

Stereochemical Studies of Oligomers. XVIII.* 1,3-Propanediyl Bis(*m*-chlorobenzoate)

BY GABRIELE BOCELLI

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

AND MARIE-FLORENCE GRENIER-LOUSTALOT

Institut Universitaire de Recherche Scientifique, ERA 895, Avenue Philippon, 64000 Pau, France

(Received 10 June 1985; accepted 12 November 1985)

Abstract. C₁₇H₁₄Cl₂O₄, $M_r = 353.2$, monoclinic, $C2/c$, $a = 11.737$ (3), $b = 9.387$ (2), $c = 16.121$ (2) Å, $\beta = 111.18$ (4)°, $V = 1656.2$ (7) Å³, $Z = 4$, $D_x = 1.42$ g cm⁻³, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 37.36$ cm⁻¹, $F(000) = 728$, room temperature, $R = 0.054$ for 1425 reflections with $I > 2\sigma(I)$. A *gauche-gauche* conformation characterizes the central segment of the aliphatic chain which becomes *trans* as it approaches the two rings. The carboxylic groups are tilted by 1.5 (1)° with respect to the planar aromatic rings.

Experimental. Pyramid-shaped crystal recrystallized from chloroform-ethanol (1:1) mixture, about 0.33 × 0.48 × 0.62 mm, Siemens AED single-crystal diffractometer equipped with a General Automation Jumbo 220 computer. Lattice parameters from 26 reflections ($14.7 \leq \theta \leq 59.1^\circ$) automatically well centered on diffractometer using a local version (Belletti, Ugozzoli, Cantoni & Pasquinnelli, 1979) of the Lehmann & Larsen (1974) method. A check reflection monitored every 50 counts with no evidence of crystal decomposition or loss in intensity; scans taken from Δ_0 below θ to Δ_1 above ($\Delta_0 = 0.55^\circ$; $\Delta_1 = \{0.55 + [(\lambda_{\alpha_1} - \lambda_{\alpha_2})/\lambda] \tan \theta\}^\circ$). 1718 independent reflections collected, $3 \leq \theta \leq 70^\circ$; 1430 considered observed at 2σ significant level;

$-14 \leq h \leq 13$, $0 \leq k \leq 11$, $0 \leq l \leq 18$; correction for Lorentz and polarization effects, not for absorption. Structure solved by direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), full-matrix least squares using *SHELX* (Sheldrick, 1976), anisotropically for non-hydrogen atoms and isotropically for hydrogens (all found in ΔF map). Final R and wR 0.054 and 0.068 with $w = 1.0/(\sigma^2 F + 0.0063 F^2)$, $\sum w(\Delta F)^2$ minimized; five reflections having larger ΔF and low θ values omitted in the last cycles because probably affected by extinction; $(\Delta\rho)_{\max}$ in final difference map = 0.24 e Å⁻³; $(\Delta/\sigma)_{\max} = 0.032$; scattering factors from *International Tables for X-ray Crystallography* (1974).† All the calculations performed on a Gould SEL 32/77 computer.

† Lists of structure factor amplitudes, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42640 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* Part XVII: Bocelli & Grenier-Loustalot (1986).

Table 1. Atomic fractional coordinates ($\times 10^4$) and U_{eq} values (Å² × 10⁴)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Cl	290 (1)	1982 (1)	1134 (1)	742 (4)
O(1)	-3436 (1)	6196 (2)	-1528 (1)	505 (6)
O(2)	-2140 (2)	4789 (2)	-1876 (1)	728 (8)
C(1)	-2136 (2)	4758 (2)	-402 (1)	416 (6)
C(2)	-1220 (2)	3733 (2)	-106 (2)	470 (8)
C(3)	-851 (2)	3261 (2)	755 (2)	476 (7)
C(4)	-1362 (2)	3782 (2)	1344 (2)	512 (8)
C(5)	-2257 (2)	4807 (3)	1048 (2)	542 (9)
C(6)	-2649 (2)	5305 (2)	186 (1)	480 (9)
C(7)	-2551 (2)	5229 (2)	-1341 (1)	464 (8)
C(8)	-3893 (2)	6777 (3)	-2427 (2)	514 (8)
C(9)	-5000	7655 (3)	-2500	534 (13)

