Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,6-Dibromoisophthalic acid monohydrate

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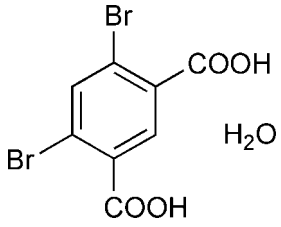
Received 22 July 2012; accepted 29 July 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.011 Å; R factor = 0.054; wR factor = 0.089; data-to-parameter ratio = 14.0.

In the crystal structure of the title hydrate, $C_8H_4Br_2O_4\cdot H_2O$, O—H···O hydrogen bonds link the molecules into a two-dimensional network parallel to (10$\bar{2}$). The acid groups of the main molecule and the water molecule are all involved in the supramolecular structure. The dihedral angles between the benzene ring and the acid groups are 37.8 (4) and 36.4 (5)°, while the dihedral angle between the acid groups is 10.9 (4)°.

Related literature

For the synthesis of the title compound, see: Singh & Bedi (1957). For a related structure, see: Song *et al.* (2008).



Experimental

Crystal data

$C_8H_4Br_2O_4\cdot H_2O$
M_r = 341.95
Monoclinic, $P2_1/c$
a = 3.8740 (8) Å
b = 17.366 (4) Å
c = 15.710 (3) Å
β = 90.91 (3)°
V = 1056.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.67 mm^{-1}
T = 293 K
0.30 × 0.05 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.207, T_{max} = 0.700
2206 measured reflections
1910 independent reflections
1109 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
3 standard reflections every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.089
S = 0.99
1910 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···OW	0.82	1.74	2.554 (8)	176
O4—H4B···O1^{i}	0.82	1.86	2.665 (8)	168
OW—HWB···O3^{ii}	0.85	2.08	2.893 (9)	159
OW—HWA···O3^{iii}	0.85	2.17	2.903 (9)	144

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

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*.

The author thanks the Center of Testing and Analysis, Nanjing University, for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2450).

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Song, G.-L., Liu, S., Liu, H.-J., Zeng, T. & Zhu, H.-J. (2008). *Acta Cryst.* E**64**, o1860.

supporting information

Acta Cryst. (2012). E**68**, o2642 [doi:10.1107/S1600536812033892]

4,6-Dibromoisophthalic acid monohydrate

Bao-fen Ye

S1. Comment

4,6-Dibromoisophthalic acid (DBPA) is an important organic intermediate for organic synthesis, which can be used in many fields such as organic light-emitting materials. We report herein the crystal structure of the hydrate of DBPA (Fig. 1). Bond lengths and angles are within normal ranges (Song *et al.*, 2008). The asymmetric unit contains one 4,6-dibromoisophthalic acid molecule and one water molecule, in general positions. In the crystal, O—H···O hydrogen bonds link the molecules to form a bidimensional framework (Fig. 2), where all OH groups of the DBPA and the water molecules are involved, as well as carbonyl groups. The water molecule serves as donor and acceptor for hydrogen bonding.

S2. Experimental

DBPA was prepared according to the literature method (Singh & Bedi, 1957). Single crystals of the title hydrate suitable for X-ray analysis were obtained by dissolving DBPA (2.0 g) in water (80 ml) and evaporating the solution slowly at room temperature for about 15 days.

S3. Refinement

All H atoms may be found in a difference map, but their positions were fixed in the final refinement with idealized bond lengths of 0.82 (OH of acid), 0.85 (water molecule) or 0.93 Å (aromatic CH). Isotropic displacement parameters for H atoms were calculated as U_{iso}(H) = xU_{eq}(parent atom), where x = 1.5 for acid OH groups, and x = 1.2 for other H atoms.

