

Dichlorodiphenoxymethane

Richard Betz, Peter Klüfers* and Moritz M. Reichvilser

Ludwig-Maximilians-Universität, Department Chemie und Biochemie, Butenandtstrasse 5–13, 81377 München, Germany

Correspondence e-mail: kluef@cup.uni-muenchen.de

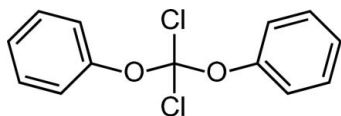
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 Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}–\text{C}) = 0.003$ Å; R factor = 0.038; wR factor = 0.094; data-to-parameter ratio = 18.0.

The title compound, $\text{C}_{13}\text{H}_{10}\text{Cl}_2\text{O}_2$, is a mixed derivative of orthocarbonic acid. The non-crystallographic symmetry of the molecule is close to C_{2v} . The aromatic residues are oriented in a *syn* conformation with respect to the Cl atoms. The least-squares planes through the phenyl rings enclose an angle of $36.11(10)^\circ$. The C–O bonds at the central carbon are relatively short, and the O–C–O and Cl–C–Cl angles are smaller than the tetrahedral angle. These metrical peculiarities including a molecular symmetry close to C_{2v} , are also observed in density functional theory (DFT) calculations, thus ruling out the decisive influence of intermolecular forces in the crystal structure. Accordingly, only few and weak intermolecular interactions are found. At distances smaller than the sum of the van der Waals radii, only two attractive interactions are detected: a weak C–H \cdots O and a weak C–H \cdots Cl hydrogen bond to one of the two potential acceptor atoms each.

Related literature

For the synthesis of the title compound, see Bromley *et al.* (1996). For the crystal structure of related tetraaryloxymethanes with slightly longer C–O bonds, see Narasimhamurthy *et al.* (1990).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_{10}\text{Cl}_2\text{O}_2$
 $M_r = 269.11$
 Monoclinic, $P2_1/c$
 $a = 15.8380(4)$ Å
 $b = 5.8973(2)$ Å
 $c = 14.2517(4)$ Å

 $\beta = 114.751(2)^\circ$
 $V = 1208.85(6)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation

 $\mu = 0.52$ mm⁻¹
 $T = 200(2)$ K
 $0.22 \times 0.20 \times 0.15$ mm

Data collection

 Nonius KappaCCD diffractometer
 Absorption correction: none
 9193 measured reflections
 2766 independent reflections
 2138 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.094$
 $S = 1.07$
 2766 reflections
 154 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.29$ e Å⁻³
Table 1

Selected geometric parameters (Å, °).

O1–C1	1.359(2)	O2–C1	1.360(2)
O1–C1–O2	103.77(13)	Cl1–C1–Cl2	105.29(9)

Table 2

Hydrogen-bond geometry (Å, °).

$D–H\cdots A$	$D–H$	$H\cdots A$	$D\cdots A$	$D–H\cdots A$
C3–H3 \cdots Cl1 ⁱ	0.95	2.81	3.723(2)	161
C10–H10 \cdots O1 ⁱⁱ	0.95	2.52	3.345(3)	145

 Symmetry codes: (i) $x, y - 1, z$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank Dr Peter Mayer for technical assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2358).

References

- Bromley, M. K., Gason, S. J., Jhingran, A. C., Looney, M. G. & Solomon, D. H. (1996). *Aust. J. Chem.* **49**, 1261–1262.
- Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Narasimhamurthy, N., Manohar, H., Samuelson, A. G. & Chandrasekhar, J. (1990). *J. Am. Chem. Soc.* **112**, 2937–2941.
- Nonius (2004). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

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Dichlorodiphenoxymethane

R. Betz, P. Klüfers and M. M. Reichvilser

Comment

The title compound (I) was prepared as starting material for the synthesis of spirocyclic orthocarbonates.

In the molecule the two aromatic moieties are oriented *syn* with respect to the Cl atoms (Fig. 1). The bond lengths between the central C atom and the O atoms are slightly shorter than in related tetraaryloxymethanes (Narasimhamurthy *et al.*, 1990). Unexpectedly, both the O—C—O and the Cl—C—Cl angles are smaller than the tetrahedral angle. The best planes through the phenyl moieties enclose an angle of 36.11 (10)°.

