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

Ling-Ling Liang,[a] Jian-She Zhao[a] and Seik Weng Ng[b]*

[a]Department of Chemistry, Shaanxi Key Laboratory for Physico-Inorganic Chemistry, Northwest University, Xi'an 710069, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

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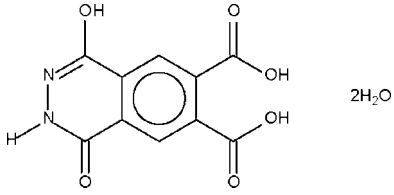
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.105; data-to-parameter ratio = 11.4.

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).

We thank Northwest University and the University of Malaya for supporting this study.

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

References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2008). *publCIF*. In preparation.

supporting information

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

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

Ling-Ling Liang, Jian-She Zhao and Seik Weng Ng

S1. 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.

S2. 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.

S3. 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.

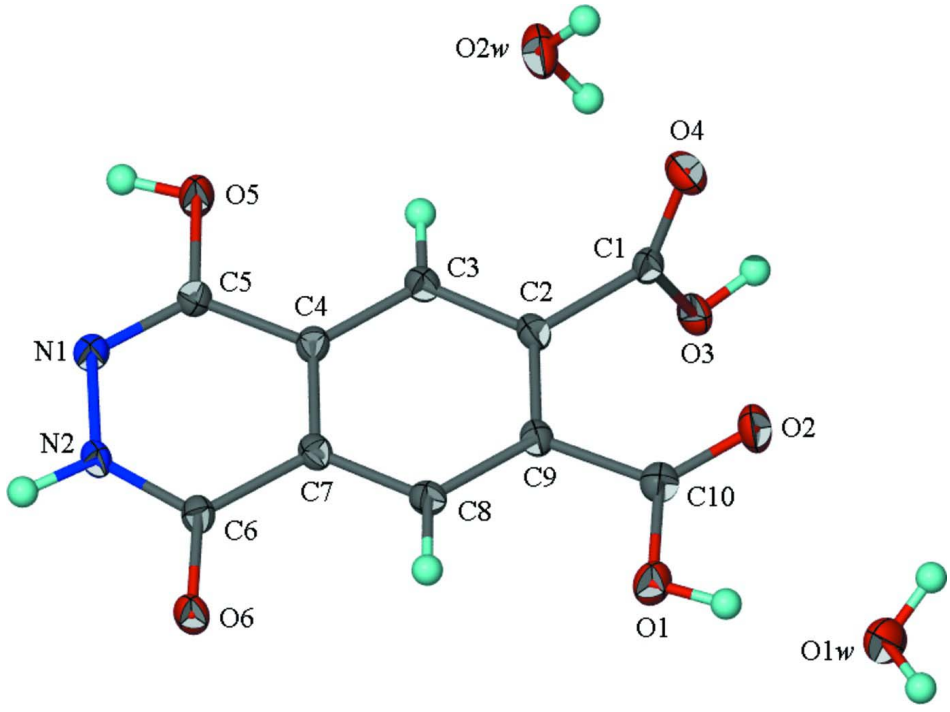

Figure 1

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

$C_{10}H_6N_2O_6{\cdot}2H_2O$
$M_r = 286.20$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.4069\ (1)$ Å
$b = 9.4254\ (2)$ Å
$c = 9.6922\ (2)$ Å
$\alpha = 82.843\ (2)°$
$\beta = 87.496\ (1)°$
$\gamma = 73.451\ (2)°$
$V = 556.65\ (2)$ Å^3
$Z = 2$
$F(000) = 296$
$D_x = 1.708$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2234 reflections
$\theta = 2.9–28.2°$
$\mu = 0.15$ mm^{-1}
$T = 100$ K
Prism, colorless
0.33 × 0.31 × 0.09 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
4702 measured reflections
2530 independent reflections
2160 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$
$h = -8\rightarrow8$
$k = -12\rightarrow11$
$l = -12\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.105$
$S = 1.06$
2530 reflections
221 parameters
10 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0657P)^2 + 0.0694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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 (Å²)

	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, º)

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, º)

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.