Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Bromo-2-({4-[(hydroxyimino)methyl]-phenyl}iminomethyl)phenol

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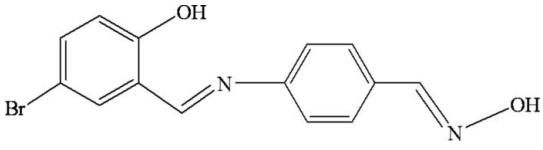
Received 11 June 2010; accepted 8 July 2010

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.027; wR factor = 0.054; data-to-parameter ratio = 24.1.

In the title compound, $C_{14}H_{11}BrN_2O_2$, the mean planes of the two benzene rings are almost parallel to each other, making a dihedral angle of 4.09 (1)°. An intramolecular O—H···N hydrogen bond occurs. In the crystal, intermolecular O—H···N and C—H···O hydrogen bonds link the molecules into a chain-like supramolecular structure.

Related literature

For background to the use of Schiff bases as ligands in coordination chemistry, see: Biswas *et al.* (2008); Dong *et al.* (2010). For the synthesis of the title compound and related structures, see: Dong *et al.* (2007, 2009); Zhao *et al.* (2009).



Experimental

Crystal data

$C_{14}H_{11}BrN_2O_2$
M_r = 319.16
Orthorhombic, $P2_12_12_1$
a = 4.4279 (5) Å
b = 12.1790 (16) Å
c = 23.196 (2) Å
V = 1250.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.29 mm^{-1}
T = 113 K
0.26 × 0.24 × 0.22 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2009)
T_{min} = 0.482, T_{max} = 0.532
15336 measured reflections
4346 independent reflections
2818 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.054
S = 0.85
4346 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Absolute structure: Flack (1983), 1615 Friedel pairs
Flack parameter: −0.022 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.87 (2)	1.84 (2)	2.593 (2)	143 (2)
O2—H2···N2^{i}	0.75 (3)	2.08 (3)	2.830 (2)	173 (2)
C5—H5···O2^{ii}	0.95	2.54	3.463 (3)	163

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

Data collection: *CrystalClear-SM Expert* (Rigaku/MSC, 2009); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2009); software used to prepare material for publication: *CrystalStructure*.

This work was supported by the Foundation of the Education Department of Gansu Province (No. 0904–11) and the 'JingLan' Talent Engineering Funds of Lanzhou Jiaotong University, which are gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2295).

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supporting information

Acta Cryst. (2010). E**66**, o2014 [https://doi.org/10.1107/S160053681002698X]

4-Bromo-2-({4-[(hydroxyimino)methyl]phenyl}iminomethyl)phenol

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S1. Comment

Schiff bases have been used widely as versatile ligands in coordination chemistry (Biswas *et al.*, 2008; Dong *et al.*, 2010). Recently, the structures of a few Schiff base compounds have been reported (Dong *et al.*, 2007; Dong *et al.*, 2009). In this paper, we report the synthesis and crystal structure of the title compound, (I).

In the title compound (Fig. 1) the bond lengths and angles are in normal ranges and agree very well with the corresponding bond lengths and angles reported in similar structures (Dong *et al.*, 2007; Dong *et al.*, 2009; Zhao *et al.*, 2009). The mean planes of the two benzene rings are almost parallel to each other making a dihedral angle of 4.09 (1) ° with respect to each other. There is an intramolecular hydrogen bond, O1—H1···N1 (Tab. 1). Besides, intermolecular hydrogen bonds, O2—H2···N2 and C5—H5···O2 link molecules into infinite catenarian supramolecular shape (Tab. 1 & Fig. 2).

