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4-Hydroxy-1-oxo-1,2-dihydrophthalazine-6,7-dicarboxylic acid dihydrate

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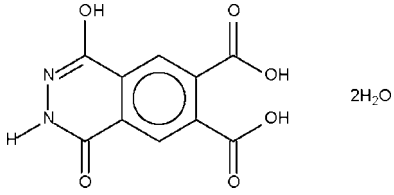
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.105; data-to-parameter ratio = 11.4.

In the crystal structure of the title compound, $C_{10}H_6N_2O_6\cdot 2H_2O$, the OH and NH groups each serve as a hydrogen-bond donor to one acceptor site whereas the water molecules each serve as a hydrogen-bond donor to two acceptor sites. The hydrogen-bonding scheme gives rise to a three-dimensional network.

Related literature

For the structure of bis(hydrazinium) 4-hydroxy-1-oxo-*2H*-phthalazine-6,7-dicarboxylate, see: Benniston *et al.* (1999).



Experimental

Crystal data

$C_{10}H_6N_2O_6\cdot 2H_2O$
M_r = 286.20
Triclinic, $P\overline{1}$
a = 6.4069 (1) Å
b = 9.4254 (2) Å
c = 9.6922 (2) Å
α = 82.843 (2)°
β = 87.496 (1)°
γ = 73.451 (2)°
V = 556.65 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 100 (2) K
0.33 × 0.31 × 0.09 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
4702 measured reflections
2530 independent reflections
2160 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.105
S = 1.06
2530 reflections
221 parameters
10 restraints
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···O1w	0.84 (1)	1.78 (1)	2.615 (1)	175 (2)
O3—H3o···O6^{i}	0.84 (1)	1.79 (1)	2.637 (1)	176 (2)
O5—H5o···N1^{ii}	0.85 (1)	1.91 (1)	2.744 (1)	168 (2)
N2—H2···O2w^{iii}	0.89 (1)	1.82 (1)	2.695 (1)	167 (2)
O1w—H11···O6^{iv}	0.85 (1)	1.91 (1)	2.758 (1)	173 (2)
O1w—H12···O3^{v}	0.85 (1)	2.31 (1)	3.052 (1)	146 (2)
O2w—H21···O4	0.84 (1)	1.96 (1)	2.771 (1)	162 (2)
O2w—H22···O2^{vi}	0.83 (1)	2.37 (2)	3.050 (1)	139 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2271).

References

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supplementary materials

Acta Cryst. (2008). E**64**, o1225 [**doi:10.1107/S1600536808014347**]

4-Hydroxy-1-oxo-1,2-dihydrophthalazine-6,7-dicarboxylic acid dihydrate

L.-L. Liang, J.-S. Zhao and S. W. Ng

Comment

Benzene-1,2,4,5-tetracarboxylic acid reacts with hydrazine to form bis(hydrazinium) 4-hydroxy-1-oxo-*2H*-phthalazine-6,7-dicarboxylate, whose anion represents a ligand possesses a recognition site for metals as well as a rich hydrogen-bonding motif (Benniston *et al.*, 1999). The neutral acid itself would be more useful for the synthesis of metal derivatives; the neutral acid has been unexpectedly obtained when the reaction was carried out in the presence of a cobaltous salt. The acid crystallizes as a dihydrate (Scheme I, Fig. 1). The –OH and –NH groups each serves as hydrogen-bond donor to one acceptor site whereas the water molecules each serves as hydrogen bond donor to two acceptor sites. The hydrogen bonding scheme gives rise to a three-dimensional network.

Experimental

Hydrazine hydrate (0.01 g, 0.2 mmol), pyromellitic acid (0.05 g, 0.2 mmol), cobaltous chloride hexahydrate (0.02 g, 0.1 mmol) and water (10 ml) were heated in a 25 ml, Teflon-lined Parr bomb at 433 K for 96 h. The bomb was cooled to room temperature at 10 K per hour.

Refinement

All hydrogen atoms were located in a difference Fouier map, and were refined with distance restraints (C–H 0.95±0.01, N–H 0.88±0.01 and O–H 0.84±0.01 Å). Temperature factors were freely refined.

Figures

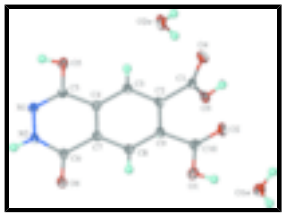

Fig. 1. Molecular structure of (I) showing atomic labelling scheme and displacement ellipsoids at the 70% probability level.