The molecular packing is shown in Figure 2. Below the limit of the sum of the van-der-Waals radii, one weak C—H···O and one weak C—H···Cl hydrogen bond were found in the asymmetric unit as well as an electrostatically repulsive C—H···H—C contact precisely at the vdW radii sum. No π stacking and no C—H··· π contacts were observed within this cutoff criterion.

In agreement with the only weak intermolecular forces, the short bonds to the central carbon atom as well as the small bond angles mentioned above are corroborated by a DFT calculation on the B3LYP/6-311+G(2 d,p) level of theory.

Experimental

The title compound was prepared according to a published procedure (Bromley *et al.*, 1996) upon chlorination of diphenylcarbonate with PCl_5 . Crystals suitable for X-ray analysis were obtained directly from the solid reaction product.

Refinement

H atoms were refined as riding on their parent atoms with U_{iso} as the 1.2-fold of the pilot atom's U_{eq} .

Figures

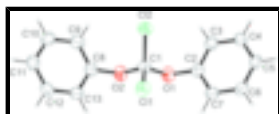


Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

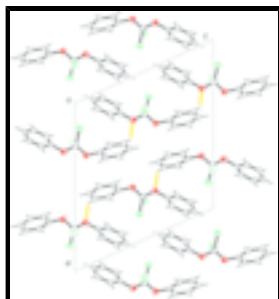


Fig. 2. The packing of (I) viewed along [0 1 0], C—H...O hydrogen bonds drawn as yellow bars.

Dichlorodiphenoxymethane

Crystal data

$C_{13}H_{10}Cl_2O_2$

$M_r = 269.11$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 15.8380$ (4) Å

$b = 5.8973$ (2) Å

$c = 14.2517$ (4) Å

$\beta = 114.751$ (2)°

$V = 1208.85$ (6) Å³

$Z = 4$

$F_{000} = 552$

$D_x = 1.479$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 12368 reflections

$\theta = 3.1$ – 27.5 °

$\mu = 0.52$ mm⁻¹

$T = 200$ (2) K

Block, colourless

$0.22 \times 0.20 \times 0.15$ mm

Data collection

Nonius KappaCCD
diffractometer

Radiation source: rotating anode

Monochromator: MONTEL, graded multilayered X-ray optics

$T = 200$ (2) K

CCD; rotation images; thick slices scans

Absorption correction: none

9193 measured reflections

2766 independent reflections

2138 reflections with $I > 2\sigma(I)$

$R_{int} = 0.034$

$\theta_{max} = 27.5$ °

$\theta_{min} = 3.2$ °

$h = -20 \rightarrow 20$

$k = -7 \rightarrow 7$

$l = -18 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.094$

$S = 1.08$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 0.3721P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