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

Cl—C(3)	1.737 (2)	C(1)—C(7)	1.480 (2)
O(1)—C(7)	1.330 (3)	C(2)—C(3)	1.370 (4)
O(1)—C(8)	1.457 (3)	C(3)—C(4)	1.383 (5)
O(2)—C(7)	1.203 (3)	C(4)—C(5)	1.377 (3)
C(1)—C(2)	1.392 (3)	C(5)—C(6)	1.378 (3)
C(1)—C(6)	1.392 (3)	C(8)—C(9)	1.507 (3)
C(7)—O(1)—C(8)	117.2 (2)	C(3)—C(4)—C(5)	118.4 (3)
C(6)—C(1)—C(7)	121.5 (2)	C(4)—C(5)—C(6)	121.4 (3)
C(2)—C(1)—C(7)	119.0 (2)	C(1)—C(6)—C(5)	119.5 (2)
C(2)—C(1)—C(6)	119.5 (2)	O(2)—C(7)—C(1)	124.2 (2)
C(1)—C(2)—C(3)	119.6 (2)	O(1)—C(7)—C(1)	112.2 (2)
C(2)—C(3)—C(4)	121.7 (2)	O(1)—C(7)—O(2)	123.7 (2)
Cl—C(3)—C(2)	120.1 (2)	O(1)—C(8)—C(9)	106.2 (2)
Cl—C(3)—C(4)	118.3 (2)	C(8)—C(9)—C(8')	113.7 (2)
C(2)—C(1)—C(7)—O(1)	179.3 (2)	C(1)—C(7)—O(1)—C(8)	178.0 (2)
C(6)—C(1)—C(7)—O(1)	0.0 (3)	O(2)—C(7)—O(1)—C(8)	-2.1 (4)
C(2)—C(1)—C(7)—O(2)	-0.6 (4)	O(1)—C(8)—C(9)—C(8')	-74.0 (3)
C(6)—C(1)—C(7)—O(2)	-179.9 (3)	C(8)—C(9)—C(8')—O(1')	-74.0 (3)
C(7)—O(1)—C(8)—C(9)	171.5 (2)		

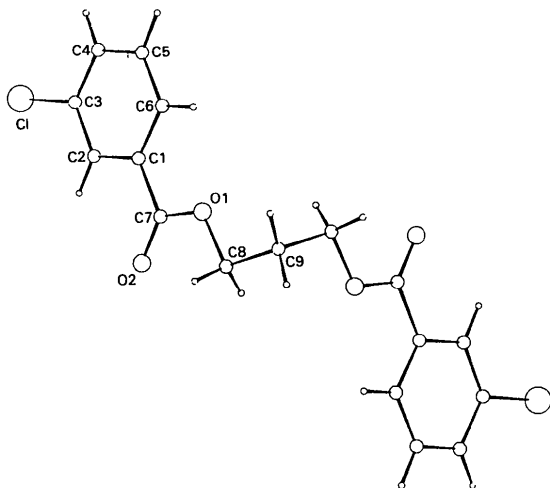


Fig. 1. Projection of the molecule along the y axis.

The atomic coordinates are given in Table 1 and bond distances and angles and selected torsion angles in Table 2. Fig. 1 gives a view of the structure with the atomic numbering.

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

References

- BELLETTI, D., UGOZZOLI, F., CANTONI, A. & PASQUINELLI, G. (1979). *Gestione on Line di Diffratometro a Cristallo Singolo Siemens AED con Sistema General Automation Jumbo 220*. Centro di Studio per la Strutturistica Diffratometrica del CNR, Parma, Internal Reports 1/3-79.
- BOCELLI, G. & GRENIER-LOUSTALOT, M. F. (1986). *Acta Cryst.* C42, 379-381.
- 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.* A30, 580-589.
- MAIN, P., FISKE, S. J., 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.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHORT COMMUNICATION

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.

Acta Cryst. (1986). C42, 511-512

Tl₃{Tl_{0.5}(H₃O)_{0.5}}H₁₄(PO₄)₈·4H₂O: corrigendum. By RICHARD E. MARSH, *Arthur Amos Noyes Laboratory of Chemical Physics,* California Institute of Technology, Pasadena, California 91125, USA*

(Received 19 September 1985; accepted 12 November 1985)

Abstract

The structure of this mixed-valence thallium compound (Chiadmi, Vicat, Tran Qui & Boudjada, 1985) was described as triclinic, space group $P\bar{1}$, with $a = 9.820$ (8), $b = 9.973$ (6), $c = 17.87$ (1) Å, $\alpha = 90.14$ (2), $\beta = 90.01$ (1), $\gamma = 119.46$ (4)°, $Z = 2$. It should be described as monoclinic, space group $C2/c$. The vectors (120), $(\bar{1}00)$, (001) describe a C-centered cell with $a' = 17.367$, $b' = 9.820$, $c' = 17.87$ Å, $\alpha' = 89.99$, $\beta' = 90.17$, $\gamma' = 90.03$ °, $Z = 4$; the correspond-

ing transformations $x' = \frac{1}{2}y, y' = -x + \frac{1}{2}y, z' = z$ yield coordinates that are consistent with $C2/c$ within the reported e.s.d.'s. The $C2/c$ coordinates are given.

Chiadmi *et al.* (1985) noted that the structure is strikingly similar to that of $(\text{H}_3\text{O})[\text{Al}_3(\text{H}_2\text{PO}_4)_6(\text{HPO}_4)_2] \cdot 4\text{H}_2\text{O}$ (Brodalla & Kniep, 1980). The two compounds are, indeed, isostructural. In the aluminium compound [$C2/c$; $a = 16.722$ (4), $b = 9.437$ (6), $c = 17.126$ (5) Å, $\beta = 90.87$ (2)°] the H_3O^+ ion is the sole occupant of the site labeled T1 in Table 1; in the thallium compound this site is shared equally by Tl and H_3O^+ .

* Contribution No. 7295. Work supported in part by the National Institutes of Health (grant GMS 16966).