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(*E*)-4-Bromo-5-methoxy-2-{[(2-methoxyphenyl)-imino]methyl}phenol monohydrate

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Keywords: crystal structure; Schiff base; hydrogen bonding; centrosymmetric tetramer.

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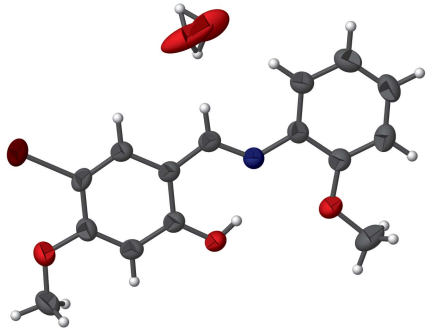
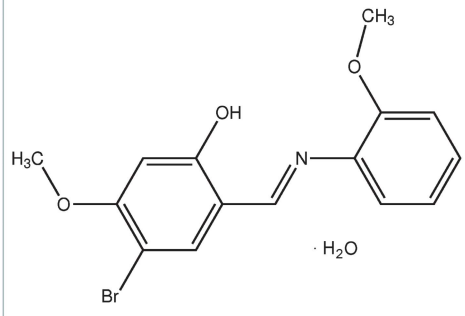
Structural data: full structural data are available from iucrdata.iucr.org

In the title Schiff base hydrate, $C_{15}H_{14}BrNO_3\cdot H_2O$, the dihedral angle between the benzene rings is 0.9 (2)° and an intramolecular O—H···N hydrogen bond closes an *S*(6) ring. In the crystal, O_w—H···O (w = water) hydrogen bonds link the components into centrosymmetric tetramers (two Schiff bases and two water molecules). Weak C—H···O_w interactions consolidate the linking of the molecules within the tetramers. The O atom of the water molecule is disordered over two adjacent sites in a 0.73 (9):0.27 (9) ratio.

3D view



Chemical scheme



Structure description

As part of our ongoing studies of Schiff bases (Köysal *et al.*, 2015), we now describe the synthesis and structure of the title compound (Fig. 1), which crystallizes as a monohydrate.

The dihedral angle between the C2–C7 and C9–C14 benzene rings is 0.9 (2)° and the entire molecule is almost planar (r.m.s. deviation for all non-H atoms = 0.018 Å) The bond distances of imino group atoms [N1—C8 = 1.306 (4); N1—C9 = 1.415 (5) Å] are consistent with those in related structures (Köysal *et al.*, 2015). An intramolecular O—H···N hydrogen bond closes an *S*(6) ring (Table 1).

In the crystal (Fig. 2), O_w—H···O (w = water) hydrogen bonds link the components into centrosymmetric tetramers (two Schiff bases and two water molecules). Weak C—H···O_w interactions are also observed (Table 1).

Synthesis and crystallization

Solutions of 5-bromo-2-hydroxy-4-methoxybenzaldehyde (0.0154 g, 0.066 mmol) in 20 ml ethanol and 2-methoxyaniline (0.0071 g, 0.066 mmol) in 20 ml ethanol were mixed and

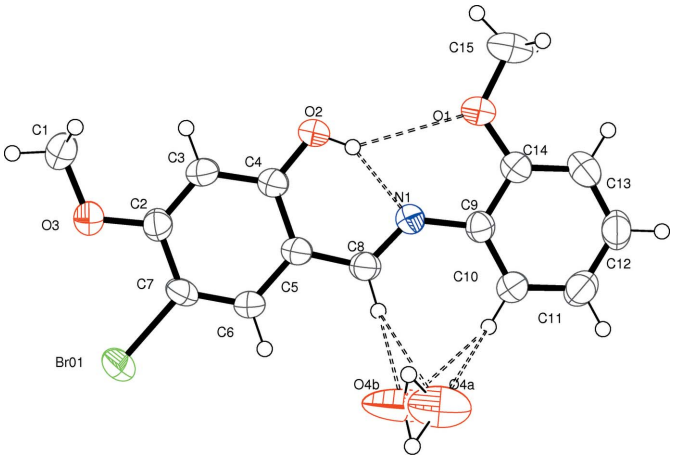


Figure 1
A view of the title compound, with 50% probability displacement ellipsoids. Intramolecular hydrogen bonds and bonds to the solvate water molecule in the asymmetric unit are drawn as double dashed lines.

stirred for 18 h under reflux. Yellow prisms of the title compound were obtained from ethanol solution by slow evaporation (yield 67%; m.p. 401–405 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The O atom of the water molecule of crystallization is disordered over two adjacent sites in a 0.73 (9):0.27 (9) ratio.

