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4-Hydroxy-6-[(4-hydroxy-1-oxo-1,2-dihydrophthalazin-6-yl)carbonyl]-phthalazin-1(2*H*)-one

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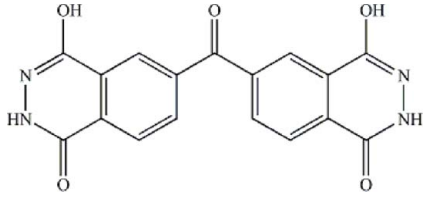
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.059; wR factor = 0.161; data-to-parameter ratio = 11.4.

In the crystal structure of the title compound, $C_{17}H_{10}N_4O_5$, the molecules lie on twofold axes (through the ketone bridge C and O atoms). The dihedral angle between the two phthalazine rings is 52.25 (1)°. In the crystal, intermolecular N—H···O and O—H···O interactions link the molecules.

Related literature

For the acylate reaction of polycarboxylate with hydrazine hydrate, see: Benniston *et al.* (1999); Hu *et al.* (2004).



Experimental

Crystal data

$C_{17}H_{10}N_4O_5$
M_r = 350.29
Monoclinic, $C2/c$
a = 11.576 (3) Å
b = 10.511 (3) Å
c = 12.274 (3) Å
β = 111.718 (4)°
V = 1387.4 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1995)
T_{min} = 0.963, T_{max} = 0.987
3800 measured reflections
1370 independent reflections
774 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.161
S = 0.99
1370 reflections
120 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.85	1.76	2.581 (3)	163
N2—H2···O1^{ii}	0.86	2.19	3.034 (4)	168

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2110).

References

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supplementary materials

Acta Cryst. (2009). E**65**, o3050 [**doi:10.1107/S1600536809046509**]

4-Hydroxy-6-[(4-hydroxy-1-oxo-1,2-dihydrophthalazin-6-yl)carbonyl]phthalazin-1(2*H*)-one

R.-S. Zhou and J.-F. Song

Comment

In situ hydrothermal acylate reaction of multidentate aromatic polycarboxylate with hydrazine hydrate has been investigated (Benniston *et al.*, 1999; Hu *et al.*, 2004). We intend to select 3, 3'-4, 4'-benzophenonetetracarboxylic dianhydride as the ligand to continue the exploration of the *in situ* acylate reaction. However, the crystals of the title compound were obtained unintentionally as the harvested product of the hydrothermal reaction of 3, 3'-4, 4'-benzophenonetetracarboxylic dianhydride, $CoCl_2$ and hydrazine hydrate. In the title compound, a twofold axis lies in the C atom and O atom of the ketone bridge (Fig. 1). Each organic molecule connects six adjacent ones into a three-dimensional supramolecular network by intermolecular O—H···O (2.576 Å) and N—H···O (3.034 Å) hydrogen bonds (Fig. 2 and Fig. 3).

Experimental

Yellow needle-like crystals of the title compound were synthesized hydrothermally from a mixture of $CoCl_2.H_2O$ (0.0230 g), 3, 3'-4, 4'-benzophenonetetracarboxylic dianhydride (0.0641 g), hydrazine hydrate (0.028 ml), and deionized water (15 ml) in a 23 ml Teflon-lined stainless steel autoclave under autogenous pressure heated to 170 °C for 4 days and cooled to room temperature. Crystalline product was filtered, washed with distilled water, and dried at ambient temperature.

Refinement

The H atoms were positioned geometrically and refined using riding model with C—H = 0.93 Å, N—H = 0.85 Å and isotropic displacement parameters $U_{iso}(H) = 1.2U(C_{eq} / N_{eq})$. However, the H of the O1 atom was located in a difference Fourier map refined as riding, with $U_{iso}(H) = 1.5U_{eq}(O)$.

Figures

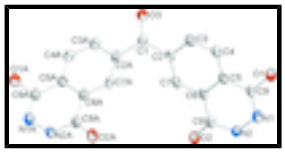


Fig. 1. The molecular structure of the title compound shown with 30% probabilty ellipsoids [symmetry code: (A) 1 - *x*, *y*, 1.5 - *z*].

