Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-*N*′-(5-Bromo-2-hydroxybenzylidene)-3,5-dihydroxybenzohydrazide monohydrate

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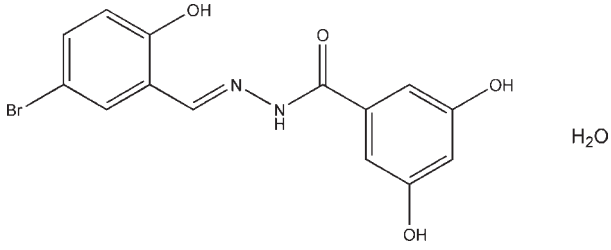
Received 6 July 2010; accepted 13 July 2010

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.077; data-to-parameter ratio = 11.9.

The Schiff base molecule in the title compound, $C_{14}H_{11}BrN_2O_4\cdot H_2O$, is almost planar with an r.m.s. deviation for the non-H atoms of 0.16 Å. In the crystal structure, the Schiff base molecules and the water molecules are linked together by intermolecular N—H···O and O—H···O hydrogen bonds, leading to layers parallel to the *bc* plane. An intramolecular O—H···N hydrogen bond involving the imine N atom and a hydroxy substituent is also observed.

Related literature

For the isotypic Cl analogue $C_{14}H_{11}ClN_2O_4\cdot H_2O$, see: Deng *et al.* (2009).



Experimental

Crystal data

$C_{14}H_{11}BrN_2O_4\cdot H_2O$
M_r = 369.17
Monoclinic, $P2_1/c$
a = 13.5685 (3) Å
b = 8.0532 (2) Å
c = 13.2447 (2) Å
β = 100.186 (1)°
V = 1424.44 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.91 mm^{-1}
T = 296 K
0.58 × 0.33 × 0.06 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.283, T_{max} = 0.845
9148 measured reflections
2579 independent reflections
2183 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.077
S = 1.04
2579 reflections
217 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.81 (2)	1.95 (2)	2.657 (2)	145 (3)
N2—H2*N*···O2	0.85 (2)	2.07 (2)	2.913 (3)	170 (2)
O11—H11···O8^{i}	0.83 (2)	1.94 (2)	2.750 (2)	168 (3)
O13—H13···O1^{ii}	0.79 (2)	2.19 (2)	2.959 (2)	165 (3)
O2—H2*A*···O8^{i}	0.81 (2)	1.98 (2)	2.776 (3)	171 (4)
O2—H2*B*···O11^{iii}	0.83 (2)	2.06 (2)	2.861 (3)	165 (3)

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$.

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: *SHELXL97* and *publCIF* (Westrip, 2010).

The authors thank the University of Malaya for funding this study (FRGS grant No. FP009/2008 C)

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2299).

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supporting information

Acta Cryst. (2010). E**66**, o2045 [https://doi.org/10.1107/S1600536810027856]

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S1. Comment

The molecular structure of the title compound is shown in Fig. 1, and the crystal structure in Fig. 2. The present study shows that Br and Cl analogues (Deng *et al.*, 2009) are isotypic crystals.

S2. Experimental

An ethanolic solution (15 ml) of 3,5-dihydroxybenzohydrazide (0.67 g, 4 mmol) and 5-bromosalicylaldehyde (0.8 g, 4 mmol) was refluxed for 2 h. The solution was then cooled and the solid product formed was filtered off, washed with cold ethanol, and dried over silica gel. Crystals of the title compound were obtained by slow evaporation of a DMSO solution at room temperature.

S3. Refinement

The carbon-bound H atoms were placed in calculated positions (C—H fixed to 0.93 Å) and treated as riding on their parent carbon atoms with U_{iso}(H) set to 1.2 U_{eq}(carrier C). The nitrogen- and oxygen-bound H atoms were located in a difference map and refined as free atoms, with N—H and O—H distances restrained to 0.86 (2) and 0.82 (2) Å, respectively.

