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2,5-Dibromoterephthalic acid dihydrate

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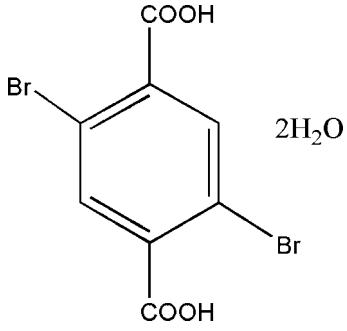
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.049; wR factor = 0.117; data-to-parameter ratio = 15.0.

The asymmetric unit of the title compound, $C_8H_4Br_2O_4\cdot 2H_2O$, contains one half-molecule of 2,5-dibromoterephthalic acid (DBTA) and one water molecule. The DBTA molecule is centrosymmetric. In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules, forming a three-dimensional framework.

Related literature

For general background, see: Yao & Tour (1999). For a related structure, see: Singh & Bedi (1957).



Experimental

Crystal data

$C_8H_4Br_2O_4\cdot 2H_2O$
M_r = 359.94
Monoclinic, $P2_1/c$
a = 10.670 (2) Å
b = 7.413 (1) Å
c = 7.074 (1) Å
β = 92.74 (3)°
V = 558.89 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 7.26 mm^{-1}
T = 293 (2) K
0.10 × 0.10 × 0.08 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.530, T_{max} = 0.594
(expected range = 0.499–0.559)
1003 measured reflections
1003 independent reflections
763 reflections with $I > 2\sigma(I)$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.117
S = 1.05
1003 reflections
67 parameters
21 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
OW—HWA···O1^{i}	0.85	2.11	2.903 (9)	155
OW—HWB···O1^{ii}	0.85	2.22	2.944 (9)	142
O2—H2A···OW	0.82	1.75	2.566 (8)	177

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2130).

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S1. Comment

2,5-Dibromoterephthalic acid (DBTA) is an important intermediate in the preparation of flame-retardant polymers (Yao *et al.*, 1999). We report herein the crystal structure of the title compound (I).

The asymmetric unit of I (Fig. 1), contains one half of a molecule of 2,5-dibromoterephthalic acid (DBTA), which is related to the other half by a center of symmetry, and one water molecule. Three neighbouring DBTA molecules are linked through one water molecule by intermolecular O—H···O hydrogen bonds, to form a three dimensional framework.

S2. Experimental

The title compound was prepared according to the method described by Singh & Bedi (1957). Crystals of (I) suitable for X-ray analysis were obtained by dissolving DBTA (2.0 g) in water (80 ml) and evaporating slowly at room temperature for about 15 d.

S3. Refinement

Anisotropic parameters of the C atoms in the phenyl ring were restrained to have equal components and approximately isotropic behavior. H atoms were positioned geometrically, with O—H = 0.82 (for OH) and 0.85 (for H_2O) and C—H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C/O), where x = 1.2 for aromatic H and x = 1.5 for other H.

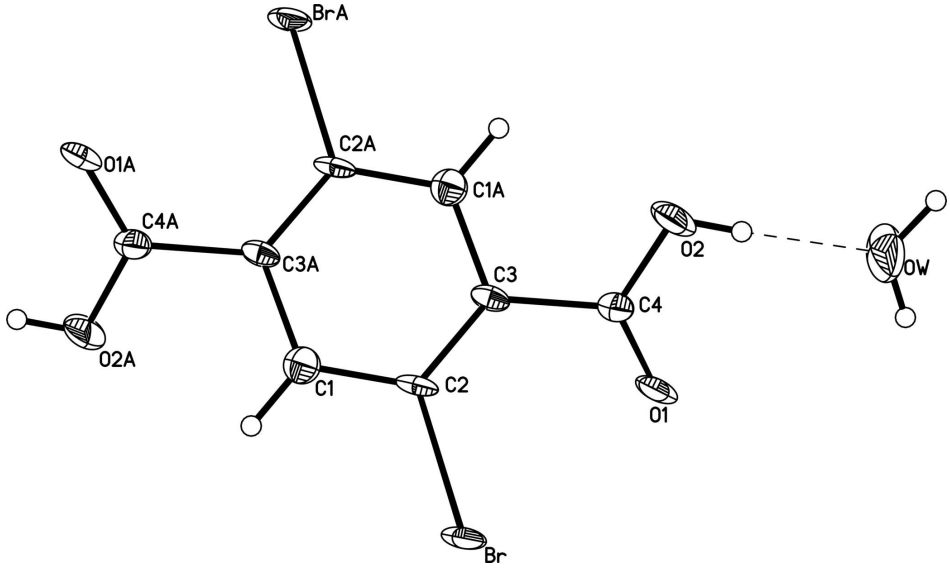

Figure 1

The molecular structure of (I), showing the atom labelling scheme. Anisotropic displacement parameters are shown at the 50% probability level.