4-Hydroxy-1-oxo-1,2-dihydrophthalazine-6,7-dicarboxylic acid dihydrate

Crystal data

$C_{10}H_6N_2O_6·2H_2O$	$Z = 2$
$M_r = 286.20$	$F_{000} = 296$
Triclinic, $P\overline{1}$	$D_x = 1.708$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å

a = 6.4069 (1) Å	Cell parameters from 2234 reflections
b = 9.4254 (2) Å	θ = 2.9–28.2º
c = 9.6922 (2) Å	μ = 0.15 mm^{-1}
α = 82.843 (2)º	T = 100 (2) K
β = 87.496 (1)º	Prism, colorless
γ = 73.451 (2)º	0.33 × 0.31 × 0.09 mm
V = 556.65 (2) Å^3	

Data collection

Bruker SMART APEX diffractometer	2160 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.017
Monochromator: graphite	θ_{max} = 27.5º
T = 100(2) K	θ_{min} = 2.1º
ω scans	h = −8→8
Absorption correction: None	k = −12→11
4702 measured reflections	l = −12→11
2530 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.035	All H-atom parameters refined
$wR(F^2)$ = 0.105	$w = 1/[\sigma^2(F_o^2) + (0.0657P)^2 + 0.0694P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
2530 reflections	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
221 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
10 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.92046 (15)	0.93958 (11)	0.64792 (10)	0.0166 (2)
O2	0.86240 (15)	0.86579 (11)	0.87194 (10)	0.0195 (2)
O3	0.44292 (15)	0.84970 (10)	1.00234 (9)	0.0155 (2)
O4	0.68492 (16)	0.62355 (11)	1.02233 (9)	0.0199 (2)
O5	0.10736 (15)	0.53241 (11)	0.66473 (9)	0.0180 (2)
O6	0.49784 (14)	0.82489 (10)	0.27322 (9)	0.0147 (2)
O1W	1.14691 (16)	1.06674 (12)	0.78414 (11)	0.0207 (2)
O2W	0.85169 (16)	0.35754 (12)	0.90954 (10)	0.0213 (2)
N1	0.16571 (17)	0.60686 (12)	0.43675 (11)	0.0136 (2)

