Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-*N*′-(3,5-Dibromo-2-hydroxybenzylidene)-4-hydroxybenzohydrazide monohydrate

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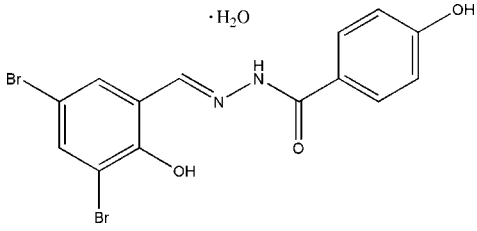
Received 3 September 2008; accepted 21 September 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.046; wR factor = 0.106; data-to-parameter ratio = 16.0.

The title compound, $C_{14}H_{10}Br_2N_2O_3\cdot H_2O$, was synthesized by the reaction of 3,5-dibromo-2-hydroxybenzaldehyde with an equimolar amount of 4-hydroxybenzohydrazide in methanol. The structure comprises a Schiff base unit and a water molecule of crystallization. The dihedral angle between the benzene rings in the Schiff base is 1.3 (3)°. In the crystal structure, molecules are linked through intermolecular O—H···O and N—H···O hydrogen bonds, with the water molecule serving as both donor and acceptor. As a result, layers are formed, which are approximately parallel to the *bc* plane.

Related literature

For related structures, see: Cao (2007*a*,*b*); Yang *et al.* (2008).



Experimental

Crystal data

$C_{14}H_{10}Br_2N_2O_3\cdot H_2O$
M_r = 432.08
Monoclinic, $P2_1/c$
a = 6.9840 (16) Å
b = 12.678 (3) Å
c = 17.722 (4) Å
β = 96.999 (4)°
V = 1557.4 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.22 mm^{-1}
T = 298 (2) K
0.23 × 0.23 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.307, T_{max} = 0.318
12695 measured reflections
3366 independent reflections
2045 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.106
S = 0.99
3366 reflections
210 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.86	2.578 (4)	146
O3—H3···O2^{i}	0.82	1.83	2.642 (4)	173
O4—H4A···O3^{ii}	0.847 (10)	2.038 (14)	2.878 (4)	171 (5)
O4—H4B···O1^{i}	0.851 (10)	2.24 (3)	2.969 (5)	144 (4)
N2—H2···O4^{iii}	0.898 (10)	2.01 (2)	2.874 (5)	162 (5)

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) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The Natural Scientific Research Foundation of the Education Office of Shanxi Province (Project No. 07JK177) is acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2193).

References

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supporting information

Acta Cryst. (2008). E**64**, o2022 [doi:10.1107/S1600536808030304]

(*E*)-*N*′-(3,5-Dibromo-2-hydroxybenzylidene)-4-hydroxybenzohydrazide monohydrate

Xiao-Ya Wang, Guo-Biao Cao and Tao Yang

S1. Comment

We have recently reported some transition metal complexes with Schiff base ligands (Cao, 2007*a*; Cao, 2007*b*). We report herein the crystal structure of the title compound, (I), derived from the reaction of 3,5-dibromo-2-hydroxybenzaldehyde with an equimolar quantity of 4-hydroxybenzohydrazide in methanol.

The compound (I), Fig. 1, comprises a Schiff base unit and a water molecule of crystallization. The dihedral angle between the two benzene rings in the Schiff base unit is 1.3 (3)°. All bond lengths are comparable to the similar compound, 3-bromo-*N*′-[(*E*)-4-hydroxybenzylidene]benzohydrazide, which we reported previously (Yang *et al.*, 2008). In the crystal structure, molecules are linked through intermolecular hydrogen bonds of types O—H···O and N—H···O (Table 1), forming 2D layers approximately parallel to the *bc* plane, as shown in Fig. 2.

S2. Experimental

The compound was prepared by refluxing equimolar quantities of 3,5-dibromo-2-hydroxybenzaldehyde with 4-hydroxybenzohydrazide in methanol. Colorless block crystals were formed when the solution was evaporated in air over five days.

S3. Refinement

Water H atoms and H2 were located in a difference map and refined isotropically, with O—H, N—H, and H···H distances restrained to 0.85 (1), 0.90 (1), and 1.37 (2) Å, respectively. The other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 Å and O—H distance of 0.82 Å, and with U_{iso}(H) set at 1.2U_{eq}(C) and 1.5U_{eq}(O).