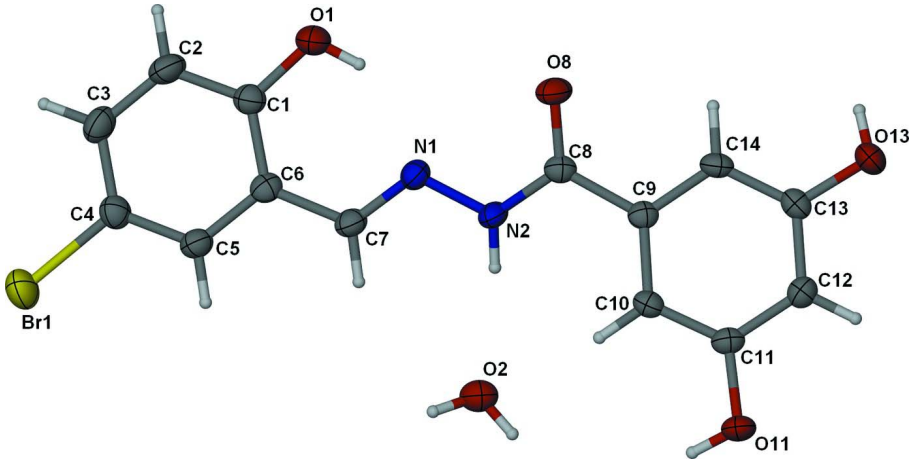

Figure 1

Thermal ellipsoid plot of the title compound at 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

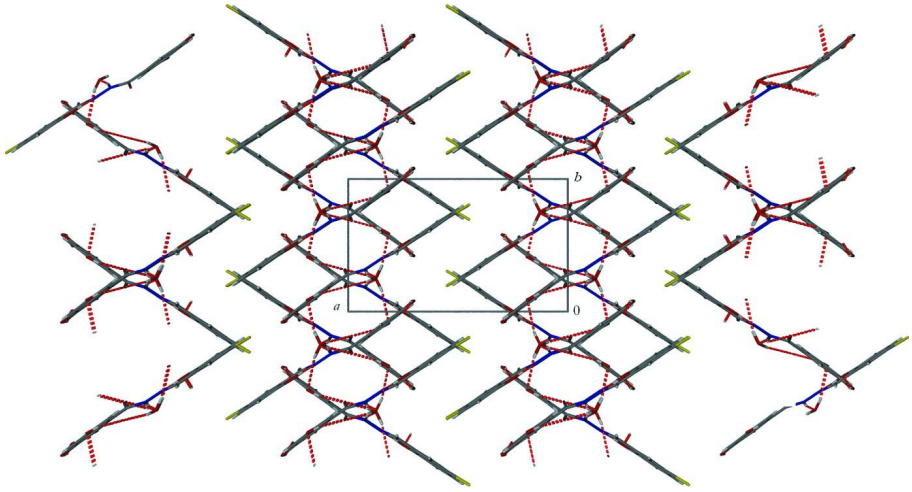

Figure 2

Packing view looking down the crystallographic *b* unit cell edge.

