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## Structure Reports

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# (E)-N'-(5-Bromo-2-hydroxybenzylidene)-3,5-dihydroxybenzohydrazide monohydrate

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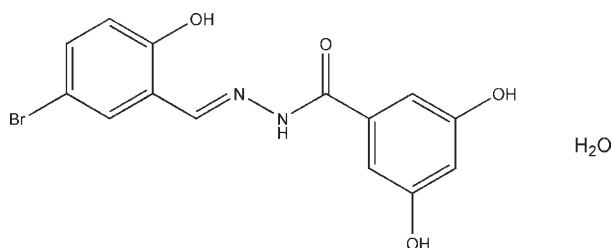
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 Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  
 $R$  factor = 0.031;  $wR$  factor = 0.077; data-to-parameter ratio = 11.9.

The Schiff base molecule in the title compound,  $\text{C}_{14}\text{H}_{11}\text{BrN}_2\text{O}_4 \cdot \text{H}_2\text{O}$ , 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  $\text{N}-\text{H} \cdots \text{O}$  and  $\text{O}-\text{H} \cdots \text{O}$  hydrogen bonds, leading to layers parallel to the  $bc$  plane. An intramolecular  $\text{O}-\text{H} \cdots \text{N}$  hydrogen bond involving the imine N atom and a hydroxy substituent is also observed.

## Related literature

 For the isotopic Cl analogue  $\text{C}_{14}\text{H}_{11}\text{ClN}_2\text{O}_4 \cdot \text{H}_2\text{O}$ , see: Deng *et al.* (2009).


## Experimental

## Crystal data

 $\text{C}_{14}\text{H}_{11}\text{BrN}_2\text{O}_4 \cdot \text{H}_2\text{O}$   
 $M_r = 369.17$ 

 Monoclinic,  $P2_1/c$   
 $a = 13.5685$  (3) Å

 $b = 8.0532$  (2) Å  
 $c = 13.2447$  (2) Å  
 $\beta = 100.186$  (1)°  
 $V = 1424.44$  (5) Å<sup>3</sup>  
 $Z = 4$ 

 Mo  $K\alpha$  radiation  
 $\mu = 2.91$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.58 \times 0.33 \times 0.06$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$ 

## 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  
 $\Delta\rho_{\text{max}} = 0.45$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.56$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O1}-\text{H1} \cdots \text{N1}$	0.81 (2)	1.95 (2)	2.657 (2)	145 (3)
$\text{N2}-\text{H2N} \cdots \text{O2}$	0.85 (2)	2.07 (2)	2.913 (3)	170 (2)
$\text{O11}-\text{H11} \cdots \text{O8}^i$	0.83 (2)	1.94 (2)	2.750 (2)	168 (3)
$\text{O13}-\text{H13} \cdots \text{O1}^{ii}$	0.79 (2)	2.19 (2)	2.959 (2)	165 (3)
$\text{O2}-\text{H2A} \cdots \text{O8}^i$	0.81 (2)	1.98 (2)	2.776 (3)	171 (4)
$\text{O2}-\text{H2B} \cdots \text{O11}^{iii}$	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).

## References

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**supplementary materials**

*Acta Cryst.* (2010). E66, o2045 [ doi:10.1107/S1600536810027856 ]

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

**S. M. Saharin, H. Khaledi and H. Mohd Ali**

**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.

**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.

**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  $U_{\text{iso}}(\text{H})$  set to 1.2  $U_{\text{eq}}(\text{carrier C})$ . 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.

**Figures**

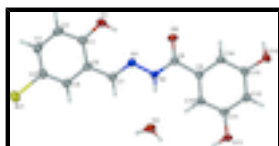


Fig. 1. Thermal ellipsoid plot of the title compound at 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

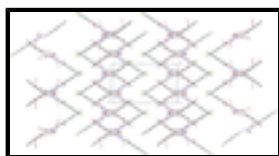


Fig. 2. Packing view looking down the crystallographic *b* unit cell edge.