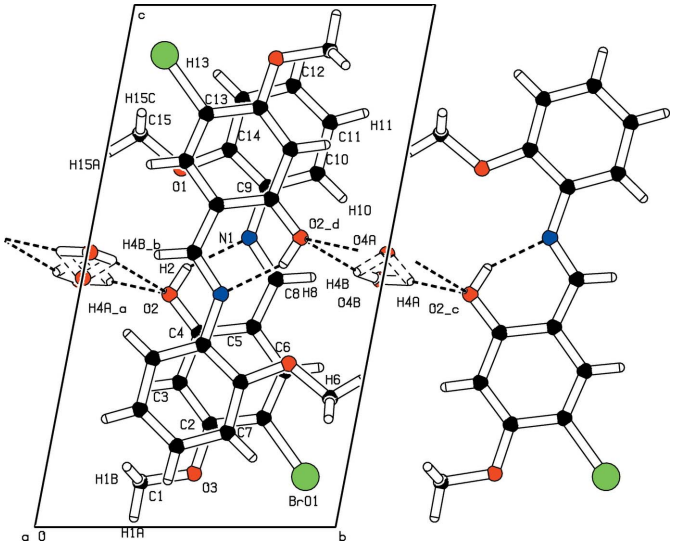


Figure 2
The packing, viewed along the *bc* plane showing the hydrogen-bonding interactions.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1	0.82	2.64	3.340 (4)	144
O2—H2···N1	0.82	1.84	2.581 (4)	149
O4B—H4A···O2^{i}	0.86	2.01	2.85 (2)	166
O4B—H4B···O2^{ii}	0.77	2.23	2.99 (3)	170
C8—H8···O4B	0.93	2.44	3.18 (2)	136
C10—H10···O4A	0.93	2.56	3.31 (6)	138

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, -y+1, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{14}BrNO_3\cdot H_2O$
M_r	354.20
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	6.922 (3), 8.498 (3), 14.064 (5)
α, β, γ (°)	78.30 (3), 85.51 (3), 69.10 (3)
V (Å^3)	756.8 (6)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.73
Crystal size (mm)	0.79 × 0.39 × 0.07
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2002)
T_{min}, T_{max}	0.115, 0.755
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11464, 3139, 2179
R_{int}	0.124
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.628
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.131, 0.98
No. of reflections	3139
No. of parameters	206
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.73, −0.53

Computer programs: *X-AREA* (Stoe & Cie, 2002), *SHELXT2016* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

Funding information

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full crystallographic data

IUCrData (2017). **2**, x171730 [https://doi.org/10.1107/S2414314617017308]

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(*E*)-4-Bromo-5-methoxy-2-{[(2-methoxyphenyl)imino]methyl}phenol monohydrate

Crystal data

$C_{15}H_{14}BrNO_3 \cdot H_2O$
$M_r = 354.20$
Triclinic, $P\overline{1}$
$a = 6.922$ (3) Å
$b = 8.498$ (3) Å
$c = 14.064$ (5) Å
$\alpha = 78.30$ (3)°
$\beta = 85.51$ (3)°
$\gamma = 69.10$ (3)°
$V = 756.8$ (6) Å^3
$Z = 2$
$F(000) = 360$
$D_x = 1.554$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 11116 reflections
$\theta = 1.5$–27.6°
$\mu = 2.73$ mm^{-1}
$T = 293$ K
Prism, yellow
0.79 × 0.39 × 0.07 mm