2766 reflections

$$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$$

154 parameters

$$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.22903 (3)	0.81935 (8)	-0.00008 (4)	0.04218 (16)
C12	0.16437 (3)	0.38393 (9)	0.03397 (4)	0.04247 (16)
O1	0.26019 (8)	0.4390 (2)	-0.07857 (9)	0.0370 (3)
O2	0.34125 (8)	0.4761 (2)	0.08404 (9)	0.0367 (3)
C1	0.25543 (12)	0.5198 (3)	0.00839 (13)	0.0327 (4)
C2	0.18098 (12)	0.4454 (3)	-0.17458 (13)	0.0323 (4)
C3	0.12316 (13)	0.2594 (3)	-0.20312 (14)	0.0367 (4)
H3	0.1345	0.1336	-0.1578	0.044*
C4	0.04817 (13)	0.2588 (4)	-0.29911 (14)	0.0379 (4)
H4	0.0071	0.1327	-0.3198	0.046*
C5	0.03320 (13)	0.4419 (3)	-0.36477 (14)	0.0371 (4)
H5	-0.0187	0.4424	-0.4301	0.045*
C6	0.09367 (13)	0.6242 (3)	-0.33543 (14)	0.0394 (5)
H6	0.0836	0.7484	-0.3813	0.047*
C7	0.16881 (13)	0.6273 (3)	-0.23968 (14)	0.0375 (4)
H7	0.2108	0.7516	-0.2194	0.045*
C8	0.36336 (11)	0.5446 (3)	0.18686 (14)	0.0332 (4)
C9	0.34856 (13)	0.3947 (3)	0.25296 (15)	0.0404 (5)
H9	0.3195	0.2525	0.2286	0.048*
C10	0.37686 (13)	0.4557 (4)	0.35513 (16)	0.0448 (5)
H10	0.3670	0.3547	0.4015	0.054*
C11	0.41948 (12)	0.6623 (4)	0.39062 (15)	0.0418 (5)
H11	0.4382	0.7039	0.4609	0.050*
C12	0.43477 (13)	0.8085 (3)	0.32332 (15)	0.0411 (5)
H12	0.4645	0.9500	0.3477	0.049*
C13	0.40687 (12)	0.7493 (3)	0.22053 (15)	0.0377 (4)
H13	0.4177	0.8486	0.1742	0.045*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0470 (3)	0.0337 (3)	0.0407 (3)	0.0093 (2)	0.0133 (2)	0.0033 (2)
C12	0.0378 (3)	0.0472 (3)	0.0451 (3)	-0.0041 (2)	0.0199 (2)	-0.0028 (2)
O1	0.0306 (7)	0.0447 (8)	0.0353 (7)	0.0066 (5)	0.0134 (5)	-0.0046 (6)
O2	0.0288 (6)	0.0427 (8)	0.0357 (7)	0.0091 (5)	0.0108 (5)	0.0002 (6)
C1	0.0302 (9)	0.0314 (10)	0.0352 (10)	0.0054 (7)	0.0126 (8)	0.0009 (8)
C2	0.0297 (9)	0.0371 (10)	0.0318 (9)	0.0045 (7)	0.0146 (7)	-0.0024 (8)
C3	0.0459 (11)	0.0300 (10)	0.0354 (10)	0.0030 (8)	0.0182 (9)	0.0028 (8)
C4	0.0406 (10)	0.0394 (11)	0.0361 (10)	-0.0066 (8)	0.0183 (8)	-0.0059 (9)
C5	0.0385 (10)	0.0471 (12)	0.0277 (9)	0.0000 (8)	0.0157 (8)	-0.0011 (9)

supplementary materials

C6	0.0474 (11)	0.0417 (11)	0.0335 (10)	-0.0002 (9)	0.0213 (9)	0.0068 (9)
C7	0.0411 (10)	0.0368 (11)	0.0398 (10)	-0.0064 (8)	0.0222 (9)	-0.0028 (9)
C8	0.0253 (9)	0.0366 (10)	0.0341 (9)	0.0071 (7)	0.0090 (7)	0.0029 (8)
C9	0.0337 (10)	0.0350 (11)	0.0450 (11)	0.0009 (8)	0.0092 (8)	0.0085 (9)
C10	0.0366 (10)	0.0520 (13)	0.0420 (11)	0.0004 (9)	0.0128 (9)	0.0154 (10)
C11	0.0320 (10)	0.0557 (13)	0.0347 (10)	0.0041 (9)	0.0108 (8)	0.0024 (9)
C12	0.0319 (10)	0.0413 (12)	0.0428 (11)	-0.0037 (8)	0.0085 (8)	-0.0018 (9)
C13	0.0318 (10)	0.0407 (11)	0.0394 (11)	-0.0008 (8)	0.0136 (8)	0.0081 (9)

Geometric parameters (Å, °)

