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1,4-Dibromobutane-2,3-dione

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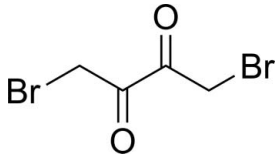
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.013 Å; R factor = 0.065; wR factor = 0.108; data-to-parameter ratio = 16.6.

The asymmetric unit of the title compound, $C_4H_4Br_2O_2$, contains one half-molecule, being located about a centre of inversion. In the crystal, there are no significant intermolecular interactions.

Related literature

For the uses of 1,4-dibromobutane-2,3-dione, see: Gogte *et al.* (1967). For the synthesis of 1,4-dibromobutane-2,3-dione, see: Ruggli & Herzog (1946). For the cystal structure of the 1,4-dichloro analogue, see: Ducourant *et al.* (1986). For bond–length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_4H_4Br_2O_2$
M_r = 243.89
Orthorhombic, *Pbca*
a = 6.945 (1) Å
b = 5.542 (1) Å
c = 17.238 (3) Å
V = 663.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 12.13 mm^{-1}
T = 298 K
0.10 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.195, T_{max} = 0.377
614 measured reflections
614 independent reflections
319 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
3 standard reflections every 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.108
S = 0.93
614 reflections
37 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2517).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Ducourant, B., Maury, C., Lere-Porte, J.-P., Petrissans, J. & Ribet, J.-L. (1986). *Acta Cryst.* C**42**, 341–343.
Enraf–Nonius (1985). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Gogte, V. N., Shan, L. G., Tilak, B. D., Gadekar, K. N. & Sahasrabudhe, M. B. (1967). *Tetrahedron*, **23**, 2437–2441.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Ruggli, P. & Herzog, M. (1946). *Helv. Chim. Acta.*, 29, 95-101.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

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1,4-Dibromobutane-2,3-dione

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S1. Comment

1,4-Dibromobutane-2,3-dione and its derivatives are important intermediates for the synthesis of compounds possessing promising anticancer activity, which is attributed to their likely interference in the hexose-monophosphate (HMP) pathway (Gogte *et al.*, 1967). We report herein on the crystal structure of the title compound.

In the title molecule, Fig. 1, the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The asymmetric unit contains one half-molecule, being located on a centre of inversion. It can be compared to the 1,4-dichloro derivative that crystallized in the monoclinic space group $P2_1/c$ (Ducourant *et al.*, 1986) but which also possesses inversion symmetry.

In the crystal, there are no significant intermolecular interactions (Fig. 2).

S2. Experimental

1,4-Dibromobutane-2,3-dione was prepared by the method reported in the literature (Ruggli & Herzog, 1946). Yellow plate-like crystals were obtained by dissolving the title compound (0.50 g, 2.05 mmol) in dichloromethane (30 ml) and evaporating the solvent slowly at room temperature for ca. 2 days.

S3. Refinement

The methylene H atoms were positioned geometrically and refined as riding atoms: C-H = 0.97 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$.

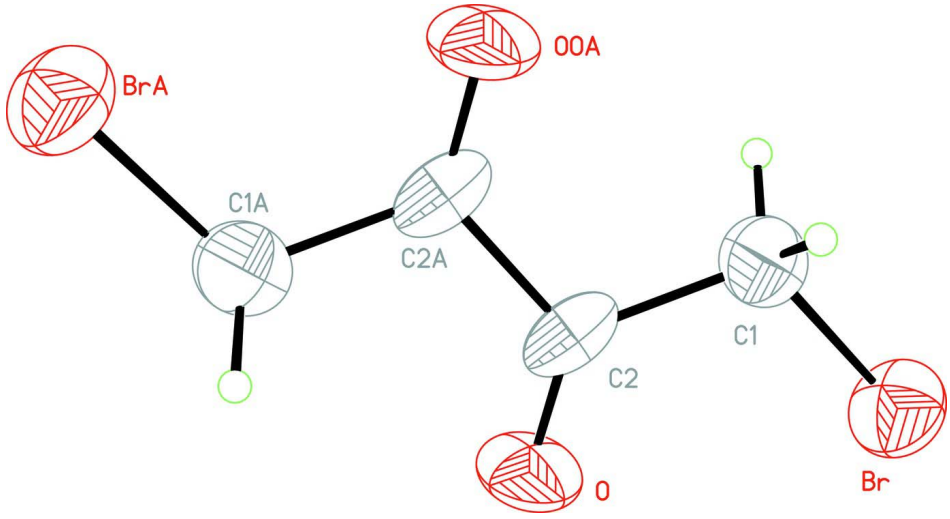

Figure 1

The molecular structure of the title molecule, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level [Symmetry code: (a) -x+2, -y+1, -z+1].