N2	0.27394 (17)	0.68218 (12)	0.34120 (11)	0.0134 (2)
C1	0.5698 (2)	0.73086 (14)	0.95287 (13)	0.0129 (3)
C2	0.54372 (19)	0.73590 (14)	0.79857 (13)	0.0120 (3)
C3	0.39766 (19)	0.66628 (14)	0.75724 (13)	0.0126 (3)
C4	0.35973 (19)	0.67201 (13)	0.61528 (12)	0.0116 (3)
C5	0.20578 (19)	0.60273 (14)	0.56725 (13)	0.0126 (3)
C6	0.41699 (19)	0.75457 (14)	0.36872 (13)	0.0117 (3)
C7	0.46733 (19)	0.74720 (14)	0.51629 (12)	0.0114 (3)
C8	0.61956 (19)	0.81353 (14)	0.55846 (13)	0.0119 (3)
C9	0.65794 (19)	0.80760 (14)	0.69923 (12)	0.0117 (3)
C10	0.8230 (2)	0.87406 (14)	0.74930 (13)	0.0137 (3)
H10	0.998 (3)	0.980 (2)	0.6878 (19)	0.042 (6)*
H30	0.458 (3)	0.838 (2)	1.0895 (10)	0.036 (5)*
H50	0.026 (3)	0.496 (2)	0.622 (2)	0.048 (6)*
H11	1.253 (2)	1.101 (2)	0.760 (2)	0.044 (6)*
H12	1.180 (3)	1.0209 (19)	0.8645 (12)	0.034 (5)*
H21	0.824 (3)	0.4435 (13)	0.9343 (19)	0.034 (5)*
H22	0.975 (2)	0.314 (3)	0.942 (3)	0.074 (8)*
H2	0.241 (3)	0.681 (2)	0.2530 (11)	0.037 (5)*
H3	0.320 (2)	0.6187 (16)	0.8236 (13)	0.017 (4)*
H8	0.693 (2)	0.8618 (17)	0.4898 (14)	0.019 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0159 (4)	0.0209 (5)	0.0166 (5)	−0.0107 (4)	0.0004 (4)	−0.0025 (4)
O2	0.0220 (5)	0.0270 (6)	0.0137 (5)	−0.0129 (4)	−0.0045 (4)	−0.0019 (4)
O3	0.0206 (5)	0.0168 (5)	0.0094 (4)	−0.0052 (4)	−0.0009 (3)	−0.0032 (4)
O4	0.0267 (5)	0.0175 (5)	0.0140 (5)	−0.0033 (4)	−0.0056 (4)	−0.0007 (4)
O5	0.0223 (5)	0.0245 (5)	0.0128 (5)	−0.0160 (4)	−0.0010 (4)	−0.0007 (4)
O6	0.0173 (4)	0.0192 (5)	0.0096 (4)	−0.0085 (4)	0.0005 (3)	−0.0015 (4)
O1W	0.0195 (5)	0.0238 (6)	0.0217 (5)	−0.0118 (4)	−0.0040 (4)	0.0009 (4)
O2W	0.0232 (5)	0.0215 (6)	0.0187 (5)	−0.0019 (4)	−0.0073 (4)	−0.0075 (4)
N1	0.0151 (5)	0.0144 (5)	0.0128 (5)	−0.0068 (4)	−0.0008 (4)	−0.0009 (4)
N2	0.0175 (5)	0.0161 (6)	0.0083 (5)	−0.0074 (4)	−0.0015 (4)	−0.0013 (4)
C1	0.0152 (6)	0.0155 (6)	0.0108 (6)	−0.0088 (5)	−0.0009 (4)	−0.0011 (5)
C2	0.0128 (6)	0.0120 (6)	0.0102 (6)	−0.0013 (5)	−0.0017 (4)	−0.0016 (5)
C3	0.0143 (6)	0.0139 (6)	0.0101 (6)	−0.0050 (5)	−0.0001 (4)	−0.0007 (5)
C4	0.0122 (6)	0.0104 (6)	0.0120 (6)	−0.0025 (5)	−0.0011 (4)	−0.0025 (5)
C5	0.0143 (6)	0.0115 (6)	0.0123 (6)	−0.0038 (5)	−0.0012 (5)	−0.0017 (5)
C6	0.0120 (5)	0.0123 (6)	0.0108 (6)	−0.0025 (5)	−0.0001 (4)	−0.0031 (4)
C7	0.0119 (5)	0.0113 (6)	0.0102 (6)	−0.0014 (4)	−0.0005 (4)	−0.0023 (5)
C8	0.0125 (6)	0.0120 (6)	0.0107 (6)	−0.0031 (5)	0.0009 (4)	−0.0009 (5)
C9	0.0120 (6)	0.0120 (6)	0.0109 (6)	−0.0026 (5)	−0.0012 (4)	−0.0020 (5)
C10	0.0126 (6)	0.0134 (6)	0.0147 (6)	−0.0025 (5)	−0.0004 (5)	−0.0025 (5)

Geometric parameters (Å, °)

O1—C10	1.3235 (15)	N2—C6	1.3415 (16)