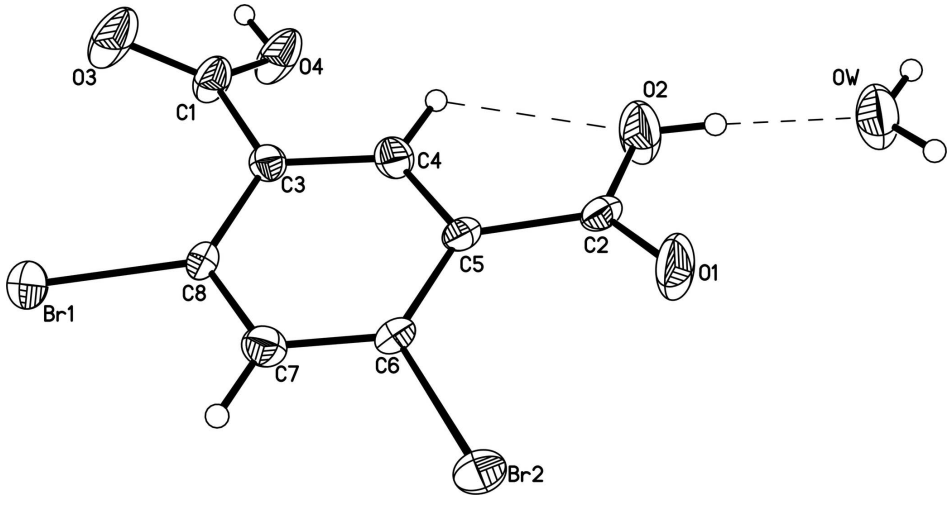

Figure 1

The molecular structure of the title compound, with displacement ellipsoids at the 30% probability level. Dashed lines are non-bonding contacts.

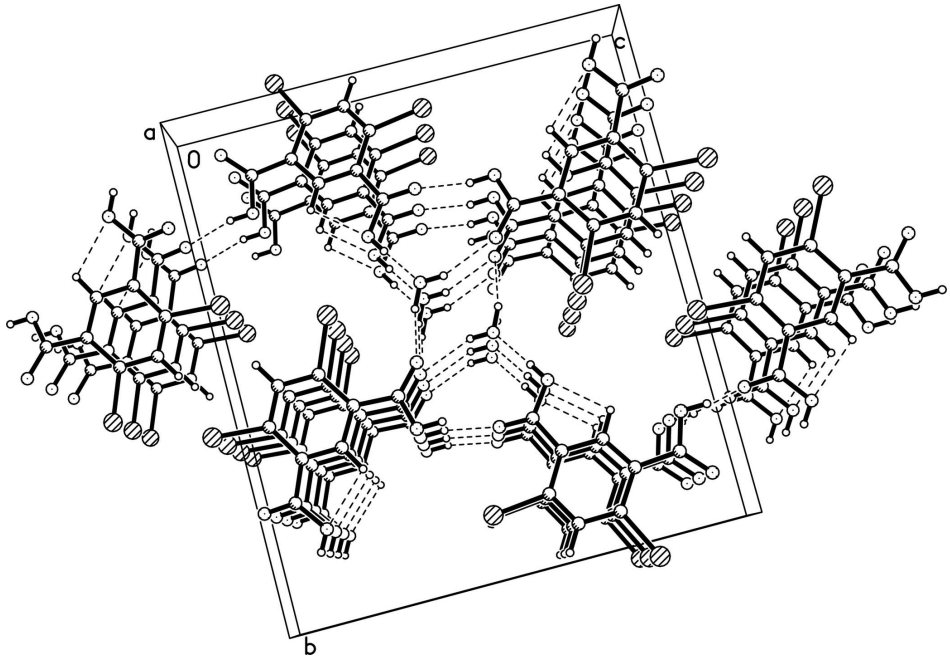

Figure 2

A packing diagram for the title compound, with O—H···O intermolecular hydrogen bonds shown as dashed lines.