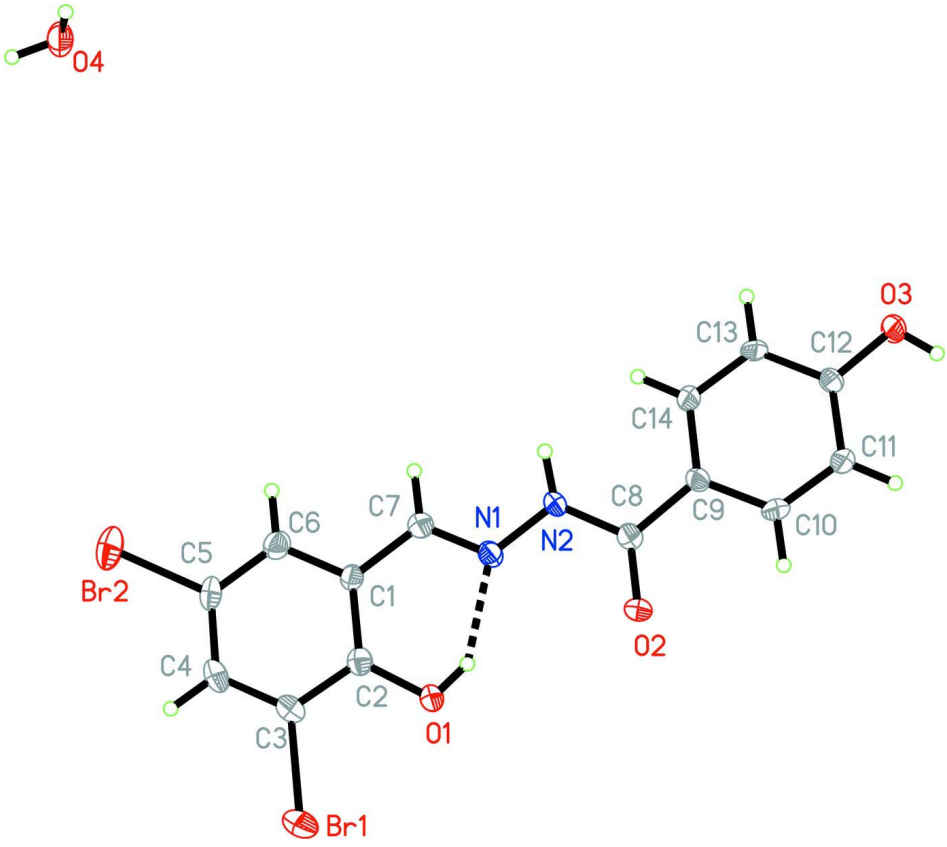

Figure 1

The molecular structure of (I) with ellipsoids drawn at the 30% probability level. Water and main molecule are placed in two different asymmetric units.

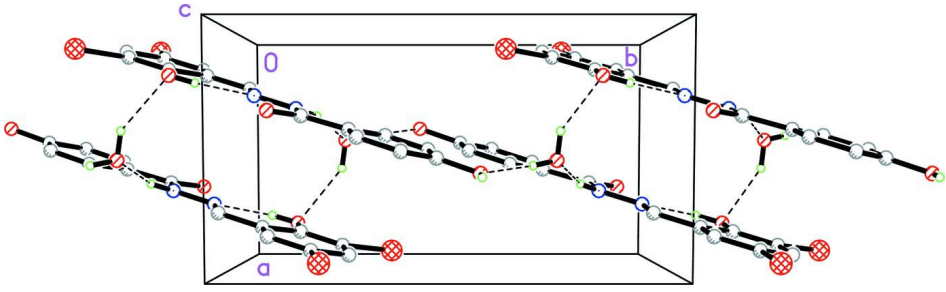

Figure 2

The molecular packing of (I), viewed along the *c* axis. Hydrogen bonds are drawn as dashed lines.

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c = 17.722 (4) Å
β = 96.999 (4)°
V = 1557.4 (6) Å^3
Z = 4
F(000) = 848
D_x = 1.843 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1545 reflections

θ = 2.3–24.9°
μ = 5.22 mm^{-1}
T = 298 K

Block, colourless
0.23 × 0.23 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.308, T_{max} = 0.318