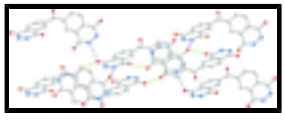


Fig. 2. View of the crystal packing of the title compound, with O—H···O and N—H···O hydrogen bonds drawn as dashed lines [see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity].

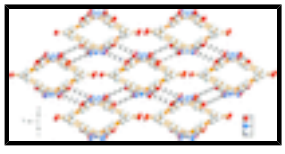


Fig. 3. Three-dimensional network formed by hydrogen bonds (dashed lines).

4-Hydroxy-6-[(4-hydroxy-1-oxo-1,2-dihydrophthalazin-6-yl)carbonyl]phthalazin- 1(2*H*)-one

Crystal data

$C_{17}H_{10}N_4O_5$	F_{000} = 720
M_r = 350.29	D_x = 1.677 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation, λ = 0.71073 Å
a = 11.576 (3) Å	θ = 2.7–26.0º
b = 10.511 (3) Å	μ = 0.13 mm^{-1}
c = 12.274 (3) Å	*T* = 293 K
β = 111.718 (4)º	Needle-like, yellow
V = 1387.4 (6) Å^3	0.30 × 0.25 × 0.10 mm
Z = 4	

Data collection

Bruker SMART APEX CCD diffractometer	1370 independent reflections
Radiation source: fine-focus sealed tube	774 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.057
T = 293 K	θ_{max} = 26.0º
ω scans	θ_{min} = 2.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1995)	*h* = −14→12
T_{min} = 0.963, T_{max} = 0.987	*k* = −12→12
3800 measured reflections	*l* = −15→14

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.059	H-atom parameters constrained
$wR(F^2)$ = 0.161	$w = 1/[\sigma^2(F_o^2) + (0.0758P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.99	$(\Delta/\sigma)_{max}$ = 0.005
1370 reflections	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
120 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.5000	−0.2105 (3)	0.7500	0.0411 (9)
O1	0.1272 (2)	0.0713 (2)	0.23363 (19)	0.0468 (7)
H1	0.0902	0.1164	0.1732	0.070*
O2	0.5078 (2)	0.3361 (2)	0.5277 (2)	0.0512 (8)
N1	0.2410 (3)	0.2518 (3)	0.2824 (2)	0.0406 (8)
N2	0.3389 (3)	0.3149 (3)	0.3641 (2)	0.0367 (8)
H2	0.3513	0.3916	0.3467	0.044*
C6	0.3861 (3)	0.1464 (3)	0.5017 (3)	0.0303 (8)
C9	0.2158 (3)	0.1394 (3)	0.3114 (3)	0.0356 (9)
C5	0.2831 (3)	0.0802 (3)	0.4238 (2)	0.0314 (8)
C4	0.2480 (3)	−0.0367 (3)	0.4556 (3)	0.0362 (9)
H4	0.1781	−0.0790	0.4052	0.043*
C7	0.4564 (3)	0.0920 (3)	0.6103 (2)	0.0320 (8)
H5	0.5254	0.1346	0.6620	0.038*
C8	0.4182 (3)	0.2707 (3)	0.4689 (3)	0.0340 (8)
C3	0.3181 (3)	−0.0884 (3)	0.5624 (3)	0.0363 (9)
H3	0.2956	−0.1668	0.5835	0.044*
C1	0.5000	−0.0954 (4)	0.7500	0.0281 (10)
C2	0.4228 (3)	−0.0255 (3)	0.6404 (2)	0.0297 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.046 (2)	0.030 (2)	0.0369 (19)	0.000	0.0043 (17)	0.000
O1	0.0448 (16)	0.0439 (16)	0.0330 (14)	−0.0014 (13)	−0.0076 (12)	0.0032 (11)
O2	0.0573 (18)	0.0442 (16)	0.0328 (14)	−0.0200 (14)	−0.0057 (13)	0.0032 (12)
N1	0.0419 (18)	0.0406 (19)	0.0291 (15)	0.0021 (15)	0.0011 (14)	−0.0015 (13)
N2	0.0413 (18)	0.0273 (16)	0.0286 (15)	−0.0031 (13)	−0.0020 (14)	0.0025 (12)
C6	0.033 (2)	0.032 (2)	0.0226 (16)	−0.0007 (15)	0.0059 (16)	−0.0040 (14)
C9	0.038 (2)	0.033 (2)	0.0260 (18)	0.0018 (17)	0.0002 (17)	−0.0027 (15)
C5	0.0308 (19)	0.033 (2)	0.0254 (16)	0.0047 (16)	0.0051 (15)	−0.0033 (14)
C4	0.037 (2)	0.032 (2)	0.0297 (18)	−0.0059 (16)	0.0016 (17)	−0.0023 (15)
C7	0.0294 (19)	0.035 (2)	0.0231 (17)	−0.0043 (16)	−0.0001 (15)	−0.0035 (14)
C8	0.038 (2)	0.033 (2)	0.0254 (17)	−0.0009 (17)	0.0053 (16)	−0.0021 (15)
C3	0.039 (2)	0.033 (2)	0.0302 (18)	−0.0042 (17)	0.0050 (16)	−0.0010 (16)
C1	0.028 (3)	0.024 (3)	0.027 (2)	0.000	0.005 (2)	0.000
C2	0.035 (2)	0.030 (2)	0.0217 (17)	0.0007 (15)	0.0074 (16)	−0.0046 (13)