S2. Experimental

The title compound was synthesized according to methods reported earlier (Zhao *et al.*, 2009; Dong *et al.*, 2009). A solution of 1-(4-aminophenyl)-methanal (1.21 g, 10 mmol) in methanol (15 ml) was added to a mixture of hydroxylamine sulfate (1.31 g, 10 mmol) and sodium acetate (2.0 g, 25 mmol). After refluxing for 4–5 h, the reaction was completed. The solvent was evaporated under vacuo. Demineralized water (40 ml) was added, cooled to 268–265 K and filtered, resulting in 4-aminobenzaldehyde oxime as a crystalline solid (yield; 1.18 g, 86.7%; m.p. 395–397 K). To an ethalnol solution (5 ml) of 4-aminobenzaldehyde oxime (0.1362 g, 1 mmol) was added dropwise an ethanol solution (5 ml) of 5-bromo-2-hydroxybenzaldehyde (0.19995 g, 1 mmol). The mixture solution was stirred at 328–333 K for 5 h. After cooling to room temperature, the precipitate was filtered off, and washed successively three times with ethanol. The product was dried *in vacuo* and purified by recrystallization from ethanol to yield 267.5 mg (Yield, 83.8%) of solid; m.p. 490–491 K. Pale-yellow needle-like single crystals suitable for X-ray diffraction studies were obtained by slow evaporation from a solution of methanol at room temperature in about one month.

S3. Refinement

An absolute structure was determined by Flack (1983) method employing 1615 Friedel pairs. H atoms were treated as riding atoms with distances C—H = 0.95 Å and U_{iso}(H) = 1.2 U_{eq}(C). The hydroxyl H-atoms were located from a difference Fourier map and were allowed to refine freely.

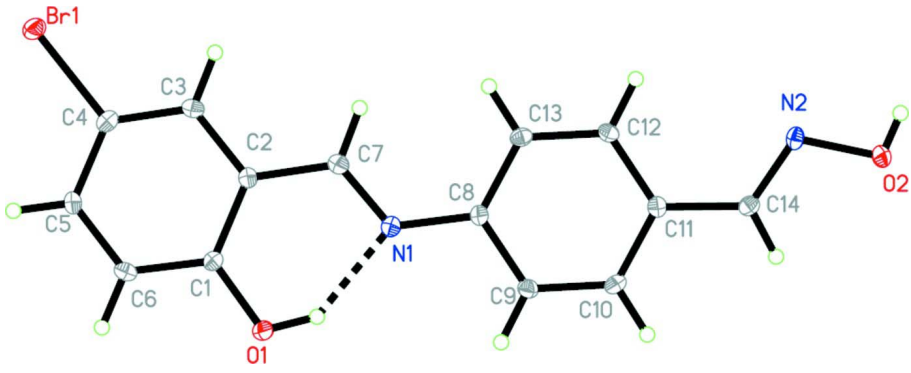

Figure 1

The molecule structure of the title complex with atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

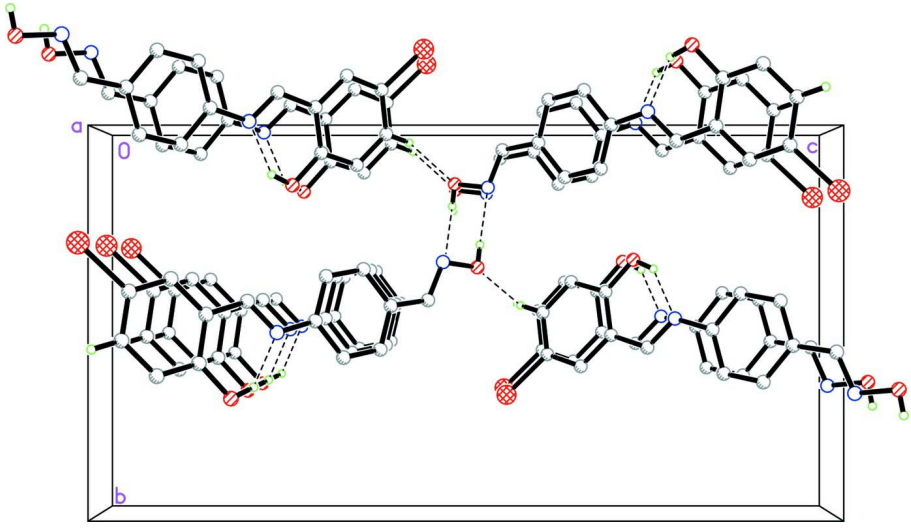

Figure 2

Unit cell packing of the title compound. Intramolecular and intermolecular hydrogen bonds are shown as dashed lines.

