

Related literature. Kaiser, Saillant, Butler & Rasmussen (1976*a,b*) discuss the preparation and structures of Rh and Ir complexes of biimidazole.

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Stereochemical Studies of Oligomers. XXIII.* 2,5-Hexanediyl Bis(*o*-chlorobenzoate)

BY GABRIELE BOCELLI

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

MARIE-FLORENCE GRENIER-LOUSTALOT

Institut Universitaire de Recherche Scientifique, ERA 895, Avenue L. Sallenave, 64000 Pau, France

AND ZOFIA URBANCZYK-LIPKOWSKA

Institute of Organic Chemistry, Polish Academy of Sciences, 01-224 Warszawa, Poland

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Abstract. $C_{20}H_{20}Cl_2O_4$, $M_r = 395.3$, monoclinic, $P2_1/c$, $a = 11.501$ (2), $b = 8.203$ (3), $c = 11.010$ (3) Å, $\beta = 112.80$ (5)°, $V = 957.6$ (6) Å³, $Z = 2$, $D_x = 1.37$ g cm⁻³, Mo $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 32.84$ cm⁻¹, $F(000) = 412$, room temperature, $R = 0.036$ for 1207 unique observed reflections. The conformation of the chain which is *trans* in the central part [O(2)–C(8)–C(9)–C(9') = 173.0 (2), C(8)–C(9)–C(9')–C(8') = 180°] is far from this arrangement approaching the carboxylic moieties [C(7)–O(2)–C(8)–C(9) = 154.3 (2)°]. The carboxylic group is tilted by 46.5 (1)° with respect to the phenyl-ring plane. The methyl groups, like the halogens, are *trans* to each other.

Experimental. Prismatic specimen approximately $0.4 \times 0.6 \times 0.9$ mm obtained by slow evaporation of a benzene/petroleum ether mixture at room temperature.

Intensity data collected at room temperature on a Siemens AED single-crystal diffractometer equipped with a General Automation Jumbo 220 computer with Ni-filtered Cu $K\alpha$ radiation. Lattice parameters from least-squares fit with 29 reflections up to $\theta_{\max} = 28.35^\circ$ automatically centered on the diffractometer. The intensities evaluated from a modified version (Belletti, Uguzzoli, Cantoni & Pasquinelli, 1979) of the Lehmann

& Larsen (1974) method. One check reflection, monitored every 50 counts, with only random deviations. Lp correction, absorption ignored.

1986 reflections measured, 1821 unique, $R_{\text{int}} = 0.016$, of which 1207 with $I \geq 2\sigma(I)$ used in the refinement, $3 \leq \theta \leq 70^\circ$, $-14 \leq h \leq 12$, $0 \leq k \leq 10$, $0 \leq l \leq 12$. The structure was solved by direct methods with *SHELX76* (Sheldrick, 1976). Refinement by full-matrix least squares using *SHELX* with anisotropic temperature factors for all non-H atoms. H atoms, found in a ΔF map, isotropic. $\sum w(\Delta F)^2$ minimized, 158 refined parameters, final $R = 0.036$ and $wR = 0.039$, $w = 1.0/(\sigma^2 F + 0.00272 F^2)$, $(\Delta/\sigma)_{\max}$ was 0.23 for heavy atoms and $(\Delta/\sigma)_{\max} = 0.20$ e Å⁻³.

All the calculations were performed on a Gould SEL 32/77 computer. Atomic scattering factors were taken from *SHELX*.

The positional parameters with equivalent values of the anisotropic temperature factors are given in Table 1. In Table 2 bond lengths and angles are listed. Fig. 1 shows the molecule and the numbering scheme.†