Geometric parameters (Å, °)

O3—C1	1.210 (5)	C9—C5	1.450 (4)
O1—C9	1.323 (4)	C5—C4	1.394 (4)
O1—H1	0.8501	C4—C3	1.373 (4)
O2—C8	1.231 (4)	C4—H4	0.9300
N1—C9	1.298 (4)	C7—C2	1.386 (4)
N1—N2	1.374 (4)	C7—H5	0.9300
N2—C8	1.357 (4)	C3—C2	1.401 (4)
N2—H2	0.8600	C3—H3	0.9300
C6—C7	1.401 (4)	C1—C2^{i}	1.502 (4)
C6—C5	1.406 (4)	C1—C2	1.502 (4)
C6—C8	1.455 (5)		
C9—O1—H1	109.5	C5—C4—H4	120.5
C9—N1—N2	116.6 (3)	C2—C7—C6	119.7 (3)
C8—N2—N1	127.8 (3)	C2—C7—H5	120.1
C8—N2—H2	116.1	C6—C7—H5	120.1
N1—N2—H2	116.1	O2—C8—N2	119.5 (3)
C7—C6—C5	119.5 (3)	O2—C8—C6	125.8 (3)
C7—C6—C8	120.8 (3)	N2—C8—C6	114.6 (3)
C5—C6—C8	119.7 (3)	C4—C3—C2	121.5 (3)
N1—C9—O1	119.2 (3)	C4—C3—H3	119.3
N1—C9—C5	123.8 (3)	C2—C3—H3	119.3
O1—C9—C5	117.0 (3)	O3—C1—C2^{i}	119.3 (2)
C4—C5—C6	120.5 (3)	O3—C1—C2	119.3 (2)
C4—C5—C9	122.3 (3)	C2^{i}—C1—C2	121.5 (4)
C6—C5—C9	117.2 (3)	C7—C2—C3	119.7 (3)
C3—C4—C5	119.0 (3)	C7—C2—C1	122.8 (3)
C3—C4—H4	120.5	C3—C2—C1	117.3 (3)
C9—N1—N2—C8	4.9 (5)	N1—N2—C8—O2	173.9 (3)
N2—N1—C9—O1	−176.2 (3)	N1—N2—C8—C6	−6.8 (5)
N2—N1—C9—C5	1.1 (5)	C7—C6—C8—O2	2.4 (5)
C7—C6—C5—C4	2.4 (5)	C5—C6—C8—O2	−177.7 (3)
C8—C6—C5—C4	−177.5 (3)	C7—C6—C8—N2	−176.8 (3)
C7—C6—C5—C9	−178.3 (3)	C5—C6—C8—N2	3.1 (4)
C8—C6—C5—C9	1.8 (5)	C5—C4—C3—C2	0.8 (5)
N1—C9—C5—C4	175.2 (3)	C6—C7—C2—C3	−0.6 (5)
O1—C9—C5—C4	−7.5 (5)	C6—C7—C2—C1	174.2 (3)
N1—C9—C5—C6	−4.1 (5)	C4—C3—C2—C7	0.6 (5)
O1—C9—C5—C6	173.2 (3)	C4—C3—C2—C1	−174.4 (3)
C6—C5—C4—C3	−2.4 (5)	O3—C1—C2—C7	−148.7 (2)
C9—C5—C4—C3	178.4 (3)	C2^{i}—C1—C2—C7	31.3 (2)
C5—C6—C7—C2	−0.9 (5)	O3—C1—C2—C3	26.2 (3)
C8—C6—C7—C2	179.0 (3)	C2^{i}—C1—C2—C3	−153.8 (3)

