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N′-(5-Bromo-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide dihydrate

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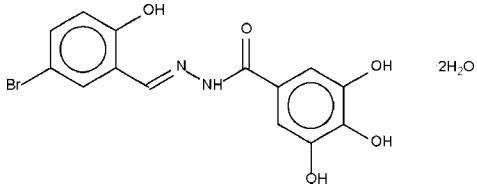
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.065; wR factor = 0.155; data-to-parameter ratio = 14.2.

The title compound, $C_{14}H_{11}BrN_2O_5\cdot 2H_2O$, crystallizes as hydrogen-bonded sheets. The 2-hydroxy group on the benzylidene group forms an intramolecular hydrogen bond to the N atom of the C=N double bond. The amino N atom is a hydrogen-bond donor to a water molecule. The hydroxy group on the benzohydrazide group is a hydrogen-bond donor to one acceptor site, whereas each water molecule is a hydrogen-bond donor to two acceptor sites.

Related literature

For the structure of a similar Schiff-base ligand, 5-bromosalicylaldehyde benzoylhydrazone, see: Liu *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{11}BrN_2O_5\cdot 2H_2O$
M_r = 403.19
Monoclinic, $P2_1/c$
a = 30.8424 (8) Å
b = 3.7999 (1) Å
c = 12.8484 (4) Å
β = 90.280 (2)°
V = 1505.79 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.77 mm^{-1}
T = 100 (2) K
0.30 × 0.03 × 0.03 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.658, T_{max} = 0.921
9964 measured reflections
3424 independent reflections
2914 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.155
S = 1.22
3424 reflections
241 parameters
10 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.08 e Å^{-3}
$\Delta\rho_{min}$ = −1.82 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*o*···N1	0.84 (1)	1.91 (5)	2.616 (6)	141 (7)
O3—H3*o*···O2*w*	0.84 (1)	1.96 (4)	2.736 (6)	153 (7)
O4—H4*o*···O2*w*^{i}	0.84 (1)	1.81 (3)	2.623 (8)	163 (9)
O5—H5*o*···O2^{ii}	0.84 (1)	1.93 (2)	2.764 (5)	171 (7)
O1*w*—H1*w*1···O2^{iii}	0.84 (1)	1.98 (2)	2.812 (5)	170 (6)
O1*w*—H1*w*2···O1^{ii}	0.84 (1)	2.09 (2)	2.914 (6)	167 (6)
O2*w*—H2*w*1···O3^{iv}	0.84 (1)	2.13 (5)	2.845 (9)	142 (8)
O2*w*—H2*w*2···O4^{v}	0.84 (1)	2.12 (4)	2.900 (8)	154 (8)

Symmetry codes: (i) $-x, -y+2, -z+2$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $x, -y+\frac{5}{2}, z+\frac{1}{2}$; (iv) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$; (v) $-x, -y+3, -z+2$.

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: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2751).

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supplementary materials

Acta Cryst. (2008). E**64**, o1584 [**doi:10.1107/S1600536808022708**]

N'-(5-Bromo-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide dihydrate

A. A. Alhadi, H. M. Ali, S. Puvaneswary, W. T. Robinson and S. W. Ng

Comment

This study extends the structural study on the Schiff base, 5-bromosalicylaldehyde benzoylhydrazone (Liu *et al.*, 2006) as the title compound (Scheme I, Fig. 1) has several hydroxy groups on one of the aromatic rings. The compound crystallizes with two lattice water molecules. Hydrogen bonding interactions (Table 1) give rise to a layer motif.

Experimental

3,4,5-Trihydroxybenzoylhydrazide (0.65 g, 3.5 mmol) and 5-bromo-2-hydroxybenzaldehyde (0.70 g, 3.5 mmol) were heated for 12 h in ethanol. The solvent was removed and the product recrystallized from ethanol.

Refinement

Carbon and nitrogen-bound H-atoms were placed in calculated positions (C—H 0.95 Å; N–H 0.88 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) 1.2 U_{eq}(C). The hydroxy and water H-atoms were located in a difference Fourier map, and were refined with distance restraints of O–H 0.84±0.01 Å and H···H 1.37±0.01 Å.

