organic compounds

N,N'-Bis[2-(quinolin-8-yloxy)acetoyl]-hydrazine dihydrate

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In the title compound, $C_{22}H_{18}N_4O_4 \cdot 2H_2O$, the hydrazine molecule lies across a twofold rotation axis, the mean planes of the two quinoline ring systems forming a dihedral angle of 64.1 (2)°. The hydrazine molecules and two water molecules are linked into infinite chains by O—H⋯O, O—H⋯N and N—H⋯O hydrogen bonds.

Related literature

For general literature concerning 8-hydroxyquinolines, see: Chen & Shi (1998).
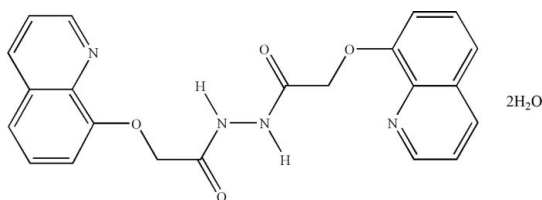
Experimental

Crystal data

$C_{22}H_{18}N_4O_4 \cdot 2H_2O$
$M_r = 438.44$
Monoclinic, $C2/c$
$a = 21.687$ (3) Å
$b = 8.3203$ (11) Å
$c = 13.8618$ (17) Å
$\beta = 124.528$ (2)°
$V = 2060.7$ (5) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 295$ K
$0.29 \times 0.25 \times 0.22$ mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.970$, $T_{max} = 0.988$
5251 measured reflections
1824 independent reflections
1263 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.088$
$S = 1.00$
1824 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O3—H13⋯O2^i	0.85	2.13	2.9745 (19)	175
O3—H12⋯N1	0.85	1.99	2.840 (1)	176
N2—H11⋯O3	0.87	1.97	2.815 (2)	164

Symmetry code: (i) $x, -y, z - \frac{1}{2}$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

References

Bruker (1997). SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, C. H. & Shi, J. M. (1998). Coord. Chem. Rev. **171**, 161–174.
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
Siemens (1996). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supplementary materials

N,N'-Bis[2-(quinolin-8-yloxy)acetoyl]hydrazine dihydrate

Z.-B. Zheng, R.-T. Wu, J.-K. Li and Y.-F. Sun

Comment

8-Hydroxyquinoline and its derivatives are amongst the most extensively investigated ligands in coordination chemistry (Chen & Shi, 1998). In the course of our studies on 8-hydroxyquinoline derivatives, the title compound was synthesized and its crystal structure determined.

In the crystal, the hydrazine molecule lies across a twofold rotation axis (Fig. 1). The mean planes of the two quinoline rings make a dihedral angle of 64.1 (2)°. The hydrazine molecules and the two water molecules are linked into infinite chains by O—H⋯O, O—H⋯N and N—H⋯O hydrogen bonds (Fig. 2).

Experimental

Sodium carbonate (0.01 mol) and 2-(quinolin-8-yloxy)acetyl chloride (0.014 mol) were added to a solution of 2-(quinolin-8-yloxy)acetohydrazide (0.01 mol) in tetrahydrofuran (20 ml) and water (20 ml). The solution was stirred at 313 K for 10 h, then poured into water (100 ml). The precipitated solid was filtered and recrystallized from ethanol (m.p. 368 K). Elemental analysis calculated: C 60.26, H 5.06, N 12.77%; found: C 60.24, H 5.08, N 12.76%. Crystals suitable for single-crystal X-ray analysis were selected directly from the sample after recrystallization.

Refinement

All H atoms were placed in idealized positions (C—H = 0.93–0.97 Å, O—H = 0.85 Å, N—H = 0.87 Å) and refined as riding atoms. For those bound to C, $U_{iso}(H) = 1.2U_{eq}(C)$. while for those bound to O or N, an isotropic displacement parameter was refined.
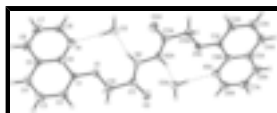
Figures

Fig. 1. The molecular structure with displacement ellipsoids drawn at the 50% probability level for non-H atoms. The dashed lines indicate hydrogen bonds.
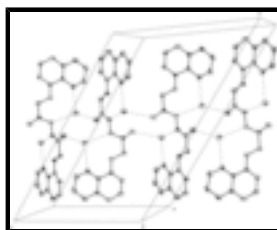

Fig. 2. The structure of the infinite chains formed *via* hydrogen bonds (dashed lines). H atoms are omitted.

supplementary materials

N,N′-Bis[2-(quinolin-8-yloxy)acetoyl]hydrazine dihydrate

Crystal data

$C_{22}H_{18}N_4O_4 \cdot 2H_2O$	$F_{000} = 920$
$M_r = 438.44$	$D_x = 1.413$ Mg m^{-3}
Monoclinic, $C2/c$	Melting point: 368 K
Hall symbol: -c 2yc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 21.687$ (3) Å	Cell parameters from 1050 reflections
$b = 8.3203$ (11) Å	$\theta = 2.7–23.7º$
$c = 13.8618$ (17) Å	$\mu = 0.11$ mm^{-1}
$\beta = 124.528$ (2)º	$T = 295$ K
$V = 2060.7$ (5) Å^3	Block, colourless
$Z = 4$	$0.29 \times 0.25 \times 0.22$ mm