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43874 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Table 1. Atomic fractional coordinates ($\times 10^4$) and U_{eq} ($\text{\AA}^2 \times 10^4$)

	x	y	z	U_{eq}^*
Cl	7415 (1)	-44 (1)	5570 (1)	725 (3)
O(1)	8474 (2)	4112 (2)	4075 (2)	641 (9)
O(2)	6657 (1)	3018 (2)	3974 (2)	481 (6)
C(1)	8146 (2)	1271 (2)	3698 (2)	388 (7)
C(2)	8015 (2)	-130 (2)	4353 (2)	432 (8)
C(3)	8408 (2)	-1634 (3)	4082 (3)	549 (10)
C(4)	8904 (2)	-1762 (3)	3136 (3)	571 (10)
C(5)	9037 (2)	-398 (3)	2469 (3)	557 (11)
C(6)	8680 (2)	1102 (3)	2770 (2)	473 (10)
C(7)	7803 (2)	2955 (2)	3954 (2)	410 (8)
C(8)	6185 (2)	4612 (3)	4186 (3)	459 (9)
C(9)	5239 (2)	4235 (3)	4777 (3)	543 (11)
C(10)	5664 (4)	5509 (4)	2888 (3)	718 (14)

* Hamilton (1959).

Table 2. Bond distances (\AA) and angles ($^\circ$)

Cl—C(2)	1.730 (3)	C(2)—C(3)	1.386 (3)
O(1)—C(7)	1.198 (3)	C(3)—C(4)	1.372 (5)
O(2)—C(7)	1.328 (3)	C(4)—C(5)	1.379 (4)
O(2)—C(8)	1.469 (3)	C(5)—C(6)	1.378 (4)
C(1)—C(2)	1.396 (3)	C(8)—C(9)	1.500 (5)
C(1)—C(6)	1.388 (4)	C(8)—C(10)	1.510 (4)
C(1)—C(7)	1.493 (3)	C(9)—C(9')	1.526 (4)
C(1)—C(7)	1.393 (3)		
C(7)—O(2)—C(8)	117.9 (1)	C(4)—C(5)—C(6)	119.4 (2)
C(6)—C(1)—C(7)	117.1 (2)	C(1)—C(6)—C(5)	121.6 (2)
C(2)—C(1)—C(7)	125.1 (2)	O(2)—C(7)—C(1)	112.1 (2)
C(2)—C(1)—C(6)	117.7 (2)	O(1)—C(7)—C(1)	123.2 (2)
Cl—C(2)—C(1)	121.5 (1)	O(1)—C(7)—O(2)	124.6 (2)
C(1)—C(2)—C(3)	120.8 (2)	O(2)—C(8)—C(10)	108.6 (2)
Cl—C(2)—C(3)	117.7 (1)	O(2)—C(8)—C(9)	105.1 (2)
C(2)—C(3)—C(4)	119.9 (2)	C(9)—C(8)—C(10)	115.0 (2)
C(3)—C(4)—C(5)	120.4 (2)	C(8)—C(9)—C(9')	112.5 (2)

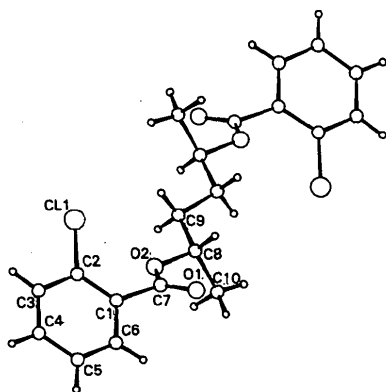


Fig. 1. Projection of the molecule.

Related literature. This compound is strictly related to the analogous derivatives with *para* and *meta* chlorines (Bocelli & Grenier-Loustalot, 1984) and to that without halogens (Bocelli & Grenier-Loustalot, 1982).

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Structure of Methyl (Z)-2-Cyano-3-[(*E*)- α -(methylthio)benzylideneamino]-3-phenylpropenoate

BY J. L. BALCAZAR

Department of Geology, University of Alcalá de Henares, Spain

AND F. FLORENCIO AND S. GARCÍA-BLANCO

X-ray Department, Institute Rocasolano, CSIC, 28006-Madrid, Spain

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Abstract. $C_{19}H_{16}N_2O_2S$, $M_r = 336.408$, monoclinic, $P2_1/n$, $a = 9.587$ (2), $b = 10.292$ (3), $c = 17.718$ (4) \AA , $\beta = 97.11$ (1) $^\circ$, $V = 1734.8$ (7) \AA^3 , $Z = 4$, $D_x =$

1.288 g cm^{-3} , $\text{Mo K}\alpha$, $\lambda = 0.7107$ \AA , $\mu = 1.897$ cm^{-1} , $F(000) = 704$, room temperature, final $R = 0.062$ and $wR = 0.055$ for 4028 observed reflections. In the

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