Data collection

Stoe IPDS 2 diffractometer
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)
$T_{min} = 0.115$, $T_{max} = 0.755$
11464 measured reflections
3139 independent reflections
2179 reflections with $I > 2\sigma(I)$
$R_{int} = 0.124$
$\theta_{max} = 26.5°$, $\theta_{min} = 1.5°$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.131$
$S = 0.98$
3139 reflections
206 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.73$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All H atoms were positioned geometrically and rifined using a riding model, with C—H distances of 0.93 Å and methyl C—H distances 0.96 Å.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O4A	0.268 (7)	0.998 (5)	0.523 (5)	0.088 (14)	0.27 (9)
O4B	0.267 (3)	0.9909 (19)	0.474 (4)	0.127 (9)	0.73 (9)
Br01	0.16383 (8)	0.86773 (6)	0.09633 (3)	0.0673 (2)	
O2	0.3544 (4)	0.3003 (3)	0.44583 (18)	0.0523 (7)	
H2	0.326710	0.340164	0.495697	0.078*	
O3	0.3291 (4)	0.4946 (4)	0.10287 (19)	0.0540 (7)	
O1	0.3128 (4)	0.2594 (4)	0.6870 (2)	0.0571 (7)	
N1	0.2236 (4)	0.5296 (4)	0.5540 (2)	0.0393 (7)	
C4	0.3119 (5)	0.4231 (5)	0.3718 (3)	0.0405 (8)	
C5	0.2295 (5)	0.5992 (5)	0.3830 (2)	0.0394 (8)	
C2	0.3019 (5)	0.5180 (5)	0.1966 (2)	0.0425 (8)	
C8	0.1872 (5)	0.6453 (5)	0.4743 (3)	0.0397 (8)	
H8	0.131949	0.760763	0.478454	0.048*	
C14	0.2419 (5)	0.4074 (5)	0.7224 (3)	0.0447 (9)	
C6	0.1875 (5)	0.7301 (5)	0.2988 (3)	0.0431 (8)	
H6	0.135573	0.844418	0.305509	0.052*	
C9	0.1913 (5)	0.5563 (5)	0.6510 (2)	0.0385 (8)	
C10	0.1166 (6)	0.7147 (5)	0.6772 (3)	0.0463 (9)	
H10	0.082481	0.813821	0.629793	0.056*	
C3	0.3453 (6)	0.3890 (5)	0.2766 (3)	0.0448 (9)	
H3	0.398395	0.275763	0.267889	0.054*	
C7	0.2221 (6)	0.6910 (5)	0.2087 (3)	0.0448 (9)	
C11	0.0924 (6)	0.7256 (6)	0.7747 (3)	0.0557 (10)	
H11	0.042377	0.832531	0.792726	0.067*	
C1	0.4151 (7)	0.3224 (6)	0.0866 (3)	0.0607 (11)	
H1A	0.426318	0.323287	0.018012	0.091*	
H1B	0.549855	0.268133	0.115233	0.091*	
H1C	0.326988	0.260190	0.115503	0.091*	
C13	0.2163 (6)	0.4197 (6)	0.8193 (3)	0.0550 (10)	
H13	0.248926	0.320906	0.866917	0.066*	
C12	0.1421 (6)	0.5790 (6)	0.8452 (3)	0.0567 (11)	
H12	0.125648	0.587391	0.910488	0.068*	
C15	0.3722 (9)	0.1000 (6)	0.7530 (4)	0.0776 (15)	
H15A	0.418462	0.007972	0.717331	0.116*	
H15C	0.482225	0.092343	0.793208	0.116*	
H15B	0.255889	0.092080	0.793013	0.116*	
H4A	0.275360	1.089784	0.471441	0.116*	
H4B	0.364529	0.912261	0.488679	0.116*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4A	0.047 (15)	0.069 (14)	0.15 (3)	−0.033 (9)	0.004 (14)	−0.001 (15)
O4B	0.075 (6)	0.044 (5)	0.26 (2)	−0.022 (4)	−0.077 (9)	0.019 (8)
Br01	0.0949 (4)	0.0441 (3)	0.0399 (2)	−0.0031 (2)	−0.00083 (19)	0.00424 (17)
O2	0.0781 (18)	0.0306 (13)	0.0399 (13)	−0.0099 (12)	−0.0063 (12)	−0.0028 (11)
O3	0.0683 (17)	0.0474 (16)	0.0378 (13)	−0.0097 (13)	0.0003 (12)	−0.0090 (11)
O1	0.0797 (19)	0.0346 (15)	0.0481 (15)	−0.0107 (13)	−0.0054 (13)	−0.0028 (12)
N1	0.0445 (16)	0.0366 (16)	0.0358 (14)	−0.0127 (12)	−0.0047 (12)	−0.0056 (12)
C4	0.0441 (19)	0.0344 (19)	0.0399 (18)	−0.0101 (15)	−0.0047 (14)	−0.0048 (14)
C5	0.0435 (19)	0.0319 (18)	0.0402 (17)	−0.0106 (14)	−0.0037 (14)	−0.0039 (14)
C2	0.0438 (19)	0.044 (2)	0.0355 (17)	−0.0099 (15)	−0.0022 (14)	−0.0083 (15)
C8	0.0421 (18)	0.0339 (18)	0.0420 (18)	−0.0119 (14)	−0.0049 (14)	−0.0053 (14)
C14	0.049 (2)	0.040 (2)	0.0422 (19)	−0.0120 (16)	−0.0058 (15)	−0.0056 (16)
C6	0.053 (2)	0.0296 (18)	0.0411 (19)	−0.0071 (15)	−0.0043 (15)	−0.0058 (14)
C9	0.0384 (18)	0.042 (2)	0.0370 (17)	−0.0151 (15)	−0.0029 (13)	−0.0076 (15)
C10	0.053 (2)	0.040 (2)	0.045 (2)	−0.0126 (16)	−0.0045 (16)	−0.0102 (16)
C3	0.050 (2)	0.0319 (19)	0.047 (2)	−0.0060 (15)	−0.0048 (15)	−0.0087 (15)
C7	0.051 (2)	0.0323 (18)	0.0392 (18)	−0.0051 (15)	−0.0046 (15)	0.0040 (15)
C11	0.061 (2)	0.053 (3)	0.057 (2)	−0.0162 (19)	−0.0006 (19)	−0.022 (2)
C1	0.075 (3)	0.056 (3)	0.046 (2)	−0.011 (2)	−0.0012 (19)	−0.0194 (19)
C13	0.061 (2)	0.055 (3)	0.042 (2)	−0.0179 (19)	−0.0062 (17)	0.0029 (18)
C12	0.064 (2)	0.067 (3)	0.038 (2)	−0.018 (2)	−0.0026 (17)	−0.0144 (19)
C15	0.097 (4)	0.039 (2)	0.081 (3)	−0.013 (2)	−0.009 (3)	0.006 (2)