Data collection

Bruker SMART CCD diffractometer	1824 independent reflections
Radiation source: fine-focus sealed tube	1263 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.032$
$T = 295$ K	$\theta_{max} = 25.1º$
φ and ω scans	$\theta_{min} = 2.3º$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -22 \rightarrow 25$
$T_{min} = 0.970$, $T_{max} = 0.988$	$k = -8 \rightarrow 9$
5251 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.039$	$w = 1/[\sigma^2(F_o^2) + (0.0279P)^2 + 1.2115P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.088$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.00$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
1824 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
146 parameters	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0018 (4)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.24176 (8)	0.20341 (19)	0.30918 (12)	0.0450 (4)
N2	0.03903 (8)	0.0311 (2)	0.28690 (13)	0.0485 (4)
H11	0.0611	0.0808	0.2592	0.058*
O1	0.18629 (6)	0.04914 (16)	0.41376 (10)	0.0442 (4)
O2	0.03928 (7)	−0.1099 (2)	0.42604 (12)	0.0648 (5)
O3	0.08291 (7)	0.20984 (19)	0.16292 (11)	0.0654 (5)
H12	0.1305	0.2125	0.2074	0.098*
H13	0.0732	0.1821	0.0966	0.098*
C1	0.26269 (9)	0.0511 (2)	0.47287 (14)	0.0371 (4)
C2	0.31124 (10)	−0.0184 (2)	0.57907 (15)	0.0472 (5)
H2	0.2928	−0.0705	0.6170	0.057*
C3	0.38840 (11)	−0.0123 (3)	0.63189 (18)	0.0605 (6)
H3	0.4204	−0.0610	0.7044	0.073*
C4	0.41744 (11)	0.0627 (3)	0.57978 (18)	0.0604 (6)
H4	0.4690	0.0652	0.6161	0.073*
C5	0.36924 (10)	0.1372 (2)	0.47012 (16)	0.0461 (5)
C6	0.39571 (12)	0.2199 (3)	0.41171 (19)	0.0596 (6)
H6	0.4468	0.2252	0.4446	0.071*
C7	0.34666 (13)	0.2913 (3)	0.3079 (2)	0.0623 (6)
H7	0.3635	0.3473	0.2690	0.075*
C8	0.27014 (12)	0.2799 (3)	0.25997 (18)	0.0555 (6)
H8	0.2371	0.3298	0.1884	0.067*
C9	0.29094 (9)	0.1327 (2)	0.41529 (14)	0.0385 (4)
C10	0.15646 (9)	−0.0393 (2)	0.46586 (15)	0.0426 (5)
H10A	0.1736	0.0080	0.5411	0.051*
H10B	0.1749	−0.1488	0.4795	0.051*
C11	0.07242 (10)	−0.0411 (2)	0.39007 (16)	0.0439 (5)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0467 (9)	0.0482 (10)	0.0398 (8)	−0.0047 (8)	0.0243 (8)	−0.0014 (8)

N2	0.0291 (8)	0.0727 (12)	0.0425 (9)	−0.0036 (8)	0.0196 (7)	0.0027 (8)
O1	0.0308 (7)	0.0596 (9)	0.0409 (7)	−0.0028 (6)	0.0196 (6)	0.0066 (6)
O2	0.0462 (8)	0.0922 (12)	0.0624 (9)	−0.0092 (8)	0.0346 (8)	0.0119 (8)
O3	0.0474 (8)	0.0945 (12)	0.0508 (8)	0.0049 (8)	0.0257 (7)	0.0052 (8)
C1	0.0303 (10)	0.0417 (11)	0.0372 (9)	−0.0058 (8)	0.0178 (8)	−0.0070 (8)
C2	0.0395 (11)	0.0552 (13)	0.0417 (11)	−0.0051 (10)	0.0198 (9)	0.0028 (10)
C3	0.0385 (12)	0.0743 (17)	0.0482 (12)	−0.0027 (11)	0.0122 (10)	0.0075 (11)
C4	0.0320 (11)	0.0773 (17)	0.0592 (13)	−0.0040 (11)	0.0182 (10)	0.0008 (12)
C5	0.0396 (11)	0.0490 (13)	0.0517 (11)	−0.0101 (10)	0.0271 (10)	−0.0102 (10)
C6	0.0516 (13)	0.0716 (16)	0.0660 (14)	−0.0215 (12)	0.0397 (12)	−0.0167 (13)
C7	0.0758 (16)	0.0670 (16)	0.0645 (14)	−0.0253 (13)	0.0520 (13)	−0.0134 (12)
C8	0.0706 (15)	0.0541 (14)	0.0480 (11)	−0.0118 (12)	0.0373 (11)	−0.0014 (10)
C9	0.0377 (10)	0.0390 (11)	0.0392 (10)	−0.0060 (8)	0.0220 (9)	−0.0081 (9)
C10	0.0388 (10)	0.0505 (12)	0.0402 (10)	−0.0041 (9)	0.0233 (9)	−0.0007 (9)
C11	0.0383 (11)	0.0537 (13)	0.0432 (11)	−0.0039 (10)	0.0253 (9)	−0.0053 (10)

