1,3-Benzodioxol-2-one

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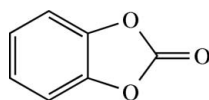Correspondence e-mail: kluef@cup.uni-muenchen.de

The title compound, $C_7H_4O_3$, is the first cyclic carbonate of an aromatic diol whose structure has been elucidated by means of single-crystal X-ray analysis. The molecule possesses crystallographic twofold rotation symmetry and non-crystallographic C_{2v} symmetry. The C—O single bonds are slightly longer than those in comparable cyclic carbonates derived from aliphatic vicinal diols. The crystal structure is built up from columns of π-stacked molecules; the inversion-related molecules are stacked along the b axis, with the centroids of the benzene rings separated by 3.631 (1) Å.

Related literature

For the synthesis of an asymmetric *spiro* orthocarbonate, see: Komatsu *et al.* (1992). For related structures, see: Betz *et al.* (2007); Darensbourg *et al.* (2003).

Experimental

Crystal data

$C_7H_4O_3$	$a = 10.224$ (3) Å
$M_r = 136.10$	$b = 8.9132$ (14) Å
Monoclinic, $C2/c$	$c = 6.9636$ (14) Å
$\beta = 116.053$ (16)°	$\mu = 0.13$ mm^{-1}
$V = 570.1$ (2) Å^3	$T = 100$ (2) K
$Z = 4$	$0.18 \times 0.07 \times 0.06$ mm
Mo $K\alpha$ radiation	

Data collection

Oxford Diffraction XCalibur diffractometer	1617 measured reflections
Absorption correction: analytical (de Meulenaer & Tompa, 1965)	654 independent reflections
$T_{min} = 0.982$, $T_{max} = 0.994$	492 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	Only H-atom displacement parameters refined
$wR(F^2) = 0.072$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.29$ e Å^{-3}
654 reflections	
48 parameters	

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; 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*.

References

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Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Darensbourg, D. J., Lewis, S. J., Rodgers, J. L. & Yarbrough, J. C. (2003). *Inorg. Chem.* **42**, 581–589.

Komatsu, S., Takata, T. & Endo, T. (1992). *Macromolecules*, **25**, 7286–7293.

Meulenaer, J. de & Tompa, H. (1965). *Acta Cryst.* **19**, 1014–1018.

Oxford Diffraction (2005). *CrysAlis CCD* and *CrysAlis RED*. Versions 1.171.27p5 beta. Oxford Diffraction Ltd., Abingdon, Oxfordshire, England.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

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R. Betz and P. Klüfers

Comment

The title compound, (I), was accidentally prepared on the attempted synthesis of an asymmetric *spiro*-orthocarbonate.

The molecule of (I) (Fig. 1) possesses crystallographic twofold symmetry, with atoms C1 and O1 lying on the twofold rotation axis. The C1=O1 bond length [1.191 (2) Å] is comparable to the corresponding distance observed in similar cyclic carbonates derived from aliphatic vicinal diols, but the C1—O2 [1.3660 (13) Å] distance between the carbonyl C atom and the diol O atom is found to be slightly longer (Darensbourg *et al.*, 2003; Betz *et al.*, 2007). The five-membered 1,3-dioxol-2-one ring, which contains the carbonate group, is essentially planar and as a result the molecule as a whole is planar.

In the crystal structure, the inversion-related molecules are stacked along the *b* axis in such a way that the centroids of the benzene rings are separated by 3.631 (1) Å [perpendicular distance 3.370 Å], indicating significant π-π interactions (Figs. 2,3,4)

Experimental

The title compound was obtained accidentally on the attempted synthesis of an asymmetric spiro orthocarbonate according to a published procedure (Komatsu *et al.*, 1992) by reacting 2,2-dichlorobenzo[1.3]dioxole (10 mmol, 1.91 g) and 1-(hydroxymethyl)-cyclobutane-1-ol (10 mmol, 1.02 g) in the presence of pyridine (20 mmol, 1.58 g) in dichloromethane (10 ml). Crystals suitable for X-ray analysis were obtained directly from the crystallized reaction product.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms. One common isotropic displacement parameter for all H atoms was refined to 0.025 (3) Å^2.
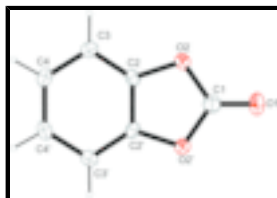
Figures

Fig. 1. The structure of one molecule of (I), with atom labels and anisotropic displacement ellipsoids (drawn at the 50% probability level) for non-H atoms.
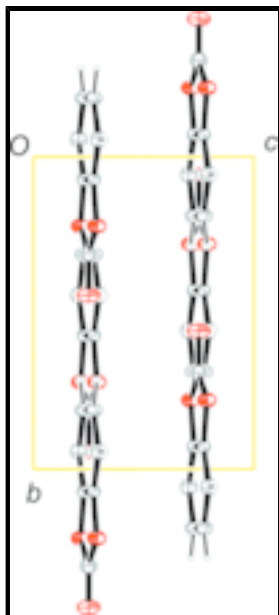
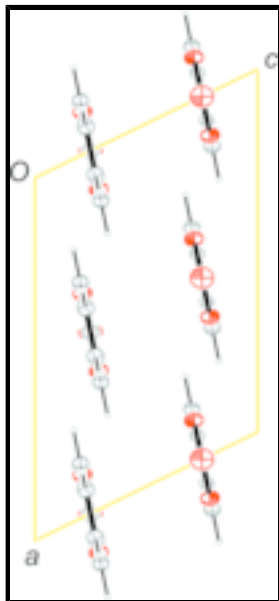Fig. 2. The molecular packing of (I), viewed along [$\bar{1}$ 0 0].