O1—H10	0.84 (1)	N2—H2	0.89 (1)
O2—C10	1.2143 (16)	C1—C2	1.5054 (17)
O3—C1	1.3139 (15)	C2—C3	1.3838 (17)
O3—H30	0.84 (1)	C2—C9	1.4061 (16)
O4—C1	1.2121 (16)	C3—C4	1.3993 (17)
O5—C5	1.3265 (15)	C3—H3	0.941 (9)
O5—H50	0.85 (1)	C4—C7	1.3961 (16)
O6—C6	1.2511 (15)	C4—C5	1.4487 (16)
O1W—H11	0.85 (1)	C6—C7	1.4688 (17)
O1W—H12	0.85 (1)	C7—C8	1.3983 (17)
O2W—H21	0.84 (1)	C8—C9	1.3884 (17)
O2W—H22	0.83 (1)	C8—H8	0.942 (9)
N1—C5	1.2953 (16)	C9—C10	1.4981 (17)
N1—N2	1.3794 (14)		
C10—O1—H10	105.3 (14)	C3—C4—C5	121.13 (11)
C1—O3—H30	107.8 (13)	N1—C5—O5	120.97 (11)
C5—O5—H50	106.0 (15)	N1—C5—C4	122.70 (11)
H11—O1W—H12	104.8 (18)	O5—C5—C4	116.32 (11)
H21—O2W—H22	104 (2)	O6—C6—N2	121.07 (11)
C5—N1—N2	117.77 (10)	O6—C6—C7	123.23 (11)
C6—N2—N1	126.82 (10)	N2—C6—C7	115.70 (11)
C6—N2—H2	119.3 (12)	C4—C7—C8	119.99 (11)
N1—N2—H2	113.9 (12)	C4—C7—C6	118.67 (11)
O4—C1—O3	125.08 (12)	C8—C7—C6	121.35 (11)
O4—C1—C2	122.51 (12)	C9—C8—C7	119.53 (11)
O3—C1—C2	112.24 (10)	C9—C8—H8	121.9 (10)
C3—C2—C9	120.51 (11)	C7—C8—H8	118.6 (10)
C3—C2—C1	116.41 (11)	C8—C9—C2	120.16 (11)
C9—C2—C1	123.07 (11)	C8—C9—C10	121.46 (11)
C2—C3—C4	119.19 (11)	C2—C9—C10	118.37 (11)
C2—C3—H3	120.6 (9)	O2—C10—O1	124.29 (11)
C4—C3—H3	120.2 (10)	O2—C10—C9	122.03 (11)
C7—C4—C3	120.56 (11)	O1—C10—C9	113.68 (11)
C7—C4—C5	118.31 (11)		
C5—N1—N2—C6	−0.85 (19)	C3—C4—C7—C6	−177.98 (11)
O4—C1—C2—C3	81.31 (15)	C5—C4—C7—C6	0.97 (17)
O3—C1—C2—C3	−94.09 (13)	O6—C6—C7—C4	176.75 (11)
O4—C1—C2—C9	−99.19 (15)	N2—C6—C7—C4	−2.25 (17)
O3—C1—C2—C9	85.40 (14)	O6—C6—C7—C8	−3.27 (19)
C9—C2—C3—C4	−1.98 (19)	N2—C6—C7—C8	177.73 (11)
C1—C2—C3—C4	177.53 (11)	C4—C7—C8—C9	−1.82 (19)
C2—C3—C4—C7	−0.12 (19)	C6—C7—C8—C9	178.19 (11)
C2—C3—C4—C5	−179.04 (11)	C7—C8—C9—C2	−0.26 (19)
N2—N1—C5—O5	179.93 (10)	C7—C8—C9—C10	178.62 (11)
N2—N1—C5—C4	−0.70 (18)	C3—C2—C9—C8	2.19 (19)
C7—C4—C5—N1	0.56 (19)	C1—C2—C9—C8	−177.29 (11)
C3—C4—C5—N1	179.50 (12)	C3—C2—C9—C10	−176.73 (11)
C7—C4—C5—O5	179.95 (11)	C1—C2—C9—C10	3.80 (18)

C3—C4—C5—O5	−1.10 (18)	C8—C9—C10—O2	−177.84 (12)
N1—N2—C6—O6	−176.72 (11)	C2—C9—C10—O2	1.06 (19)
N1—N2—C6—C7	2.30 (18)	C8—C9—C10—O1	1.46 (17)
C3—C4—C7—C8	2.03 (19)	C2—C9—C10—O1	−179.64 (10)
C5—C4—C7—C8	−179.02 (11)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···O1w	0.84 (1)	1.78 (1)	2.615 (1)	175 (2)
O3—H3o···O6^{i}	0.84 (1)	1.79 (1)	2.637 (1)	176 (2)
O5—H5o···N1^{ii}	0.85 (1)	1.91 (1)	2.744 (1)	168 (2)
N2—H2···O2w^{iii}	0.89 (1)	1.82 (1)	2.695 (1)	167 (2)
O1w—H11···O6^{iv}	0.85 (1)	1.91 (1)	2.758 (1)	173 (2)
O1w—H12···O3^{v}	0.85 (1)	2.31 (1)	3.052 (1)	146 (2)
O2w—H21···O4	0.84 (1)	1.96 (1)	2.771 (1)	162 (2)
O2w—H22···O2^{vi}	0.83 (1)	2.37 (2)	3.050 (1)	139 (2)

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) −*x*, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) −*x*+2, −*y*+2, −*z*+1; (v) *x*+1, *y*, *z*; (vi) −*x*+2, −*y*+1, −*z*+2.

Fig. 1