4-Bromo-2-({4-[(hydroxyimino)methyl]phenyl}iminomethyl)phenol

Crystal data

$C_{14}H_{11}BrN_2O_2$
$M_r = 319.16$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 4.4279\ (5)$ Å
$b = 12.1790\ (16)$ Å
$c = 23.196\ (2)$ Å
$V = 1250.9\ (3)$ Å^3
$Z = 4$
$F(000) = 640$

$D_x = 1.695$ Mg m^{-3}
Melting point = 490–491 K
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 4929 reflections
$\theta = 1.9$–$32.9°$
$\mu = 3.29$ mm^{-1}
$T = 113$ K
Needle, pale-yellow
0.26 × 0.24 × 0.22 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Radiation source: Rotating Anode
Multilayer monochromator
Detector resolution: 14.222 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2009)
T_{min} = 0.482, T_{max} = 0.532
15336 measured reflections
4346 independent reflections
2818 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 32.9°, θ_{min} = 1.8°
h = −6→6
k = −18→17
l = −34→33

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.054
S = 0.85
4346 reflections
180 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0171P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Absolute structure: Flack (1983), 1615 Friedel pairs
Absolute structure parameter: −0.022 (7)

Special details

Experimental. Anal. Calc. for $C_{14}H_{11}BrN_2O_2$: C, 52.69; H, 3.47; N, 8.78. Found: C, 52.67; H, 3.44; N, 8.81.

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
Br1	0.93894 (6)	0.310770 (17)	0.055477 (9)	0.02449 (6)
O1	0.5216 (4)	0.65997 (11)	0.22190 (6)	0.0219 (4)
O2	−0.9560 (4)	0.35901 (13)	0.51519 (6)	0.0228 (4)
N1	0.1808 (4)	0.51372 (14)	0.27053 (7)	0.0158 (4)
N2	−0.7439 (4)	0.34661 (14)	0.47039 (7)	0.0165 (4)
C1	0.6150 (5)	0.57912 (16)	0.18617 (8)	0.0156 (5)
C2	0.5081 (5)	0.46983 (15)	0.19213 (8)	0.0150 (5)
C3	0.6111 (5)	0.38957 (16)	0.15310 (8)	0.0174 (5)
H3	0.5429	0.3159	0.1565	0.021*
C4	0.8098 (5)	0.41758 (17)	0.11007 (9)	0.0181 (5)
C5	0.9174 (5)	0.52501 (16)	0.10466 (8)	0.0174 (4)
H5	1.0571	0.5431	0.0751	0.021*
C6	0.8195 (5)	0.60434 (17)	0.14244 (9)	0.0189 (5)