2,5-Dibromoterephthalic acid dihydrate

Crystal data

$C_8H_4Br_2O_4·2H_2O$
M_r = 359.94
Monoclinic, $P2_1/c$
a = 10.670 (2) Å
b = 7.413 (1) Å
c = 7.074 (1) Å
β = 92.74 (3)°
V = 558.89 (15) Å^3
Z = 2
$F(000)$ = 348
D_x = 2.139 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–13°
μ = 7.26 mm^{-1}
T = 293 K
Block, colorless
0.10 × 0.10 × 0.08 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} = 0.530, T_{max} = 0.594
1003 measured reflections
1003 independent reflections
763 reflections with $I > 2\sigma(I)$
R_{int} = 0.000
θ_{max} = 25.2°, θ_{min} = 1.9°
h = −12→12
k = 0→8
l = 0→8
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.117
S = 1.05
1003 reflections
67 parameters
21 restraints
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

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 1.5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.70$ 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. *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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.31754 (7)	0.35260 (9)	0.52687 (10)	0.0366 (3)
OW	−0.0069 (5)	−0.2828 (11)	0.7105 (11)	0.093 (3)
HWA	−0.0608	−0.2028	0.6780	0.111*
HWB	−0.0299	−0.3786	0.7651	0.111*
O1	0.1532 (5)	0.0220 (6)	0.5124 (8)	0.0495 (14)
O2	0.2259 (5)	−0.2252 (7)	0.6570 (9)	0.0554 (15)
H2A	0.1515	−0.2402	0.6767	0.083*
C1	0.5431 (6)	0.1725 (10)	0.4729 (9)	0.034
H1A	0.5725	0.2885	0.4518	0.040*
C2	0.4182 (6)	0.1457 (8)	0.5085 (9)	0.0276 (13)
C3	0.3736 (6)	−0.0245 (8)	0.5317 (8)	0.0255 (13)
C4	0.2396 (6)	−0.0748 (9)	0.5662 (9)	0.0314 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0487 (4)	0.0111 (4)	0.0496 (5)	0.0050 (3)	−0.0007 (3)	0.0002 (3)
OW	0.030 (3)	0.111 (6)	0.139 (7)	0.008 (3)	0.013 (3)	0.073 (5)
O1	0.045 (3)	0.016 (3)	0.087 (4)	−0.004 (2)	−0.002 (3)	0.015 (3)
O2	0.051 (3)	0.028 (3)	0.087 (4)	−0.006 (3)	−0.004 (3)	0.029 (3)
C1	0.034	0.034	0.034	0.000	0.002	0.000
C2	0.043 (3)	0.009 (3)	0.030 (3)	0.002 (3)	−0.009 (3)	0.000 (3)
C3	0.038 (3)	0.013 (3)	0.025 (3)	−0.002 (3)	−0.003 (2)	−0.004 (3)
C4	0.035 (3)	0.022 (3)	0.038 (4)	0.003 (3)	0.004 (3)	0.005 (3)

Geometric parameters (Å, º)

Br—C2	1.880 (6)	C1—C2	1.383 (8)
OW—HWA	0.8500	C1—C3^{i}	1.413 (9)
OW—HWB	0.8500	C1—H1A	0.9300
O1—C4	1.215 (8)	C2—C3	1.361 (8)

O2—C4	1.299 (8)	C3—C1^{i}	1.413 (9)
O2—H2A	0.8200	C3—C4	1.508 (9)
HWA—OW—HWB	120.0	C1—C2—Br	117.0 (5)
C4—O2—H2A	109.5	C2—C3—C1^{i}	119.5 (6)
C2—C1—C3^{i}	120.4 (6)	C2—C3—C4	126.0 (6)
C2—C1—H1A	119.8	C1^{i}—C3—C4	114.5 (5)
C3^{i}—C1—H1A	119.8	O1—C4—O2	124.1 (6)
C3—C2—C1	120.1 (6)	O1—C4—C3	121.0 (6)
C3—C2—Br	122.9 (5)	O2—C4—C3	115.0 (6)
C3^{i}—C1—C2—C3	−2.7 (10)	Br—C2—C3—C4	2.8 (9)
C3^{i}—C1—C2—Br	176.2 (5)	C2—C3—C4—O1	26.1 (10)
C1—C2—C3—C1^{i}	2.7 (10)	C1^{i}—C3—C4—O1	−154.9 (6)
Br—C2—C3—C1^{i}	−176.2 (5)	C2—C3—C4—O2	−153.4 (7)
C1—C2—C3—C4	−178.3 (6)	C1^{i}—C3—C4—O2	25.6 (8)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O*W*—H*WA*···O1^{ii}	0.85	2.11	2.903 (9)	155
O*W*—H*WB*···O1^{iii}	0.85	2.22	2.944 (9)	142
O2—H2*A*···O*W*	0.82	1.75	2.566 (8)	177

Symmetry codes: (ii) −*x*, −*y*, −*z*+1; (iii) −*x*, *y*−1/2, −*z*+3/2.