The final difference Fourier map had a peak of 1.37eÅ^{-3} at 0.69Å from Br1 and a hole of -1.81eÅ^{-3} at 1.33Å from C2.

Figures

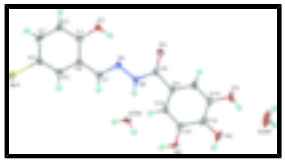


Fig. 1. View (Barbour, 2001) of *N*-(5-bromo-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide with displacement ellipsoids at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

N'-(5-Bromo-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide dihydrate

Crystal data

$C_{14}H_{11}BrN_2O_5 \cdot 2H_2O$	F_{000} = 816
M_r = 403.19	D_x = 1.779 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2758 reflections
a = 30.8424 (8) Å	θ = 3.2–27.4°
b = 3.7999 (1) Å	μ = 2.77 mm^{-1}
c = 12.8484 (4) Å	T = 100 (2) K

β = 90.280 (2)°	Needle, colorless
V = 1505.79 (7) Å^3	0.30 × 0.03 × 0.03 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	3424 independent reflections
Radiation source: fine-focus sealed tube	2914 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.039
T = 100(2) K	θ_{max} = 27.5°
ω scans	θ_{min} = 1.3°
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	h = −38→40
T_{min} = 0.658, T_{max} = 0.921	k = −4→4
9964 measured reflections	l = −15→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.064	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.155	$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2 + 10.2476P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.22	$(\Delta/\sigma)_{max}$ = 0.001
3424 reflections	$\Delta\rho_{max}$ = 1.08 e Å^{-3}
241 parameters	$\Delta\rho_{min}$ = −1.82 e Å^{-3}
10 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.467910 (16)	0.31017 (16)	0.89799 (4)	0.01787 (17)
O1	0.32358 (12)	0.6446 (12)	0.5944 (3)	0.0201 (9)
H1O	0.3020 (15)	0.745 (18)	0.621 (5)	0.030*
O2	0.20078 (11)	0.9755 (11)	0.6545 (3)	0.0182 (8)
O3	0.05472 (13)	1.3768 (13)	0.7962 (4)	0.0295 (11)