4,6-Dibromoisophthalic acid monohydrate

Crystal data

$C_8H_4Br_2O_4·H_2O$
M_r = 341.95
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 3.8740 (8) Å
b = 17.366 (4) Å
c = 15.710 (3) Å
β = 90.91 (3)°
V = 1056.8 (4) Å^3
Z = 4
$F(000)$ = 656
D_x = 2.149 Mg m^{-3}
Melting point = 441–443 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–14°
μ = 7.67 mm^{-1}
T = 293 K
Rodlike, colourless
0.30 × 0.05 × 0.05 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} = 0.207, T_{max} = 0.700
2206 measured reflections
1910 independent reflections
1109 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 25.2°, θ_{min} = 1.8°
h = −4→4
k = 0→20
l = 0→18
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.089$
$S = 0.99$
1910 reflections
136 parameters
0 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.63$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
OW	0.6960 (16)	0.0557 (3)	−0.0592 (4)	0.079 (2)
HWB	0.8013	0.0536	−0.0114	0.094*
HWA	0.6946	0.0116	−0.0828	0.094*
Br1	−0.1479 (2)	0.52659 (5)	−0.24930 (5)	0.0486 (3)
O1	0.6060 (18)	0.2282 (4)	−0.0212 (4)	0.073 (2)
C1	−0.041 (2)	0.3595 (5)	−0.3421 (5)	0.041 (2)
Br2	0.3840 (2)	0.38158 (6)	0.03673 (5)	0.0473 (3)
C2	0.4568 (19)	0.2307 (5)	−0.0891 (5)	0.0314 (19)
O2	0.4320 (16)	0.1710 (3)	−0.1373 (4)	0.071 (2)
H2A	0.5248	0.1342	−0.1136	0.107*
C3	0.0622 (17)	0.3658 (4)	−0.2523 (5)	0.0297 (19)
O3	−0.0053 (17)	0.4099 (4)	−0.3936 (4)	0.070 (2)
C4	0.1931 (18)	0.3018 (5)	−0.2089 (5)	0.034 (2)
H4A	0.2057	0.2556	−0.2385	0.041*
O4	−0.1724 (15)	0.2937 (4)	−0.3616 (3)	0.0598 (18)
H4B	−0.2295	0.2935	−0.4121	0.090*
C5	0.3038 (18)	0.3022 (5)	−0.1261 (4)	0.0294 (19)
C6	0.2627 (17)	0.3713 (5)	−0.0796 (4)	0.0315 (19)
C7	0.1313 (18)	0.4354 (5)	−0.1184 (5)	0.036 (2)
H7A	0.1089	0.4807	−0.0873	0.043*
C8	0.0319 (18)	0.4341 (4)	−0.2022 (4)	0.0293 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
OW	0.131 (6)	0.051 (4)	0.053 (4)	0.036 (4)	−0.027 (4)	−0.007 (4)
Br1	0.0605 (6)	0.0433 (5)	0.0422 (5)	0.0132 (5)	0.0028 (4)	0.0097 (5)
O1	0.131 (6)	0.049 (4)	0.039 (4)	0.029 (4)	−0.031 (4)	0.003 (3)
C1	0.057 (6)	0.040 (6)	0.027 (5)	−0.004 (5)	−0.007 (4)	0.006 (5)
Br2	0.0647 (6)	0.0517 (6)	0.0254 (4)	0.0066 (5)	−0.0047 (4)	−0.0072 (5)
C2	0.036 (5)	0.039 (5)	0.019 (4)	0.000 (4)	0.003 (4)	0.001 (4)
O2	0.126 (6)	0.038 (4)	0.050 (4)	0.023 (4)	−0.025 (4)	−0.006 (4)
C3	0.029 (5)	0.032 (5)	0.028 (4)	0.004 (4)	0.000 (3)	0.000 (4)
O3	0.125 (6)	0.061 (5)	0.024 (3)	−0.023 (4)	−0.011 (3)	0.006 (4)

C4	0.039 (5)	0.025 (4)	0.038 (5)	−0.002 (4)	0.002 (4)	−0.003 (4)
O4	0.097 (5)	0.056 (5)	0.026 (3)	−0.024 (4)	−0.014 (3)	0.005 (3)
C5	0.035 (5)	0.031 (5)	0.022 (4)	0.000 (4)	0.007 (4)	0.002 (4)
C6	0.038 (5)	0.037 (5)	0.020 (4)	0.001 (4)	0.005 (3)	0.004 (4)
C7	0.037 (5)	0.036 (5)	0.034 (5)	0.002 (4)	0.001 (4)	−0.005 (4)
C8	0.038 (5)	0.026 (4)	0.024 (4)	0.001 (4)	0.003 (3)	0.007 (4)