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Crystal data

$C_{14}H_{11}BrN_2O_4 \cdot H_2O$
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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.5685\ (3)$ Å
$b = 8.0532\ (2)$ Å
$c = 13.2447\ (2)$ Å
$\beta = 100.186\ (1)°$
$V = 1424.44\ (5)$ Å^3
$Z = 4$
$F(000) = 744$
$D_x = 1.721$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3109 reflections
$\theta = 3.0–26.1°$
$\mu = 2.91$ mm^{-1}
$T = 296$ K
Plate, yellow
0.58 × 0.33 × 0.06 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.283$, $T_{max} = 0.845$
9148 measured reflections
2579 independent reflections
2183 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.3°$, $\theta_{min} = 3.0°$
$h = -16\rightarrow16$
$k = -9\rightarrow9$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.077$
$S = 1.04$
2579 reflections
217 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2 + 0.7124P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.54976 (2)	0.19522 (4)	0.67677 (2)	0.05405 (13)
O1	0.25489 (15)	0.4914 (3)	0.33122 (13)	0.0448 (5)
H1	0.210 (2)	0.542 (4)	0.351 (2)	0.067*
O8	−0.01522 (14)	0.6853 (2)	0.34596 (12)	0.0375 (4)
O11	−0.18429 (14)	0.9113 (2)	0.71231 (12)	0.0378 (4)
H11	−0.1355 (18)	0.868 (4)	0.749 (2)	0.057*
O13	−0.29944 (14)	1.0800 (2)	0.37286 (13)	0.0440 (5)
H13	−0.285 (3)	1.075 (4)	0.3181 (17)	0.066*
N1	0.15274 (14)	0.5993 (2)	0.47190 (14)	0.0279 (4)
N2	0.07629 (15)	0.6835 (2)	0.50575 (14)	0.0277 (4)
H2N	0.088 (2)	0.712 (3)	0.5686 (14)	0.033*
C1	0.31939 (19)	0.4270 (3)	0.41219 (17)	0.0312 (5)
C2	0.3994 (2)	0.3354 (3)	0.39071 (19)	0.0402 (6)
H2	0.4072	0.3208	0.3229	0.048*
C3	0.4676 (2)	0.2657 (3)	0.4683 (2)	0.0391 (6)
H3	0.5211	0.2037	0.4534	0.047*
C4	0.45524 (18)	0.2895 (3)	0.56914 (18)	0.0330 (6)
C5	0.37678 (18)	0.3807 (3)	0.59223 (17)	0.0319 (5)
H5	0.3701	0.3951	0.6603	0.038*
C6	0.30662 (17)	0.4524 (3)	0.51387 (16)	0.0278 (5)
C7	0.22369 (18)	0.5450 (3)	0.54062 (17)	0.0300 (5)
H7	0.2219	0.5656	0.6094	0.036*
C8	−0.00598 (18)	0.7237 (3)	0.43815 (16)	0.0266 (5)
C9	−0.08615 (17)	0.8158 (3)	0.47789 (16)	0.0248 (5)
C10	−0.09485 (18)	0.8153 (3)	0.58150 (16)	0.0274 (5)
H10	−0.0499	0.7559	0.6293	0.033*
C11	−0.17126 (17)	0.9044 (3)	0.61131 (16)	0.0276 (5)
C12	−0.23974 (18)	0.9932 (3)	0.54147 (17)	0.0303 (5)
H12	−0.2908	1.0530	0.5632	0.036*
C13	−0.23079 (18)	0.9912 (3)	0.43882 (16)	0.0291 (5)
C14	−0.15405 (17)	0.9038 (3)	0.40695 (16)	0.0283 (5)
H14	−0.1479	0.9040	0.3381	0.034*
O2	0.12746 (16)	0.7432 (3)	0.72574 (13)	0.0428 (5)
H2A	0.091 (2)	0.772 (4)	0.764 (2)	0.064*
H2B	0.153 (3)	0.656 (3)	0.750 (2)	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03931 (19)	0.0753 (2)	0.04679 (19)	0.02026 (15)	0.00548 (13)	0.01280 (14)
O1	0.0449 (12)	0.0626 (13)	0.0261 (9)	0.0149 (10)	0.0044 (8)	−0.0005 (8)
O8	0.0353 (10)	0.0545 (11)	0.0224 (8)	0.0033 (9)	0.0047 (7)	−0.0065 (7)
O11	0.0417 (11)	0.0525 (11)	0.0209 (8)	0.0101 (9)	0.0098 (7)	0.0018 (7)
O13	0.0447 (11)	0.0571 (12)	0.0291 (9)	0.0212 (10)	0.0033 (8)	0.0079 (8)
N1	0.0269 (11)	0.0292 (10)	0.0281 (10)	0.0012 (9)	0.0061 (8)	−0.0039 (8)

N2	0.0273 (11)	0.0341 (11)	0.0221 (9)	0.0035 (9)	0.0048 (8)	−0.0063 (8)
C1	0.0308 (13)	0.0351 (13)	0.0275 (12)	0.0001 (11)	0.0047 (10)	0.0000 (10)
C2	0.0397 (16)	0.0533 (17)	0.0302 (13)	0.0037 (13)	0.0133 (11)	−0.0057 (11)
C3	0.0316 (15)	0.0451 (15)	0.0431 (15)	0.0054 (12)	0.0135 (11)	−0.0041 (11)
C4	0.0266 (14)	0.0382 (14)	0.0338 (13)	0.0010 (11)	0.0039 (10)	0.0021 (10)
C5	0.0316 (14)	0.0382 (13)	0.0271 (12)	0.0016 (11)	0.0082 (10)	−0.0020 (10)
C6	0.0268 (13)	0.0294 (12)	0.0276 (11)	−0.0035 (10)	0.0058 (9)	−0.0041 (9)
C7	0.0306 (13)	0.0348 (13)	0.0251 (11)	0.0009 (11)	0.0066 (10)	−0.0049 (9)
C8	0.0278 (13)	0.0289 (12)	0.0233 (12)	−0.0034 (10)	0.0053 (9)	−0.0001 (9)
C9	0.0243 (12)	0.0269 (11)	0.0233 (11)	−0.0032 (9)	0.0041 (9)	−0.0015 (9)
C10	0.0290 (13)	0.0307 (12)	0.0216 (11)	0.0007 (10)	0.0015 (9)	0.0012 (9)
C11	0.0301 (13)	0.0315 (12)	0.0222 (11)	−0.0034 (10)	0.0072 (9)	−0.0009 (9)
C12	0.0290 (13)	0.0327 (13)	0.0304 (12)	0.0029 (10)	0.0080 (10)	−0.0006 (9)
C13	0.0289 (13)	0.0299 (12)	0.0271 (11)	0.0012 (10)	0.0008 (10)	0.0032 (9)
C14	0.0322 (14)	0.0340 (13)	0.0182 (10)	−0.0018 (11)	0.0034 (9)	−0.0002 (9)
O2	0.0497 (13)	0.0487 (11)	0.0308 (10)	0.0051 (10)	0.0093 (8)	0.0000 (8)