C11—C1	1.8078 (18)	C6—C7	1.385 (3)
C12—C1	1.8154 (18)	C6—H6	0.9500
O1—C1	1.359 (2)	C7—H7	0.9500
O1—C2	1.418 (2)	C8—C13	1.373 (3)
O2—C1	1.360 (2)	C8—C9	1.381 (3)
O2—C8	1.415 (2)	C9—C10	1.380 (3)
C2—C3	1.377 (3)	C9—H9	0.9500
C2—C7	1.378 (3)	C10—C11	1.382 (3)
C3—C4	1.386 (3)	C10—H10	0.9500
C3—H3	0.9500	C11—C12	1.384 (3)
C4—C5	1.383 (3)	C11—H11	0.9500
C4—H4	0.9500	C12—C13	1.386 (3)
C5—C6	1.383 (3)	C12—H12	0.9500
C5—H5	0.9500	C13—H13	0.9500
C1—O1—C2	120.43 (13)	C7—C6—H6	119.7
C1—O2—C8	119.92 (13)	C2—C7—C6	118.23 (18)
O1—C1—O2	103.77 (13)	C2—C7—H7	120.9
O1—C1—C11	112.41 (12)	C6—C7—H7	120.9
O2—C1—C11	111.39 (13)	C13—C8—C9	121.85 (18)
O1—C1—C12	112.36 (12)	C13—C8—O2	118.89 (16)
O2—C1—C12	111.80 (12)	C9—C8—O2	118.98 (17)
C11—C1—C12	105.29 (9)	C10—C9—C8	118.69 (19)
C3—C2—C7	122.22 (17)	C10—C9—H9	120.7
C3—C2—O1	118.32 (16)	C8—C9—H9	120.7
C7—C2—O1	119.22 (16)	C9—C10—C11	120.60 (19)
C2—C3—C4	118.85 (18)	C9—C10—H10	119.7
C2—C3—H3	120.6	C11—C10—H10	119.7
C4—C3—H3	120.6	C10—C11—C12	119.71 (19)
C5—C4—C3	119.95 (18)	C10—C11—H11	120.1
C5—C4—H4	120.0	C12—C11—H11	120.1
C3—C4—H4	120.0	C11—C12—C13	120.33 (19)
C4—C5—C6	120.11 (18)	C11—C12—H12	119.8
C4—C5—H5	119.9	C13—C12—H12	119.8
C6—C5—H5	119.9	C8—C13—C12	118.81 (18)
C5—C6—C7	120.59 (18)	C8—C13—H13	120.6
C5—C6—H6	119.7	C12—C13—H13	120.6
C2—O1—C1—O2	177.16 (14)	C3—C2—C7—C6	2.3 (3)
C2—O1—C1—C11	-62.36 (19)	O1—C2—C7—C6	176.56 (16)

C2—O1—C1—C12	56.20 (19)	C5—C6—C7—C2	-0.6 (3)
C8—O2—C1—O1	178.00 (15)	C1—O2—C8—C13	-94.4 (2)
C8—O2—C1—C11	56.83 (19)	C1—O2—C8—C9	91.6 (2)
C8—O2—C1—C12	-60.67 (19)	C13—C8—C9—C10	1.3 (3)
C1—O1—C2—C3	-91.8 (2)	O2—C8—C9—C10	175.07 (16)
C1—O1—C2—C7	93.7 (2)	C8—C9—C10—C11	-0.2 (3)
C7—C2—C3—C4	-2.4 (3)	C9—C10—C11—C12	-0.7 (3)
O1—C2—C3—C4	-176.73 (15)	C10—C11—C12—C13	0.5 (3)
C2—C3—C4—C5	0.8 (3)	C9—C8—C13—C12	-1.4 (3)
C3—C4—C5—C6	0.9 (3)	O2—C8—C13—C12	-175.20 (16)
C4—C5—C6—C7	-1.0 (3)	C11—C12—C13—C8	0.5 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3 \cdots C11 ⁱ	0.95	2.81	3.723 (2)	161
C10—H10 \cdots O1 ⁱⁱ	0.95	2.52	3.345 (3)	145

Symmetry codes: (i) $x, y-1, z$; (ii) $x, -y+1/2, z+1/2$.

Fig. 1

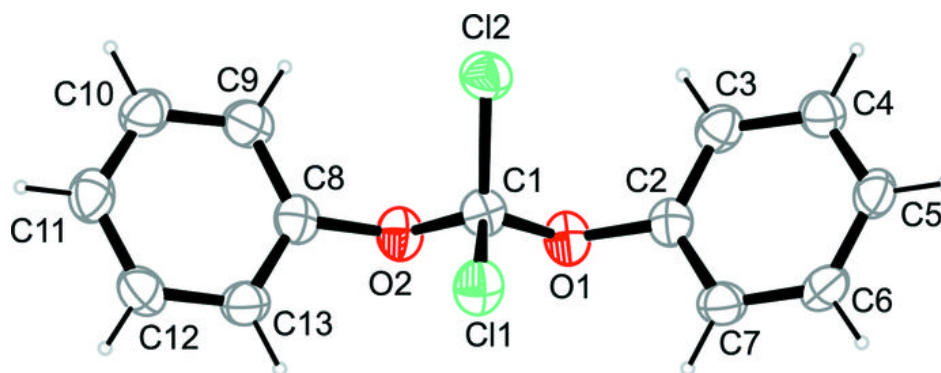


Fig. 2

