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Cyclobutane-1,2-dione

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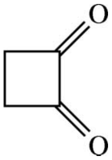
Key indicators: single-crystal X-ray study; *T* = 200 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.031; *wR* factor = 0.081; data-to-parameter ratio = 9.9.

The title compound, $C_4H_4O_2$, was prepared as an intermediate in the synthesis of α-hydroxycyclopropanecarboxylic acid. The structure of this intermediate has only been deduced previously from ^{1}H NMR spectra, elemental analysis or chemical derivatization. A single-crystal X-ray analysis was carried out to unambiguously assign the correct structure. The cyclobutane ring is almost planar.

Related literature

Details of the synthesis of the title compound were given by Heine (1971). For related literature, see: Flack (1983).



Experimental

Crystal data

$C_4H_4O_2$
$M_r = 84.07$
Orthorhombic, $P2_12_12_1$
$a = 5.3719\ (3)$ Å
$b = 6.8819\ (3)$ Å
$c = 10.8378\ (6)$ Å
$V = 400.66\ (4)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 200\ (2)$ K
0.20 × 0.17 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
3074 measured reflections
557 independent reflections
488 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.081$
$S = 1.09$
557 reflections
56 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{\rm max} = 0.12$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.15$ e Å^{-3}

Data collection: *COLLECT* (Bruker Nonius, 2004); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2049).

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supplementary materials

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Cyclobutane-1,2-dione

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Comment

The title compound, $C_4H_4O_2$, was prepared as an intermediate in the synthesis of α-hydroxycyclopropanecarboxylic acid. In the crystal structure the cyclobutane ring is almost planar (Fig. 1).

Experimental

The title compound was prepared according to a published procedure (Heine, 1971) by reaction of bromine on 1,2-bis(trimethylsilyloxy)-cyclobut-1,2-ene in *n*-pentane. Crystals were obtained upon warming to room temperature and storage of the reaction batch at ambient temperature under exclusion of light for 72 h.

Spectroscopic data: ^{1}H NMR (399.8 MHz, $CDCl_3$, 24 °C) δ/p.p.m.: 3.07 (s, 4 H, CH_2). $^{13}C\{^1H\}$ NMR (100.5 MHz, $CDCl_3$, 26 °C) δ/p.p.m.: 207.3 (CO), 41.8 (CH_2).

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 $U_{iso}(H) = 0.048$ (3) Å^2.

Due to the absence of significant anomalous scattering the absolute structure factor (Flack, 1983), which is 2.9 with an estimated standard deviation of 2 for the unmerged data set, is meaningless. Thus, Friedel opposites (351 pairs) have been merged. The absolute structure has been arbitrarily chosen.

Figures

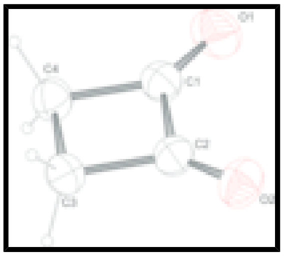

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

Cyclobutane-1,2-dione

Crystal data

$C_4H_4O_2$	$F_{000} = 176$

$M_r = 84.07$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 5.3719$ (3) Å

$b = 6.8819$ (3) Å

$c = 10.8378$ (6) Å

$V = 400.66$ (4) Å^3

$Z = 4$

$D_x = 1.394$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 5161 reflections

$\theta = 3.1$–27.5°

$\mu = 0.11$ mm^{-1}

$T = 200$ (2) K

Block, yellow–orange

0.20 × 0.17 × 0.14 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

3074 measured reflections

557 independent reflections

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

$R_{\text{int}} = 0.038$

$\theta_{\text{max}} = 27.5$°

$\theta_{\text{min}} = 3.5$°

$h = -6 \rightarrow 6$

$k = -8 \rightarrow 8$

$l = -13 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.081$

$S = 1.09$

557 reflections

56 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

Only H-atom displacement parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.294P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\text{max}} = 0.12$ e Å^{-3}

$\Delta\rho_{\text{min}} = -0.15$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

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

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.0632 (3)	0.2198 (2)	0.25237 (14)	0.0496 (4)
O2	−0.0783 (3)	0.42078 (19)	0.51173 (12)	0.0429 (4)
C1	0.1626 (4)	0.3358 (2)	0.31712 (16)	0.0327 (4)
C2	0.0924 (3)	0.4362 (2)	0.44147 (14)	0.0310 (4)
C3	0.3323 (4)	0.5531 (3)	0.43585 (16)	0.0359 (4)
H31	0.4477	0.5256	0.5049	0.048 (3)*
H32	0.3051	0.6947	0.4267	0.048 (3)*
C4	0.4048 (4)	0.4512 (3)	0.31457 (16)	0.0358 (4)
H41	0.4181	0.5408	0.2434	0.048 (3)*
H42	0.5555	0.3693	0.3216	0.048 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0511 (9)	0.0464 (8)	0.0513 (7)	−0.0045 (8)	−0.0136 (9)	−0.0149 (7)
O2	0.0370 (7)	0.0500 (8)	0.0416 (7)	0.0022 (7)	0.0056 (7)	0.0056 (6)
C1	0.0340 (9)	0.0310 (8)	0.0331 (8)	0.0007 (8)	−0.0038 (8)	0.0012 (7)
C2	0.0303 (9)	0.0314 (8)	0.0311 (8)	0.0020 (8)	−0.0043 (8)	0.0029 (7)
C3	0.0328 (9)	0.0337 (9)	0.0413 (9)	−0.0030 (8)	−0.0033 (8)	−0.0053 (8)
C4	0.0338 (9)	0.0356 (9)	0.0380 (8)	−0.0026 (8)	0.0028 (8)	0.0031 (8)

Geometric parameters (Å, °)

O1—C1	1.190 (2)	C3—C4	1.540 (2)
O2—C2	1.196 (2)	C3—H31	0.9900
C1—C4	1.524 (3)	C3—H32	0.9900
C1—C2	1.561 (2)	C4—H41	0.9900
C2—C3	1.520 (3)	C4—H42	0.9900
O1—C1—C4	136.23 (18)	C2—C3—H32	113.6
O1—C1—C2	134.26 (18)	C4—C3—H32	113.6
C4—C1—C2	89.50 (13)	H31—C3—H32	110.8
O2—C2—C3	136.21 (16)	C1—C4—C3	90.33 (14)
O2—C2—C1	134.07 (17)	C1—C4—H41	113.6
C3—C2—C1	89.70 (13)	C3—C4—H41	113.6
C2—C3—C4	90.44 (13)	C1—C4—H42	113.6
C2—C3—H31	113.6	C3—C4—H42	113.6
C4—C3—H31	113.6	H41—C4—H42	110.9
O1—C1—C2—O2	3.7 (4)	C1—C2—C3—C4	−1.11 (12)
C4—C1—C2—O2	−177.2 (2)	O1—C1—C4—C3	178.0 (2)
O1—C1—C2—C3	−178.0 (2)	C2—C1—C4—C3	−1.11 (12)
C4—C1—C2—C3	1.12 (12)	C2—C3—C4—C1	1.14 (13)
O2—C2—C3—C4	177.2 (2)		

Fig. 1