Geometric parameters (Å, º)

Br1—C4	1.899 (2)	C4—C5	1.372 (3)
O1—C1	1.361 (3)	C5—C6	1.402 (3)
O1—H1	0.812 (18)	C5—H5	0.9300
O8—C8	1.244 (3)	C6—C7	1.445 (3)
O11—C11	1.381 (3)	C7—H7	0.9300
O11—H11	0.827 (18)	C8—C9	1.487 (3)
O13—C13	1.361 (3)	C9—C14	1.389 (3)
O13—H13	0.785 (18)	C9—C10	1.398 (3)
N1—C7	1.279 (3)	C10—C11	1.375 (3)
N1—N2	1.379 (3)	C10—H10	0.9300
N2—C8	1.341 (3)	C11—C12	1.388 (3)
N2—H2N	0.851 (17)	C12—C13	1.386 (3)
C1—C2	1.383 (4)	C12—H12	0.9300
C1—C6	1.403 (3)	C13—C14	1.383 (3)
C2—C3	1.375 (4)	C14—H14	0.9300
C2—H2	0.9300	O2—H2A	0.807 (18)
C3—C4	1.389 (3)	O2—H2B	0.825 (18)
C3—H3	0.9300		

C1—O1—H1	110 (2)	N1—C7—C6	121.5 (2)
C11—O11—H11	109 (2)	N1—C7—H7	119.2
C13—O13—H13	108 (3)	C6—C7—H7	119.2
C7—N1—N2	116.85 (18)	O8—C8—N2	121.5 (2)
C8—N2—N1	119.13 (18)	O8—C8—C9	121.2 (2)
C8—N2—H2N	125.0 (18)	N2—C8—C9	117.29 (18)
N1—N2—H2N	115.7 (18)	C14—C9—C10	120.3 (2)
O1—C1—C2	117.4 (2)	C14—C9—C8	117.02 (19)
O1—C1—C6	122.0 (2)	C10—C9—C8	122.7 (2)
C2—C1—C6	120.6 (2)	C11—C10—C9	118.6 (2)

C3—C2—C1	120.9 (2)	C11—C10—H10	120.7
C3—C2—H2	119.6	C9—C10—H10	120.7
C1—C2—H2	119.6	C10—C11—O11	122.1 (2)
C2—C3—C4	118.8 (2)	C10—C11—C12	121.8 (2)
C2—C3—H3	120.6	O11—C11—C12	116.1 (2)
C4—C3—H3	120.6	C13—C12—C11	118.9 (2)
C5—C4—C3	121.3 (2)	C13—C12—H12	120.5
C5—C4—Br1	119.64 (18)	C11—C12—H12	120.5
C3—C4—Br1	119.10 (19)	O13—C13—C14	122.5 (2)
C4—C5—C6	120.5 (2)	O13—C13—C12	117.0 (2)
C4—C5—H5	119.8	C14—C13—C12	120.4 (2)
C6—C5—H5	119.8	C13—C14—C9	119.91 (19)
C5—C6—C1	117.9 (2)	C13—C14—H14	120.0
C5—C6—C7	119.1 (2)	C9—C14—H14	120.0
C1—C6—C7	123.0 (2)	H2A—O2—H2B	105 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.81 (2)	1.95 (2)	2.657 (2)	145 (3)
N2—H2*N*···O2	0.85 (2)	2.07 (2)	2.913 (3)	170 (2)
O1—H1···O2^{i}	0.81 (2)	2.52 (3)	2.942 (3)	114 (3)
O11—H11···O8^{ii}	0.83 (2)	1.94 (2)	2.750 (2)	168 (3)
O13—H13···O1^{iii}	0.79 (2)	2.19 (2)	2.959 (2)	165 (3)
O2—H2*A*···O8^{ii}	0.81 (2)	1.98 (2)	2.776 (3)	171 (4)
O2—H2*B*···O11^{iv}	0.83 (2)	2.06 (2)	2.861 (3)	165 (3)

Symmetry codes: (i) x, $-y+3/2$, $z-1/2$; (ii) x, $-y+3/2$, $z+1/2$; (iii) $-x$, $y+1/2$, $-z+1/2$; (iv) $-x$, $y-1/2$, $-z+3/2$.