Geometric parameters (Å, º)

O4A—H4A	0.96 (8)	C14—C13	1.380 (5)
O4A—H4B	0.9759	C14—C9	1.399 (5)
O4B—H4A	0.86 (7)	C6—C7	1.357 (5)
O4B—H4B	0.7704	C6—H6	0.9300
Br01—C7	1.902 (3)	C9—C10	1.374 (5)
O2—C4	1.284 (4)	C10—C11	1.387 (6)
O2—H2	0.8200	C10—H10	0.9300
O3—C2	1.361 (4)	C3—H3	0.9300
O3—C1	1.429 (5)	C11—C12	1.379 (6)
O1—C14	1.359 (5)	C11—H11	0.9300
O1—C15	1.420 (5)	C1—H1A	0.9600
N1—C8	1.306 (4)	C1—H1B	0.9600
N1—C9	1.415 (5)	C1—H1C	0.9600
C4—C3	1.412 (5)	C13—C12	1.380 (7)
C4—C5	1.436 (5)	C13—H13	0.9300
C5—C8	1.398 (5)	C12—H12	0.9300
C5—C6	1.418 (5)	C15—H15A	0.9600
C2—C3	1.369 (5)	C15—H15C	0.9600
C2—C7	1.416 (5)	C15—H15B	0.9600
C8—H8	0.9300		