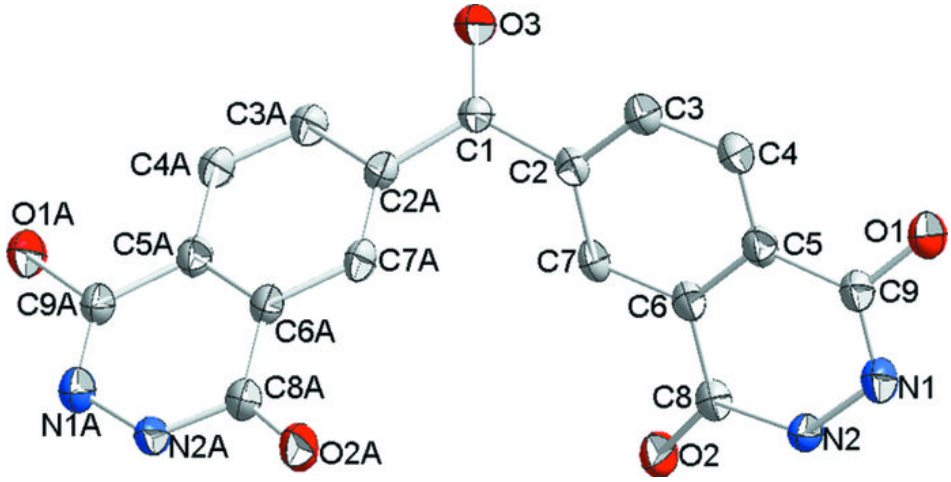
Symmetry codes: (i) −*x*+1, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{ii}	0.85	1.76	2.581 (3)	163
N2—H2···O1^{iii}	0.86	2.19	3.034 (4)	168

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

Fig. 1


O3
C3A
C1
C3
C4A
C2
C4
C2A
O1A
C5A
C7A
C7
C5
O1
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O2
N2

Fig. 2

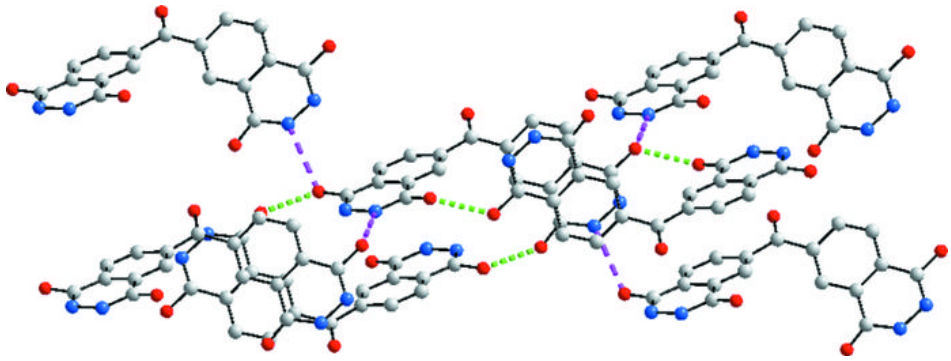

Fig. 3