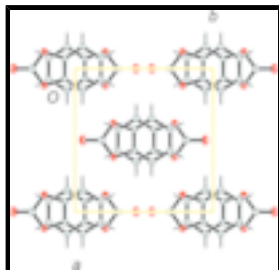Fig. 3. The molecular packing of (I), viewed along [0 1 0].

Fig. 4. The molecular packing of (I), viewed along [0 0 $\bar{1}$].

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Crystal data

$C_7H_4O_3$	$Z = 4$
$M_r = 136.10$	$F_{000} = 280$
Monoclinic, $C2/c$	$D_x = 1.586$ Mg m^{-3}
Hall symbol: -C 2yc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.224$ (3) Å	$\theta = 3.8–27.5º$
$b = 8.9132$ (14) Å	$\mu = 0.13$ mm^{-1}
$c = 6.9636$ (14) Å	$T = 100$ (2) K
$\beta = 116.053$ (16)º	Rod, colourless
$V = 570.1$ (2) Å^3	$0.18 \times 0.07 \times 0.06$ mm

Data collection

Oxford Diffraction XCalibur diffractometer	$R_{int} = 0.025$
Radiation source: fine-focus sealed tube	$\theta_{max} = 27.5º$
Monochromator: graphite	$\theta_{min} = 3.8º$
$T = 100(2)$ K	$h = -11 \rightarrow 13$
ω–scans	$k = -11 \rightarrow 9$
Absorption correction: analytical (de Meulenaer & Tompa, 1965)	$l = -9 \rightarrow 8$
$T_{min} = 0.982$, $T_{max} = 0.994$	Standard reflections: ?;
1617 measured reflections	every ? reflections
654 independent reflections	intensity decay: ?
492 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.030$	Only H-atom displacement parameters refined
$wR(F^2) = 0.072$	$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
654 reflections	$\Delta\rho_{max} = 0.16$ e Å^{-3}
48 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	y	z	$U_{iso}*/U_{eq}$

O1	0.0000	0.44258 (14)	0.2500	0.0311 (4)
O2	0.11806 (9)	0.22115 (9)	0.28638 (13)	0.0204 (3)
C1	0.0000	0.3090 (2)	0.2500	0.0213 (4)
C2	0.07241 (13)	0.07251 (13)	0.27193 (18)	0.0160 (3)
C3	0.15072 (14)	−0.05641 (13)	0.29563 (19)	0.0197 (3)
H3	0.2508	−0.0554	0.3260	0.025 (3)*
C4	0.07345 (13)	−0.18920 (14)	0.27216 (19)	0.0209 (3)
H4	0.1221	−0.2823	0.2867	0.025 (3)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0363 (8)	0.0159 (8)	0.0426 (8)	0.000	0.0187 (7)	0.000
O2	0.0180 (5)	0.0165 (5)	0.0274 (5)	−0.0010 (4)	0.0107 (4)	0.0002 (4)
C1	0.0212 (10)	0.0224 (11)	0.0207 (9)	0.000	0.0097 (8)	0.000
C2	0.0193 (6)	0.0152 (7)	0.0144 (5)	−0.0033 (5)	0.0083 (5)	−0.0009 (4)
C3	0.0174 (6)	0.0221 (7)	0.0200 (6)	0.0030 (5)	0.0087 (5)	0.0000 (5)
C4	0.0251 (7)	0.0166 (7)	0.0221 (6)	0.0038 (5)	0.0114 (6)	0.0008 (5)

Geometric parameters (Å, °)

O1—C1	1.191 (2)	C3—C4	1.3922 (17)
O2—C1	1.3660 (13)	C3—H3	0.95
O2—C2	1.3935 (15)	C4—C4^i	1.394 (3)
C2—C3	1.3684 (17)	C4—H4	0.95
C2—C2^i	1.374 (2)		
C1—O2—C2	106.92 (10)	C2—C3—C4	115.34 (12)
O1—C1—O2	124.98 (7)	C2—C3—H3	122.3
O2^i—C1—O2	110.04 (15)	C4—C3—H3	122.3
C3—C2—C2^i	122.89 (7)	C3—C4—C4^i	121.77 (7)
C3—C2—O2	129.05 (11)	C3—C4—H4	119.1
C2^i—C2—O2	108.06 (6)	C4^i—C4—H4	119.1

Symmetry codes: (i) −x, y, −z+1/2.

Fig. 1

Fig. 2

Fig. 3

Fig. 4