Geometric parameters (Å, °)

N1—C8	1.313 (2)	C3—C4	1.351 (3)
N1—C9	1.367 (2)	C3—H3	0.930
N2—C11	1.325 (2)	C4—C5	1.409 (3)
N2—N2^i	1.396 (3)	C4—H4	0.930
N2—H11	0.870	C5—C6	1.409 (3)
O1—C1	1.3704 (19)	C5—C9	1.414 (2)
O1—C10	1.418 (2)	C6—C7	1.349 (3)
O2—C11	1.224 (2)	C6—H6	0.930
O3—H12	0.850	C7—C8	1.398 (3)
O3—H13	0.850	C7—H7	0.930
C1—C2	1.361 (2)	C8—H8	0.930
C1—C9	1.424 (2)	C10—C11	1.502 (2)
C2—C3	1.396 (3)	C10—H10A	0.970
C2—H2	0.930	C10—H10B	0.970
C8—N1—C9	117.26 (16)	C7—C6—C5	119.74 (19)
C11—N2—N2^i	119.00 (16)	C7—C6—H6	120.1
C11—N2—H11	126.3	C5—C6—H6	120.1
N2^i—N2—H11	114.7	C6—C7—C8	118.9 (2)
C1—O1—C10	116.58 (13)	C6—C7—H7	120.5
H12—O3—H13	104.3	C8—C7—H7	120.5
C2—C1—O1	125.01 (16)	N1—C8—C7	124.4 (2)
C2—C1—C9	119.59 (16)	N1—C8—H8	117.8
O1—C1—C9	115.40 (15)	C7—C8—H8	117.8
C1—C2—C3	120.95 (18)	N1—C9—C5	122.26 (16)
C1—C2—H2	119.5	N1—C9—C1	119.16 (15)
C3—C2—H2	119.5	C5—C9—C1	118.58 (16)
C4—C3—C2	121.31 (19)	O1—C10—C11	111.84 (14)
C4—C3—H3	119.3	O1—C10—H10A	109.2
C2—C3—H3	119.3	C11—C10—H10A	109.2
C3—C4—C5	119.64 (18)	O1—C10—H10B	109.2

C3—C4—H4	120.2	C11—C10—H10B	109.2
C5—C4—H4	120.2	H10A—C10—H10B	107.9
C6—C5—C4	122.68 (18)	O2—C11—N2	124.29 (17)
C6—C5—C9	117.40 (18)	O2—C11—C10	118.66 (17)
C4—C5—C9	119.92 (17)	N2—C11—C10	117.04 (16)
C10—O1—C1—C2	−3.3 (3)	C8—N1—C9—C1	178.83 (17)
C10—O1—C1—C9	176.44 (15)	C6—C5—C9—N1	0.7 (3)
O1—C1—C2—C3	178.99 (18)	C4—C5—C9—N1	179.78 (18)
C9—C1—C2—C3	−0.8 (3)	C6—C5—C9—C1	−179.47 (18)
C1—C2—C3—C4	0.2 (3)	C4—C5—C9—C1	−0.4 (3)
C2—C3—C4—C5	0.2 (3)	C2—C1—C9—N1	−179.35 (17)
C3—C4—C5—C6	178.9 (2)	O1—C1—C9—N1	0.9 (2)
C3—C4—C5—C9	−0.1 (3)	C2—C1—C9—C5	0.9 (3)
C4—C5—C6—C7	−178.7 (2)	O1—C1—C9—C5	−178.91 (16)
C9—C5—C6—C7	0.3 (3)	C1—O1—C10—C11	−176.56 (15)
C5—C6—C7—C8	−0.7 (3)	N2^{i}—N2—C11—O2	0.4 (3)
C9—N1—C8—C7	1.0 (3)	N2^{i}—N2—C11—C10	179.43 (14)
C6—C7—C8—N1	0.0 (3)	O1—C10—C11—O2	−178.11 (18)
C8—N1—C9—C5	−1.4 (3)	O1—C10—C11—N2	2.8 (2)

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

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O3—H13⋯O2^{ii}	0.85	2.13	2.9745 (19)	175
O3—H12⋯N1	0.85	1.99	2.840 (1)	176
N2—H11⋯O3	0.87	1.97	2.815 (2)	164

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

Fig. 1

Fig. 2