12695 measured reflections
3366 independent reflections
2045 reflections with $I > 2\sigma(I)$
R_{int} = 0.075
θ_{max} = 27.0°, θ_{min} = 2.0°
h = −8→8
k = −16→15
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.106
S = 1.00
3366 reflections
210 parameters
4 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.07183 (8)	0.86697 (4)	−0.00493 (3)	0.0601 (2)
Br2	0.05286 (8)	0.71867 (5)	−0.30593 (3)	0.0664 (2)
O1	0.1871 (5)	0.6446 (2)	0.03203 (16)	0.0405 (7)
H1	0.2190	0.5834	0.0420	0.061*
O2	0.3386 (4)	0.4075 (2)	0.14777 (16)	0.0426 (8)
O3	0.5946 (6)	−0.0702 (2)	0.20309 (16)	0.0594 (10)
H3	0.6086	−0.0733	0.2497	0.089*
O4	0.4571 (5)	0.2539 (3)	0.39995 (18)	0.0494 (8)
N1	0.2698 (5)	0.4520 (3)	0.00247 (19)	0.0323 (8)
N2	0.3257 (5)	0.3529 (3)	0.02714 (19)	0.0336 (8)
C1	0.1784 (6)	0.5746 (3)	−0.0955 (2)	0.0301 (10)
C2	0.1564 (6)	0.6572 (3)	−0.0440 (2)	0.0336 (10)
C3	0.1013 (6)	0.7560 (3)	−0.0737 (3)	0.0388 (11)
C4	0.0691 (6)	0.7749 (4)	−0.1503 (3)	0.0422 (11)
H4	0.0323	0.8414	−0.1687	0.051*
C5	0.0927 (6)	0.6928 (4)	−0.1997 (2)	0.0385 (11)
C6	0.1470 (6)	0.5942 (4)	−0.1731 (2)	0.0398 (11)
H6	0.1628	0.5401	−0.2073	0.048*
C7	0.2368 (6)	0.4704 (3)	−0.0687 (2)	0.0358 (10)
H7	0.2504	0.4166	−0.1033	0.043*
C8	0.3580 (6)	0.3355 (3)	0.1033 (2)	0.0311 (10)
C9	0.4152 (6)	0.2264 (3)	0.1270 (2)	0.0292 (9)

C10	0.4475 (7)	0.2041 (3)	0.2037 (2)	0.0430 (12)
H10	0.4294	0.2573	0.2383	0.052*
C11	0.5056 (7)	0.1059 (3)	0.2311 (2)	0.0438 (12)
H11	0.5262	0.0933	0.2832	0.053*
C12	0.5325 (7)	0.0275 (3)	0.1807 (2)	0.0385 (11)
C13	0.4980 (7)	0.0477 (3)	0.1040 (2)	0.0528 (14)
H13	0.5146	−0.0059	0.0696	0.063*
C14	0.4397 (7)	0.1452 (3)	0.0773 (2)	0.0462 (12)
H14	0.4163	0.1569	0.0252	0.055*
H2	0.356 (7)	0.307 (3)	−0.008 (2)	0.080*
H4A	0.436 (6)	0.309 (3)	0.373 (3)	0.080*
H4B	0.5787 (19)	0.245 (4)	0.408 (3)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0799 (4)	0.0359 (3)	0.0643 (4)	0.0158 (3)	0.0076 (3)	−0.0051 (3)
Br2	0.0707 (4)	0.0883 (5)	0.0380 (3)	0.0029 (3)	−0.0022 (2)	0.0233 (3)
O1	0.061 (2)	0.0292 (17)	0.0304 (16)	0.0048 (16)	0.0005 (15)	0.0034 (13)
O2	0.066 (2)	0.0267 (16)	0.0348 (17)	0.0075 (15)	0.0035 (15)	−0.0053 (14)
O3	0.122 (3)	0.0215 (17)	0.0333 (18)	0.0134 (19)	0.003 (2)	0.0046 (14)
O4	0.070 (2)	0.041 (2)	0.0359 (18)	−0.0058 (18)	0.0031 (17)	0.0069 (15)
N1	0.035 (2)	0.026 (2)	0.034 (2)	0.0008 (16)	0.0007 (16)	0.0021 (16)
N2	0.046 (2)	0.023 (2)	0.031 (2)	0.0049 (17)	0.0024 (17)	0.0037 (15)
C1	0.032 (2)	0.032 (2)	0.026 (2)	0.0011 (19)	0.0028 (18)	0.0022 (19)
C2	0.032 (2)	0.035 (3)	0.033 (2)	0.001 (2)	0.0032 (19)	0.004 (2)
C3	0.039 (3)	0.030 (2)	0.047 (3)	0.004 (2)	0.004 (2)	0.001 (2)
C4	0.041 (3)	0.035 (3)	0.050 (3)	0.006 (2)	0.001 (2)	0.016 (2)
C5	0.035 (2)	0.049 (3)	0.030 (2)	0.000 (2)	0.0011 (19)	0.015 (2)
C6	0.042 (3)	0.045 (3)	0.032 (2)	0.004 (2)	0.001 (2)	0.000 (2)
C7	0.043 (3)	0.032 (3)	0.032 (2)	0.003 (2)	0.002 (2)	−0.0007 (19)
C8	0.029 (2)	0.029 (2)	0.034 (2)	−0.0019 (19)	0.0004 (19)	0.000 (2)
C9	0.034 (2)	0.023 (2)	0.030 (2)	−0.0003 (19)	0.0010 (18)	0.0003 (18)
C10	0.067 (3)	0.033 (3)	0.030 (2)	0.007 (2)	0.006 (2)	−0.010 (2)
C11	0.073 (3)	0.032 (3)	0.026 (2)	0.009 (2)	0.006 (2)	0.001 (2)
C12	0.064 (3)	0.019 (2)	0.033 (3)	0.003 (2)	0.009 (2)	0.0036 (19)
C13	0.104 (4)	0.025 (3)	0.029 (3)	0.014 (3)	0.006 (3)	−0.008 (2)
C14	0.081 (4)	0.030 (3)	0.026 (2)	0.009 (3)	0.001 (2)	−0.001 (2)