H3O	0.0335 (16)	1.36 (2)	0.836 (5)	0.044*
O4	0.05208 (14)	1.1036 (15)	0.9894 (4)	0.0353 (12)
H4O	0.048 (3)	0.933 (15)	1.030 (6)	0.053*
O5	0.12324 (12)	0.7692 (12)	1.0737 (3)	0.0182 (9)
H5O	0.1471 (12)	0.682 (18)	1.092 (5)	0.027*
O1W	0.25912 (12)	1.3152 (13)	0.9988 (3)	0.0209 (9)
H1W1	0.2444 (17)	1.382 (18)	1.050 (3)	0.031*
H1W2	0.2806 (14)	1.199 (17)	1.020 (4)	0.031*
O2W	−0.02887 (15)	1.3435 (17)	0.8657 (5)	0.0506 (16)
H2W1	−0.042 (3)	1.30 (2)	0.810 (4)	0.076*
H2W2	−0.038 (3)	1.534 (14)	0.890 (7)	0.076*
N1	0.27850 (13)	0.8683 (12)	0.7531 (3)	0.0139 (9)
N2	0.24174 (13)	0.9859 (13)	0.8027 (3)	0.0138 (9)
H2N	0.2431	1.0511	0.8684	0.017*
C1	0.35563 (16)	0.5778 (15)	0.6648 (4)	0.0144 (10)
C2	0.39381 (16)	0.4251 (16)	0.6295 (4)	0.0173 (11)
H2	0.3970	0.3730	0.5576	0.021*
C3	0.42746 (17)	0.3479 (16)	0.6985 (4)	0.0193 (12)
H3	0.4535	0.2431	0.6743	0.023*
C4	0.42245 (16)	0.4256 (16)	0.8025 (4)	0.0160 (11)
C5	0.38470 (16)	0.5781 (15)	0.8398 (4)	0.0143 (10)
H5	0.3820	0.6294	0.9118	0.017*
C6	0.35065 (15)	0.6562 (15)	0.7712 (4)	0.0126 (10)
C7	0.31105 (16)	0.7988 (15)	0.8138 (4)	0.0130 (10)
H7	0.3089	0.8415	0.8865	0.016*
C8	0.20343 (16)	1.0010 (13)	0.7503 (4)	0.0108 (10)
C9	0.16507 (16)	1.0460 (15)	0.8180 (4)	0.0130 (10)
C10	0.12793 (16)	1.2046 (16)	0.7771 (4)	0.0163 (11)
H10	0.1281	1.2984	0.7086	0.020*
C11	0.09101 (17)	1.2242 (15)	0.8368 (4)	0.0176 (12)
C12	0.09010 (17)	1.0775 (17)	0.9364 (4)	0.0204 (12)
C13	0.12713 (17)	0.9146 (15)	0.9777 (4)	0.0152 (11)
C14	0.16479 (17)	0.9040 (15)	0.9188 (4)	0.0149 (11)
H14	0.1903	0.8006	0.9468	0.018*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0094 (2)	0.0195 (3)	0.0247 (3)	−0.0001 (2)	−0.00302 (17)	−0.0020 (3)
O1	0.0160 (18)	0.032 (3)	0.0125 (17)	0.0031 (18)	−0.0028 (14)	−0.0016 (17)
O2	0.0127 (17)	0.027 (2)	0.0148 (18)	−0.0039 (16)	−0.0015 (14)	0.0021 (17)
O3	0.0142 (19)	0.041 (3)	0.034 (2)	0.0120 (19)	−0.0051 (17)	−0.005 (2)
O4	0.016 (2)	0.060 (4)	0.030 (2)	0.009 (2)	0.0035 (18)	−0.013 (2)
O5	0.0129 (17)	0.031 (2)	0.0110 (17)	−0.0007 (17)	0.0027 (14)	−0.0045 (16)
O1W	0.0162 (18)	0.035 (2)	0.0113 (17)	0.0027 (18)	0.0015 (14)	−0.0044 (18)
O2W	0.016 (2)	0.049 (4)	0.087 (5)	0.010 (2)	0.010 (2)	0.013 (3)
N1	0.0107 (19)	0.014 (3)	0.017 (2)	−0.0016 (17)	0.0010 (16)	−0.0008 (18)
N2	0.0096 (19)	0.019 (3)	0.013 (2)	−0.0004 (18)	−0.0007 (15)	−0.0028 (18)

C1	0.014 (2)	0.016 (3)	0.014 (2)	−0.002 (2)	−0.0013 (19)	−0.002 (2)
C2	0.013 (2)	0.025 (3)	0.014 (2)	−0.001 (2)	0.0024 (19)	0.000 (2)
C3	0.013 (2)	0.020 (3)	0.025 (3)	0.001 (2)	0.006 (2)	−0.003 (2)
C4	0.012 (2)	0.014 (3)	0.021 (3)	−0.001 (2)	−0.003 (2)	0.003 (2)
C5	0.015 (2)	0.013 (3)	0.015 (2)	−0.001 (2)	0.0005 (19)	−0.002 (2)
C6	0.010 (2)	0.015 (3)	0.013 (2)	−0.003 (2)	0.0008 (18)	0.001 (2)
C7	0.013 (2)	0.015 (3)	0.012 (2)	−0.006 (2)	0.0022 (18)	−0.003 (2)
C8	0.013 (2)	0.004 (3)	0.015 (2)	−0.0007 (18)	−0.0019 (18)	0.0009 (19)
C9	0.011 (2)	0.013 (3)	0.015 (2)	0.000 (2)	−0.0027 (19)	−0.003 (2)
C10	0.013 (2)	0.016 (3)	0.020 (3)	0.001 (2)	−0.0034 (19)	0.000 (2)
C11	0.017 (2)	0.013 (3)	0.022 (3)	0.008 (2)	−0.007 (2)	−0.007 (2)
C12	0.012 (2)	0.027 (3)	0.022 (3)	0.002 (2)	0.001 (2)	−0.012 (2)
C13	0.016 (2)	0.017 (3)	0.013 (2)	0.001 (2)	0.0025 (19)	−0.008 (2)
C14	0.013 (2)	0.016 (3)	0.015 (2)	0.001 (2)	−0.0022 (19)	−0.006 (2)

Geometric parameters (Å, °)