H4A—O4A—H4B	91.5	C11—C10—H10	120.2
H4A—O4B—H4B	116.8	C2—C3—C4	121.9 (4)
C4—O2—H2	109.5	C2—C3—H3	119.1
C2—O3—C1	117.6 (3)	C4—C3—H3	119.1
C14—O1—C15	119.2 (4)	C6—C7—C2	120.5 (3)
C8—N1—C9	127.9 (3)	C6—C7—Br01	120.6 (3)
O2—C4—C3	120.9 (3)	C2—C7—Br01	118.8 (3)
O2—C4—C5	121.3 (3)	C12—C11—C10	120.4 (4)
C3—C4—C5	117.8 (3)	C12—C11—H11	119.8
C8—C5—C6	119.1 (3)	C10—C11—H11	119.8
C8—C5—C4	121.9 (3)	O3—C1—H1A	109.5
C6—C5—C4	119.0 (3)	O3—C1—H1B	109.5
O3—C2—C3	125.0 (4)	H1A—C1—H1B	109.5
O3—C2—C7	115.3 (3)	O3—C1—H1C	109.5
C3—C2—C7	119.7 (3)	H1A—C1—H1C	109.5
N1—C8—C5	121.4 (3)	H1B—C1—H1C	109.5
N1—C8—H8	119.3	C12—C13—C14	119.8 (4)
C5—C8—H8	119.3	C12—C13—H13	120.1
O1—C14—C13	125.8 (4)	C14—C13—H13	120.1
O1—C14—C9	114.3 (3)	C11—C12—C13	120.2 (4)
C13—C14—C9	119.9 (4)	C11—C12—H12	119.9
C7—C6—C5	121.0 (3)	C13—C12—H12	119.9
C7—C6—H6	119.5	O1—C15—H15A	109.5
C5—C6—H6	119.5	O1—C15—H15C	109.5
C10—C9—C14	120.1 (3)	H15A—C15—H15C	109.5
C10—C9—N1	124.5 (3)	O1—C15—H15B	109.5
C14—C9—N1	115.4 (3)	H15A—C15—H15B	109.5
C9—C10—C11	119.6 (4)	H15C—C15—H15B	109.5
C9—C10—H10	120.2		

O2—C4—C5—C8	−1.6 (5)	C8—N1—C9—C14	179.2 (3)
C3—C4—C5—C8	178.6 (3)	C14—C9—C10—C11	0.2 (6)
O2—C4—C5—C6	179.2 (3)	N1—C9—C10—C11	−179.5 (3)
C3—C4—C5—C6	−0.6 (5)	O3—C2—C3—C4	−179.6 (3)
C1—O3—C2—C3	−1.9 (6)	C7—C2—C3—C4	0.3 (6)
C1—O3—C2—C7	178.2 (4)	O2—C4—C3—C2	−179.7 (4)
C9—N1—C8—C5	179.3 (3)	C5—C4—C3—C2	0.1 (5)
C6—C5—C8—N1	−179.8 (3)	C5—C6—C7—C2	−0.3 (6)
C4—C5—C8—N1	1.0 (5)	C5—C6—C7—Br01	179.7 (3)
C15—O1—C14—C13	−1.6 (6)	O3—C2—C7—C6	179.7 (3)
C15—O1—C14—C9	179.0 (4)	C3—C2—C7—C6	−0.3 (6)
C8—C5—C6—C7	−178.6 (4)	O3—C2—C7—Br01	−0.2 (5)
C4—C5—C6—C7	0.7 (5)	C3—C2—C7—Br01	179.8 (3)
O1—C14—C9—C10	179.6 (3)	C9—C10—C11—C12	−0.2 (6)
C13—C14—C9—C10	0.2 (6)	O1—C14—C13—C12	−179.9 (4)
O1—C14—C9—N1	−0.6 (5)	C9—C14—C13—C12	−0.5 (6)
C13—C14—C9—N1	179.9 (3)	C10—C11—C12—C13	−0.1 (7)

C8—N1—C9—C10	−1.1 (6)	C14—C13—C12—C11	0.4 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.82	2.64	3.340 (4)	144
O2—H2···N1	0.82	1.84	2.581 (4)	149
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O4*B*—H4*B*···O2^{ii}	0.77	2.23	2.99 (3)	170
C8—H8···O4*B*	0.93	2.44	3.18 (2)	136
C10—H10···O4*A*	0.93	2.56	3.31 (6)	138

Symmetry codes: (i) x, y+1, z; (ii) −x+1, −y+1, −z+1.