H6	0.8922	0.6774	0.1387	0.023*
C7	0.2943 (5)	0.44092 (17)	0.23670 (9)	0.0172 (5)
H7	0.2362	0.3663	0.2411	0.021*
C8	−0.0289 (5)	0.48808 (15)	0.31419 (8)	0.0142 (4)
C9	−0.1308 (5)	0.57570 (17)	0.34784 (9)	0.0181 (5)
H9	−0.0580	0.6477	0.3405	0.022*
C10	−0.3373 (5)	0.55866 (17)	0.39193 (9)	0.0185 (5)
H10	−0.4072	0.6195	0.4138	0.022*
C11	−0.4435 (5)	0.45385 (15)	0.40458 (8)	0.0154 (4)
C12	−0.3426 (5)	0.36586 (16)	0.37064 (9)	0.0175 (5)
H12	−0.4139	0.2938	0.3783	0.021*
C13	−0.1407 (5)	0.38292 (16)	0.32620 (8)	0.0170 (5)
H13	−0.0766	0.3225	0.3034	0.020*
C14	−0.6628 (5)	0.44081 (17)	0.45110 (9)	0.0171 (5)
H14	−0.7489	0.5048	0.4678	0.021*
H1	0.384 (5)	0.6368 (17)	0.2459 (9)	0.022 (7)*
H2	−1.018 (7)	0.302 (2)	0.5186 (12)	0.070 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03228 (13)	0.02054 (9)	0.02065 (10)	0.00167 (11)	0.00598 (11)	−0.00300 (9)
O1	0.0290 (11)	0.0162 (7)	0.0204 (8)	−0.0007 (7)	0.0061 (8)	−0.0010 (6)
O2	0.0245 (10)	0.0219 (8)	0.0220 (8)	−0.0066 (8)	0.0081 (8)	−0.0010 (6)
N1	0.0149 (10)	0.0174 (9)	0.0150 (9)	0.0026 (8)	−0.0014 (8)	0.0015 (7)
N2	0.0123 (10)	0.0234 (10)	0.0138 (8)	−0.0013 (7)	0.0002 (8)	−0.0013 (7)
C1	0.0158 (13)	0.0163 (10)	0.0146 (10)	0.0027 (9)	−0.0017 (9)	−0.0003 (8)
C2	0.0134 (14)	0.0171 (9)	0.0146 (9)	0.0017 (8)	−0.0016 (9)	0.0026 (7)
C3	0.0174 (14)	0.0151 (10)	0.0196 (11)	−0.0007 (9)	−0.0048 (10)	0.0005 (8)
C4	0.0184 (13)	0.0202 (11)	0.0157 (10)	0.0042 (9)	−0.0025 (10)	−0.0015 (9)
C5	0.0149 (12)	0.0215 (10)	0.0157 (10)	0.0002 (10)	0.0016 (11)	0.0023 (8)
C6	0.0217 (13)	0.0148 (10)	0.0201 (11)	−0.0032 (9)	−0.0022 (10)	0.0014 (8)
C7	0.0159 (12)	0.0164 (10)	0.0194 (11)	−0.0005 (9)	−0.0019 (10)	0.0015 (9)
C8	0.0112 (12)	0.0177 (9)	0.0136 (9)	−0.0004 (9)	−0.0016 (9)	0.0009 (7)
C9	0.0186 (14)	0.0149 (10)	0.0209 (11)	0.0001 (9)	0.0010 (10)	0.0005 (8)
C10	0.0210 (13)	0.0168 (10)	0.0178 (11)	0.0031 (9)	−0.0005 (10)	−0.0037 (8)
C11	0.0129 (11)	0.0199 (10)	0.0134 (9)	−0.0015 (10)	−0.0030 (10)	−0.0001 (7)
C12	0.0195 (13)	0.0140 (10)	0.0190 (10)	0.0003 (9)	−0.0009 (10)	0.0027 (8)
C13	0.0189 (14)	0.0151 (10)	0.0170 (10)	0.0028 (9)	−0.0033 (9)	−0.0012 (8)
C14	0.0161 (11)	0.0184 (10)	0.0169 (11)	0.0001 (8)	−0.0019 (10)	−0.0024 (9)

Geometric parameters (Å, º)

Br1—C4	1.903 (2)	C5—H5	0.9500
O1—C1	1.352 (2)	C6—H6	0.9500
O1—H1	0.87 (2)	C7—H7	0.9500
O2—N2	1.409 (2)	C8—C9	1.397 (3)
O2—H2	0.75 (3)	C8—C13	1.401 (3)

N1—C7	1.286 (2)	C9—C10	1.387 (3)
N1—C8	1.409 (3)	C9—H9	0.9500
N2—C14	1.283 (2)	C10—C11	1.392 (3)
C1—C6	1.394 (3)	C10—H10	0.9500
C1—C2	1.419 (3)	C11—C12	1.403 (3)
C2—C3	1.408 (3)	C11—C14	1.460 (3)
C2—C7	1.445 (3)	C12—C13	1.380 (3)
C3—C4	1.374 (3)	C12—H12	0.9500
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.398 (3)	C14—H14	0.9500
C5—C6	1.375 (3)		