Br1—C4	1.909 (5)	C1—C6	1.408 (7)
O1—C1	1.361 (6)	C2—C3	1.393 (8)
O1—H1O	0.840 (10)	C2—H2	0.9500
O2—C8	1.236 (6)	C3—C4	1.378 (8)
O3—C11	1.362 (6)	C3—H3	0.9500
O3—H3O	0.838 (10)	C4—C5	1.388 (7)
O4—C12	1.362 (7)	C5—C6	1.400 (7)
O4—H4O	0.840 (10)	C5—H5	0.9500
O5—C13	1.357 (7)	C6—C7	1.446 (7)
O5—H5O	0.841 (10)	C7—H7	0.9500
O1W—H1W1	0.839 (10)	C8—C9	1.482 (7)
O1W—H1W2	0.838 (10)	C9—C10	1.395 (7)
O2W—H2W1	0.838 (10)	C9—C14	1.403 (7)
O2W—H2W2	0.839 (10)	C10—C11	1.378 (7)
N1—C7	1.296 (7)	C10—H10	0.9500
N1—N2	1.378 (6)	C11—C12	1.396 (8)
N2—C8	1.359 (6)	C12—C13	1.401 (8)
N2—H2N	0.8800	C13—C14	1.390 (7)
C1—C2	1.391 (7)	C14—H14	0.9500
C1—O1—H1O	113 (5)	C5—C6—C7	118.3 (4)
C11—O3—H3O	111 (6)	C1—C6—C7	122.9 (5)
C12—O4—H4O	112 (6)	N1—C7—C6	120.1 (4)
C13—O5—H5O	110 (5)	N1—C7—H7	120.0
H1W1—O1W—H1W2	109.6 (18)	C6—C7—H7	120.0
H2W1—O2W—H2W2	109.5 (18)	O2—C8—N2	122.9 (4)
C7—N1—N2	115.1 (4)	O2—C8—C9	123.0 (4)
C8—N2—N1	120.0 (4)	N2—C8—C9	114.1 (4)
C8—N2—H2N	120.0	C10—C9—C14	120.3 (5)
N1—N2—H2N	120.0	C10—C9—C8	119.0 (5)
O1—C1—C2	118.3 (5)	C14—C9—C8	120.4 (5)
O1—C1—C6	121.6 (5)	C11—C10—C9	119.5 (5)
C2—C1—C6	120.1 (5)	C11—C10—H10	120.2