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

*Crystal data*

$\text{C}_{14}\text{H}_{11}\text{BrN}_2\text{O}_4\cdot\text{H}_2\text{O}$

$M_r = 369.17$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 13.5685(3) \text{ \AA}$

$F(000) = 744$

$D_x = 1.721 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3109 reflections

$\theta = 3.0\text{--}26.1^\circ$

# supplementary materials

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$b = 8.0532 (2) \text{ \AA}$   
 $c = 13.2447 (2) \text{ \AA}$   
 $\beta = 100.186 (1)^\circ$   
 $V = 1424.44 (5) \text{ \AA}^3$   
 $Z = 4$

$\mu = 2.91 \text{ mm}^{-1}$   
 $T = 296 \text{ K}$   
Plate, yellow  
 $0.58 \times 0.33 \times 0.06 \text{ mm}$

## Data collection

Bruker APEXII CCD diffractometer  
Radiation source: fine-focus sealed tube graphite  
 $\varphi$  and  $\omega$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$   
 $\theta_{\max} = 25.3^\circ$ ,  $\theta_{\min} = 3.0^\circ$   
 $h = -16 \rightarrow 16$   
 $k = -9 \rightarrow 9$   
 $l = -15 \rightarrow 15$

## 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  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.45 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.56 \text{ e \AA}^{-3}$

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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)

## supplementary materials

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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, °)

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, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1···N1	0.81 (2)	1.95 (2)	2.657 (2)	145 (3)
N2—H2N···O2	0.85 (2)	2.07 (2)	2.913 (3)	170 (2)
O1—H1···O2 <sup>i</sup>	0.81 (2)	2.52 (3)	2.942 (3)	114 (3)
O11—H11···O8 <sup>ii</sup>	0.83 (2)	1.94 (2)	2.750 (2)	168 (3)
O13—H13···O1 <sup>iii</sup>	0.79 (2)	2.19 (2)	2.959 (2)	165 (3)
O2—H2A···O8 <sup>ii</sup>	0.81 (2)	1.98 (2)	2.776 (3)	171 (4)
O2—H2B···O11 <sup>iv</sup>	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)  $-x, y-1/2, -z+3/2$ .

Fig. 1

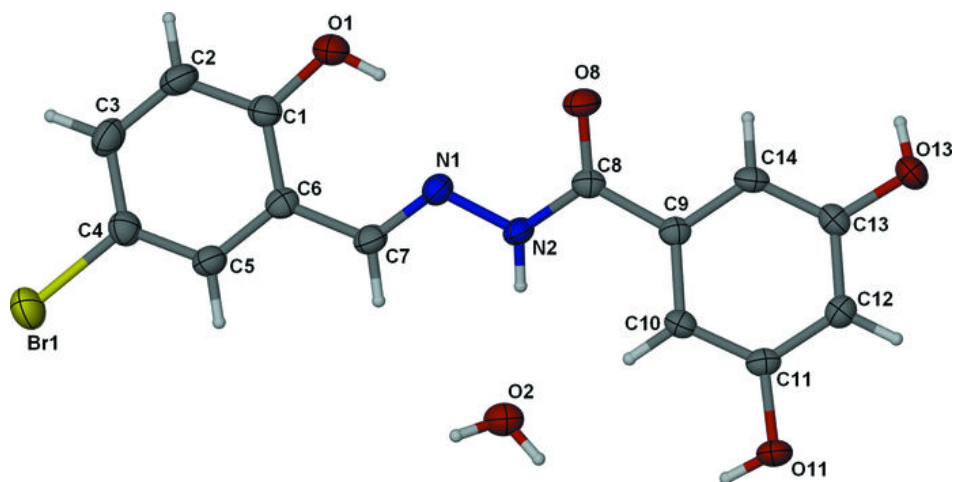




Fig. 2