C1—O1—H1	111.7 (14)	N1—C7—H7	119.2
N2—O2—H2	103 (2)	C2—C7—H7	119.2
C7—N1—C8	122.91 (18)	C9—C8—C13	118.23 (19)
C14—N2—O2	110.38 (17)	C9—C8—N1	116.44 (18)
O1—C1—C6	118.97 (18)	C13—C8—N1	125.33 (18)
O1—C1—C2	121.43 (19)	C10—C9—C8	120.70 (19)
C6—C1—C2	119.60 (18)	C10—C9—H9	119.6
C3—C2—C1	118.72 (19)	C8—C9—H9	119.6
C3—C2—C7	120.18 (18)	C9—C10—C11	121.0 (2)
C1—C2—C7	121.09 (18)	C9—C10—H10	119.5
C4—C3—C2	120.14 (19)	C11—C10—H10	119.5
C4—C3—H3	119.9	C10—C11—C12	118.3 (2)
C2—C3—H3	119.9	C10—C11—C14	118.71 (18)
C3—C4—C5	121.05 (19)	C12—C11—C14	122.92 (18)
C3—C4—Br1	120.41 (16)	C13—C12—C11	120.70 (19)
C5—C4—Br1	118.53 (16)	C13—C12—H12	119.7
C6—C5—C4	119.5 (2)	C11—C12—H12	119.7
C6—C5—H5	120.2	C12—C13—C8	120.99 (19)
C4—C5—H5	120.2	C12—C13—H13	119.5
C5—C6—C1	120.93 (19)	C8—C13—H13	119.5
C5—C6—H6	119.5	N2—C14—C11	122.78 (18)
C1—C6—H6	119.5	N2—C14—H14	118.6
N1—C7—C2	121.63 (19)	C11—C14—H14	118.6

O1—C1—C2—C3	179.22 (19)	C7—N1—C8—C9	179.5 (2)
C6—C1—C2—C3	−0.5 (3)	C7—N1—C8—C13	−0.4 (3)
O1—C1—C2—C7	0.1 (3)	C13—C8—C9—C10	0.0 (3)
C6—C1—C2—C7	−179.5 (2)	N1—C8—C9—C10	−179.95 (19)
C1—C2—C3—C4	−0.3 (3)	C8—C9—C10—C11	1.3 (3)
C7—C2—C3—C4	178.75 (19)	C9—C10—C11—C12	−1.6 (3)
C2—C3—C4—C5	1.0 (3)	C9—C10—C11—C14	−179.44 (19)
C2—C3—C4—Br1	−177.75 (16)	C10—C11—C12—C13	0.6 (3)
C3—C4—C5—C6	−1.0 (3)	C14—C11—C12—C13	178.34 (19)
Br1—C4—C5—C6	177.87 (17)	C11—C12—C13—C8	0.7 (3)
C4—C5—C6—C1	0.1 (3)	C9—C8—C13—C12	−1.0 (3)
O1—C1—C6—C5	−179.1 (2)	N1—C8—C13—C12	178.95 (19)

C2—C1—C6—C5	0.5 (3)	O2—N2—C14—C11	179.74 (18)
C8—N1—C7—C2	179.75 (19)	C10—C11—C14—N2	−171.1 (2)
C3—C2—C7—N1	−175.3 (2)	C12—C11—C14—N2	11.2 (3)
C1—C2—C7—N1	3.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.87 (2)	1.84 (2)	2.593 (2)	143 (2)
O2—H2···N2^{i}	0.75 (3)	2.08 (3)	2.830 (2)	173 (2)
C5—H5···O2^{ii}	0.95	2.54	3.463 (3)	163

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