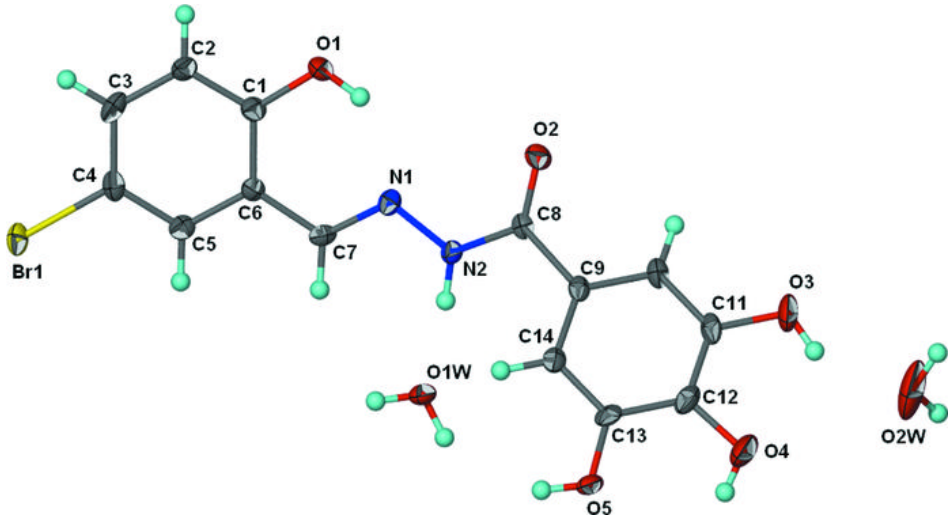
C1—C2—C3	120.6 (5)	C9—C10—H10	120.2
C1—C2—H2	119.7	O3—C11—C10	119.3 (5)
C3—C2—H2	119.7	O3—C11—C12	120.1 (5)
C4—C3—C2	119.1 (5)	C10—C11—C12	120.6 (5)
C4—C3—H3	120.5	O4—C12—C11	116.7 (5)
C2—C3—H3	120.5	O4—C12—C13	123.0 (5)
C3—C4—C5	121.5 (5)	C11—C12—C13	120.3 (5)
C3—C4—Br1	119.2 (4)	O5—C13—C14	124.1 (5)
C5—C4—Br1	119.3 (4)	O5—C13—C12	116.7 (5)
C4—C5—C6	120.0 (5)	C14—C13—C12	119.2 (5)
C4—C5—H5	120.0	C13—C14—C9	120.0 (5)
C6—C5—H5	120.0	C13—C14—H14	120.0
C5—C6—C1	118.7 (5)	C9—C14—H14	120.0
C7—N1—N2—C8	−167.2 (5)	N2—C8—C9—C10	154.0 (5)
O1—C1—C2—C3	−179.3 (5)	O2—C8—C9—C14	147.8 (5)
C6—C1—C2—C3	0.1 (9)	N2—C8—C9—C14	−31.4 (7)
C1—C2—C3—C4	−0.1 (9)	C14—C9—C10—C11	0.6 (9)
C2—C3—C4—C5	0.1 (9)	C8—C9—C10—C11	175.2 (5)
C2—C3—C4—Br1	178.5 (4)	C9—C10—C11—O3	−179.5 (5)
C3—C4—C5—C6	0.0 (9)	C9—C10—C11—C12	−2.0 (9)
Br1—C4—C5—C6	−178.4 (4)	O3—C11—C12—O4	−1.4 (8)
C4—C5—C6—C1	−0.1 (8)	C10—C11—C12—O4	−178.9 (6)
C4—C5—C6—C7	177.0 (5)	O3—C11—C12—C13	178.9 (5)
O1—C1—C6—C5	179.4 (5)	C10—C11—C12—C13	1.4 (9)
C2—C1—C6—C5	0.1 (8)	O4—C12—C13—O5	2.2 (9)
O1—C1—C6—C7	2.5 (9)	C11—C12—C13—O5	−178.1 (5)
C2—C1—C6—C7	−176.9 (5)	O4—C12—C13—C14	−179.1 (6)
N2—N1—C7—C6	176.8 (5)	C11—C12—C13—C14	0.6 (9)
C5—C6—C7—N1	−178.7 (5)	O5—C13—C14—C9	176.7 (5)
C1—C6—C7—N1	−1.8 (8)	C12—C13—C14—C9	−1.9 (8)
N1—N2—C8—O2	−13.6 (8)	C10—C9—C14—C13	1.3 (8)
N1—N2—C8—C9	165.7 (5)	C8—C9—C14—C13	−173.2 (5)
O2—C8—C9—C10	−26.7 (8)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···N1	0.84 (1)	1.91 (5)	2.616 (6)	141 (7)
O3—H3o···O2w	0.84 (1)	1.96 (4)	2.736 (6)	153 (7)
O4—H4o···O2w^{i}	0.84 (1)	1.81 (3)	2.623 (8)	163 (9)
O5—H5o···O2^{ii}	0.84 (1)	1.93 (2)	2.764 (5)	171 (7)
O1w—H1w1···O2^{iii}	0.84 (1)	1.98 (2)	2.812 (5)	170 (6)
O1w—H1w2···O1^{ii}	0.84 (1)	2.09 (2)	2.914 (6)	167 (6)
O2w—H2w1···O3^{iv}	0.84 (1)	2.13 (5)	2.845 (9)	142 (8)
O2w—H2w2···O4^{v}	0.84 (1)	2.12 (4)	2.900 (8)	154 (8)

Symmetry codes: (i) −*x*, −*y*+2, −*z*+2; (ii) *x*, −*y*+3/2, *z*+1/2; (iii) *x*, −*y*+5/2, *z*+1/2; (iv) −*x*, *y*−1/2, −*z*+3/2; (v) −*x*, −*y*+3, −*z*+2.

Fig. 1


C2
O1
C3
C1
O2
C4
N1
C6
C8
C5
C7
N2
C9
Br1
O3
C11
C14
O1W
C12
O2W
C13
O4
O5