(6)	121·3 (2)	C(18)–C(17)–C(22)	118·9 (3)
C(1)–C(6)–C(5)	118·8 (3)	C(17)–C(18)–C(19)	120·7 (2)
O(2)–C(7)–C(4)	124·1 (2)	C(18)–C(19)–C(20)	119·2 (3)
O(1)–C(7)–C(4)	112·4 (2)	Cl(2)–C(20)–C(19)	119·0 (2)
O(1)–C(7)–O(2)	123·5 (2)	C(19)–C(20)–C(21)	121·0 (2)
O(1)–C(8)–C(9)	107·3 (2)	Cl(2)–C(20)–C(21)	120·1 (2)
C(8)–C(9)–C(10)	111·7 (2)	C(20)–C(21)–C(22)	119·5 (2)
C(9)–C(10)–C(11)	112·9 (3)	C(17)–C(22)–C(21)	120·6 (3)

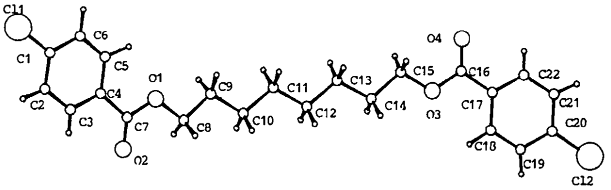

Fig. 1. Drawing of the molecule showing the atom-numbering scheme.

Related literature. This is a further example of an all-*trans* conformation of the chain that had been widely observed in these kinds of derivatives (Bocelli & Grenier-Loustalot, 1986, and papers cited therein).

This work was supported by CNR and CNRS through an International Scientific Project.

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* Lists of structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43758 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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. Internal Report 1/3/79. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma.

Bocelli, G. & Grenier-Loustalot, M. F. (1986). *Acta Cryst.* C**42**, 378–379.

BOCELLI, G. & GRENIER-LOUSTALOT, M. F. (1987). *Acta Cryst.* C**43**, 1223–1224.
HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A**30**, 580–589.
RIZZOLI, C., SANGERMANO, V., CALESTANI, G. & ANDREETTI, G. D. (1987). *J. Appl. Cryst.* **20**. In the press.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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Structure of Ethyl 2-Cyanomethyl-1-β-D-arabinofuranosyl-1*H*-pyrrole-3-carboxylate

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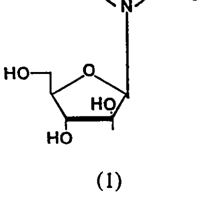
Abstract. $C_{14}H_{18}N_2O_6$, $M_r = 310.31$, orthorhombic, $P2_12_12_1$, $a = 4.8660\,(2)$, $b = 14.7729\,(11)$, $c = 21.4296\,(14)$ Å, $V = 1540.4\,(2)$ Å^3, $Z = 4$, $D_x = 1.338$ g cm^{-3}, $\lambda(\text{Cu}\,K\alpha) = 1.5418$ Å, $\mu = 8.518$ cm^{-1}, $F(000) = 656$, room temperature, $R = 0.0453$ for 1411 reflections ($F \geq 4\sigma_F$). The β anomeric configuration is confirmed. The sugar ring is C(3′)-*endo* ($^3T_4$ form) with a phase angle of pseudorotation of 23·8° and an amplitude of pucker of 36·7° [Altona & Sundaralingam (1972). *J. Am. Chem. Soc.* **94**, 8205–8212]. The C(5′)—O(5′) side chain is in the *gauche–trans* conformation. The nucleoside conformation can be considered as *anti* with a torsion angle about the glycosidic bond [$\chi =$ O(1′)—C(1′)—N(1)—C(5)] of 43·0 (5)°. All atoms of the base ring are within 0·012 Å of its mean plane. The ester moiety [excluding C(13)] is fairly planar as well. The dihedral angle between these planes is 7 (1)°. The cyanomethyl mean plane makes an angle of 95·6 (1)° with the base ring. The hydroxyl hydrogens are involved in intermolecular hydrogen bonding.

Experimental. The title compound (1) was prepared as previously described (Girgis, Cottam, Larson & Robins, 1987*b*). Crystals grew as colorless, transparent flat needles. A crystal was mounted on the end of a glass fiber with epoxy. Details of data collection and structural refinement are given in Table 1.



(1)

Table 1. *Summary of data collection and structural refinement*

Data collection (295 K)	
Mode	ω scan
Scan range (°)	$0.80 + 0.15\tan\theta$
Background (°)	scan 0·25 times scan range before and after scan
Scan rate (° min^{-1})	1·27–5·5
Exposure time (h)	26·7
Stability correction range on I	1·000–1·010
2θ range (°)	3·0–150·0
Total refls: measd, unique	1875, 1875
Range in hkl: min.	0,0,0
max.	6,18,26
Crystal dimensions (mm)	0·33 × 0·20 × 0·09
Crystal volume (mm^3)	0·00594
Transmission factor range:	0·794–0·928
Structure refinement	
Instability factor p	0·02
Reflections used, m ($I \geq 2\sigma_I$)	1411
No. of variables, n	226
Goodness of fit, S	2·364
R, wR	0·0453, 0·0531
R (all data)	0·0908
Max. shift/e.s.d.	0·01
Max., min. density in $\Delta\rho$ map (e Å^{-3})	0·15, −0·18

Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $32.90 \leq 2\theta \leq 53.22°$.
Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used. Data reduction was performed with the *SDP-Plus* software (Frenz, 1985). Crystal and instrument stability was monitored by remeasurement of three check reflections every hour. A linear fit of the intensities of these reflections was used to correct the data.
Function minimized was $\sum w(F_o - F_c)^2$, where $w = \sigma_F^{-2}$. $\sigma_F = F\sigma_I/2I$; $\sigma_I = [N_{\text{pk}} + N_{\text{bg1}} + N_{\text{bg2}} + (pI)^2]^{1/2}$.

21 non-hydrogen atoms were located with *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982); the remaining non-hydrogen atom was located in an electron density difference map. H atoms were located in difference

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