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cis-Perhydro-1,4-benzodioxin-2,3-dione

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Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.005 Å
R factor = 0.061
wR factor = 0.141
Data-to-parameter ratio = 8.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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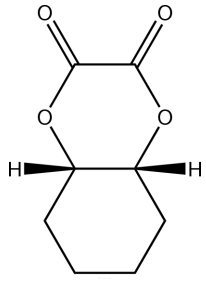
The title compound, $C_8H_{10}O_4$, crystallizes in the non-centrosymmetric space group $Pna2_1$ with two molecules in the asymmetric unit. The structure is characterized by fused cyclohexyl and cyclic diester rings. There is a twist of 60° about the ring fusion. The molecules are characterized by an average C═O bond of 1.199 (8) Å, an average sp^2 C—O bond of 1.328 (8) Å, and an average sp^3 C—O bond of 1.469 (8) Å.

Comment

The title compound, (I), is the major product of the reaction of oxalyl chloride and *cis*-1,2-cyclohexanediol in the presence of triethylamine; the corresponding cyclic carbonate forms as the minor product (Itaya & Iida, 1994; Itaya *et al.*, 2002). Despite significant study in the literature (Lloyd *et al.*, 1975; Kim *et al.*, 1991; Itaya & Iida, 1994; Itaya *et al.*, 2002), a crystallographic study has never been published. We report the structure of (I) here.



(I)

The structure of (I) contains two independent molecules in the asymmetric unit. Each comprises two fused six-membered rings: a cyclohexyl ring and a cyclic diester. The average $Csp^3—Csp^3$ bond distance is 1.524 (10) Å within both molecules *A* and *B*. All C═O and C—O bonds are in close agreement both within each molecule and between molecules A and B. Both cyclic diesters are twisted by about 60° about the ring fusion (C2–C7) as indicated by their respective torsion angles [O3—C2—C7—O4 = −60.0 (3)° and −60.7 (3)° for *A* and *B*, respectively]. In the crystal structure, there are three types of weak intermolecular C—H···O interactions (see Table 2).

Experimental

The title compound was prepared according to literature methods (Itaya *et al.*, 2002). X-ray quality crystals were grown by diffusion of hexanes into a concentrated benzene solution at 283 K.