Geometric parameters (Å, º)

Br1—C3	1.888 (4)	C3—C4	1.371 (6)
Br2—C5	1.897 (4)	C4—C5	1.382 (6)
O1—C2	1.348 (5)	C4—H4	0.9300
O1—H1	0.8200	C5—C6	1.374 (6)
O2—C8	1.225 (5)	C6—H6	0.9300
O3—C12	1.355 (5)	C7—H7	0.9300
O3—H3	0.8200	C8—C9	1.486 (5)

O4—H4A	0.847 (10)	C9—C14	1.379 (5)
O4—H4B	0.851 (10)	C9—C10	1.379 (6)
N1—C7	1.275 (5)	C10—C11	1.380 (6)
N1—N2	1.371 (4)	C10—H10	0.9300
N2—C8	1.359 (5)	C11—C12	1.365 (6)
N2—H2	0.898 (10)	C11—H11	0.9300
C1—C6	1.387 (5)	C12—C13	1.375 (6)
C1—C2	1.411 (6)	C13—C14	1.368 (6)
C1—C7	1.446 (6)	C13—H13	0.9300
C2—C3	1.395 (6)	C14—H14	0.9300
C2—O1—H1	109.5	N1—C7—C1	120.2 (4)
C12—O3—H3	109.5	N1—C7—H7	119.9
H4A—O4—H4B	108 (2)	C1—C7—H7	119.9
C7—N1—N2	119.5 (4)	O2—C8—N2	120.0 (4)
C8—N2—N1	118.2 (3)	O2—C8—C9	123.9 (4)
C8—N2—H2	124 (4)	N2—C8—C9	116.1 (4)
N1—N2—H2	117 (4)	C14—C9—C10	117.3 (4)
C6—C1—C2	119.4 (4)	C14—C9—C8	124.3 (4)
C6—C1—C7	119.6 (4)	C10—C9—C8	118.4 (4)
C2—C1—C7	120.9 (4)	C9—C10—C11	122.5 (4)
O1—C2—C3	119.1 (4)	C9—C10—H10	118.7
O1—C2—C1	122.8 (4)	C11—C10—H10	118.7
C3—C2—C1	118.1 (4)	C12—C11—C10	119.0 (4)
C4—C3—C2	122.3 (4)	C12—C11—H11	120.5
C4—C3—Br1	119.5 (3)	C10—C11—H11	120.5
C2—C3—Br1	118.2 (3)	O3—C12—C11	122.6 (4)
C3—C4—C5	118.6 (4)	O3—C12—C13	118.1 (4)
C3—C4—H4	120.7	C11—C12—C13	119.3 (4)
C5—C4—H4	120.7	C14—C13—C12	121.3 (4)
C6—C5—C4	121.1 (4)	C14—C13—H13	119.4
C6—C5—Br2	120.0 (4)	C12—C13—H13	119.4
C4—C5—Br2	118.9 (3)	C13—C14—C9	120.6 (4)
C5—C6—C1	120.5 (4)	C13—C14—H14	119.7
C5—C6—H6	119.7	C9—C14—H14	119.7
C1—C6—H6	119.7		

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