Geometric parameters (Å, º)

OW—HWB	0.8500	O2—H2A	0.8200
OW—HWA	0.8502	C3—C4	1.394 (10)
Br1—C8	1.896 (7)	C3—C8	1.430 (10)
O1—C2	1.205 (9)	C4—C5	1.363 (9)
C1—O3	1.201 (9)	C4—H4A	0.9300
C1—O4	1.285 (10)	O4—H4B	0.8200
C1—C3	1.465 (10)	C5—C6	1.414 (10)
Br2—C6	1.888 (7)	C6—C7	1.363 (10)
C2—O2	1.287 (9)	C7—C8	1.367 (10)
C2—C5	1.491 (10)	C7—H7A	0.9300
HWB—OW—HWA	110.2	C1—O4—H4B	109.5
O3—C1—O4	122.5 (7)	C4—C5—C6	117.4 (7)
O3—C1—C3	124.2 (8)	C4—C5—C2	119.0 (7)
O4—C1—C3	113.3 (8)	C6—C5—C2	123.6 (6)
O1—C2—O2	121.4 (8)	C7—C6—C5	120.4 (7)
O1—C2—C5	123.9 (8)	C7—C6—Br2	116.2 (6)
O2—C2—C5	114.6 (6)	C5—C6—Br2	123.4 (6)
C2—O2—H2A	109.5	C6—C7—C8	121.0 (8)
C4—C3—C8	115.1 (6)	C6—C7—H7A	119.5
C4—C3—C1	120.2 (7)	C8—C7—H7A	119.5
C8—C3—C1	124.7 (7)	C7—C8—C3	121.3 (7)
C5—C4—C3	124.7 (8)	C7—C8—Br1	117.3 (6)
C5—C4—H4A	117.7	C3—C8—Br1	121.4 (5)
C3—C4—H4A	117.7		
O3—C1—C3—C4	−144.6 (9)	C4—C5—C6—C7	−2.9 (11)
O4—C1—C3—C4	34.9 (11)	C2—C5—C6—C7	177.8 (7)
O3—C1—C3—C8	37.0 (13)	C4—C5—C6—Br2	177.5 (5)
O4—C1—C3—C8	−143.5 (8)	C2—C5—C6—Br2	−1.9 (10)
C8—C3—C4—C5	−3.2 (11)	C5—C6—C7—C8	1.0 (12)
C1—C3—C4—C5	178.2 (7)	Br2—C6—C7—C8	−179.3 (6)
C3—C4—C5—C6	4.1 (11)	C6—C7—C8—C3	−0.1 (12)
C3—C4—C5—C2	−176.5 (7)	C6—C7—C8—Br1	179.9 (6)
O1—C2—C5—C4	169.0 (8)	C4—C3—C8—C7	1.0 (11)
O2—C2—C5—C4	−8.0 (10)	C1—C3—C8—C7	179.5 (7)
O1—C2—C5—C6	−11.7 (13)	C4—C3—C8—Br1	−178.9 (5)
O2—C2—C5—C6	171.3 (7)	C1—C3—C8—Br1	−0.4 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O*W*	0.82	1.74	2.554 (8)	176
O4—H4*B*···O1^{i}	0.82	1.86	2.665 (8)	168
O*W*—H*WB*···O3^{ii}	0.85	2.08	2.893 (9)	159
O*W*—H*WA*···O3^{iii}	0.85	2.17	2.903 (9)	144
C4—H4*A*···O2	0.93	2.33	2.692 (10)	103

Symmetry codes: (i) $x-1, -y+1/2, z-1/2$; (ii) $x+1, -y+1/2, z+1/2$; (iii) $-x+1, y-1/2, -z-1/2$.