Crystal data

$C_8H_{10}O_4$
M_r = 170.16
Orthorhombic, $Pna2_1$
a = 12.088 (5) Å
b = 20.492 (9) Å
c = 6.289 (3) Å
V = 1557.8 (12) Å^3
Z = 8
D_x = 1.451 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 54 reflections
θ = 5.2–46.1°
μ = 0.12 mm^{-1}
T = 110 (2) K
Needle, colorless
0.65 × 0.25 × 0.05 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω and φ scans
Absorption correction: none
9120 measured reflections
1930 independent reflections
1657 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.5°
$h = -15 \rightarrow 14$
$k = -23 \rightarrow 26$
$l = -8 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.141
S = 1.11
1930 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0926P)^2 + 0.117P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1*A*—O1*A*	1.196 (4)	O1*B*—C1*B*	1.197 (4)
C1*A*—O3*A*	1.327 (4)	O2*B*—C8*B*	1.205 (4)
O2*A*—C8*A*	1.197 (4)	C1*B*—O3*B*	1.331 (4)
O3*A*—C2*A*	1.468 (4)	O3*B*—C2*B*	1.467 (4)
C7*A*—O4*A*	1.464 (4)	C7*B*—O4*B*	1.475 (4)
O4*A*—C8*A*	1.323 (4)	O4*B*—C8*B*	1.331 (4)
O1*A*—C1*A*—O3*A*	122.1 (3)	O1*B*—C1*B*—O3*B*	122.1 (3)
O1*A*—C1*A*—C8*A*	120.3 (3)	O1*B*—C1*B*—C8*B*	120.1 (3)
O3*A*—C1*A*—C8*A*	117.5 (3)	O3*B*—C1*B*—C8*B*	117.6 (3)
C1*A*—O3*A*—C2*A*	116.8 (2)	C1*B*—O3*B*—C2*B*	116.2 (3)
O3*A*—C2*A*—C7*A*	109.1 (3)	O3*B*—C2*B*—C7*B*	108.6 (3)
O3*A*—C2*A*—C3*A*	106.6 (2)	O3*B*—C2*B*—C3*B*	107.1 (3)
O4*A*—C7*A*—C2*A*	109.3 (3)	O4*B*—C7*B*—C2*B*	108.8 (3)
O4*A*—C7*A*—C6*A*	110.4 (3)	O4*B*—C7*B*—C6*B*	111.2 (3)
C8*A*—O4*A*—C7*A*	119.2 (2)	C8*B*—O4*B*—C7*B*	119.8 (3)
O2*A*—C8*A*—O4*A*	121.5 (3)	O2*B*—C8*B*—O4*B*	121.0 (3)
O2*A*—C8*A*—C1*A*	119.3 (3)	O2*B*—C8*B*—C1*B*	120.2 (3)
O4*A*—C8*A*—C1*A*	119.2 (3)	O4*B*—C8*B*—C1*B*	118.7 (3)
C1*A*—O3*A*—C2*A*—C3*A*	170.9 (3)	C1*B*—O3*B*—C2*B*—C3*B*	174.9 (3)
O3*A*—C2*A*—C7*A*—O4*A*	−60.0 (3)	O3*B*—C2*B*—C7*B*—O4*B*	−60.3 (3)
C6*A*—C7*A*—O4*A*—C8*A*	−91.9 (4)	C6*B*—C7*B*—O4*B*—C8*B*	−93.3 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2*B*—H2*B*···O4A^{i}	1.00	2.53	3.355 (4)	139
C2*B*—H2*B*···O1A^{ii}	1.00	2.43	3.136 (4)	127
C5*A*—H511···O2A^{iii}	0.99	2.45	3.195 (4)	131

Symmetry codes: (i) $x, y, 1+z$; (ii) $x-\frac{1}{2}, \frac{3}{2}-y, 1+z$; (iii) $x-\frac{1}{2}, \frac{3}{2}-y, z$.

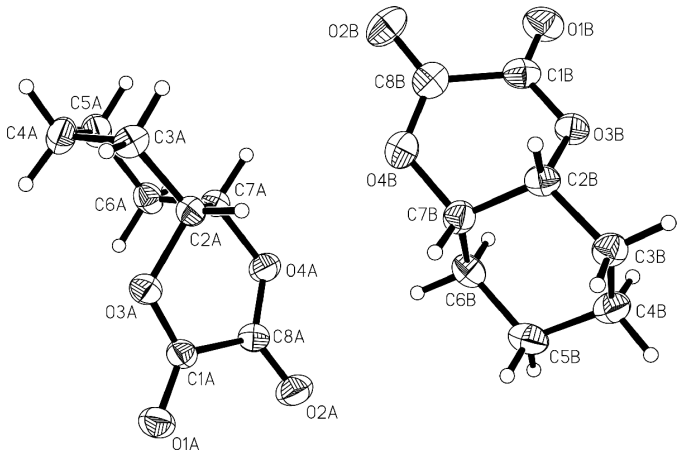


Figure 1
The asymmetric unit of (I) (50% probability displacement ellipsoids), with the atomic numbering scheme.

All H atoms were placed in calculated positions, with C—H distances fixed at 0.99–1.00 Å and U_{iso} values at 1.2U_{eq} of the carrier C atom. In the absence of anomalous dispersion effects, all Friedel pairs were merged for the final refinement. We are, however, certain of the reported stereochemistry as we began from *cis*-1,2-cyclohexanediol as described in the literature (Itaya *et al.*, 2002).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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