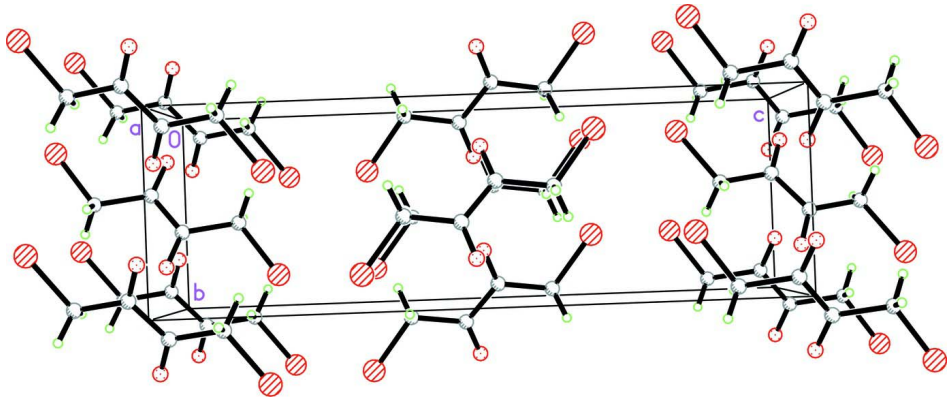

Figure 2

A view along the a axis of the crystal packing of the title compound.

1,4-Dibromobutane-2,3-dione

Crystal data

$C_4H_4Br_2O_2$
$M_r = 243.89$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 6.945$ (1) Å
$b = 5.542$ (1) Å
$c = 17.238$ (3) Å
$V = 663.5$ (2) Å^3
$Z = 4$
$F(000) = 456$

$D_x = 2.442$ Mg m^{-3}
Melting point < 395 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–14°
$\mu = 12.13$ mm^{-1}
$T = 298$ K
Cube, yellow
0.10 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.195, T_{max} = 0.377
614 measured reflections
614 independent reflections
319 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
θ_{max} = 25.4°, θ_{min} = 2.4°
h = 0→8
k = −6→6
l = −20→20
3 standard reflections every 120 min
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.108
S = 0.93
614 reflections
37 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

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 $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.96430 (19)	0.17863 (19)	0.67746 (8)	0.0634 (5)
O	0.8640 (13)	0.2368 (15)	0.5056 (4)	0.062 (2)
C1	1.0276 (16)	0.4425 (18)	0.6134 (6)	0.055 (3)
H1A	0.9689	0.5875	0.6342	0.066*
H1B	1.1661	0.4652	0.6138	0.066*
C2	0.9577 (15)	0.4056 (11)	0.5267 (6)	0.039 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0755 (7)	0.0501 (7)	0.0647 (9)	−0.0027 (7)	0.0041 (7)	0.0097 (6)
O	0.076 (5)	0.036 (3)	0.073 (6)	−0.026 (5)	−0.019 (5)	0.007 (3)
C1	0.045 (6)	0.068 (7)	0.052 (7)	0.000 (6)	0.009 (6)	−0.016 (7)
C2	0.033 (5)	0.028 (4)	0.057 (7)	0.013 (6)	0.023 (5)	0.007 (5)

Geometric parameters (Å, º)

Br—C1	1.885 (10)	C1—H1A	0.9700
O—C2	1.196 (11)	C1—H1B	0.9700
C1—C2	1.585 (9)	C2—C2^{i}	1.512 (16)
C2—C1—Br	112.4 (6)	H1A—C1—H1B	107.9
C2—C1—H1A	109.1	O—C2—C2^{i}	124.6 (12)
Br—C1—H1A	109.1	O—C2—C1	123.7 (8)
C2—C1—H1B	109.1	C2^{i}—C2—C1	111.4 (10)
Br—C1—H1B	109.1		
Br—C1—C2—O	4.7 (13)	Br—C1—C2—C2^{i}	−169.0 (8)

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.