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4-Bromo-2-*{(E)-3-[1-(hydroxyimino)-ethyl]phenyliminomethyl}phenol*

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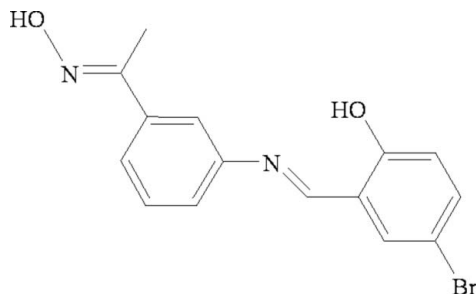
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.039; wR factor = 0.100; data-to-parameter ratio = 13.7.

In the title compound, $\text{C}_{15}\text{H}_{13}\text{BrN}_2\text{O}_2$, the oxime unit adopts an *E* conformation with respect to the O—H group. A classical intramolecular O—H...N hydrogen bond results in the formation of a six-membered ring. The crystal structure is stabilized by intermolecular O—H...N hydrogen bonds between the hydroxy groups and the oxime N atoms. In addition, the crystal structure also features short intermolecular Br...Br short contacts with a distance of 3.8768 (5) Å.

Related literature

For background to Schiff bases, see: Dong *et al.* (2007, 2008); Wang *et al.* (2009). For background to oximes, see: Golovnia *et al.* (2009); Liu *et al.* (2008); Dong *et al.* (2009a); Öztürk *et al.* (2009). For the synthesis, see: Dong *et al.* (2009b).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{13}\text{BrN}_2\text{O}_2$
 $M_r = 333.18$
Monoclinic, $P2_1/c$
 $a = 17.020$ (2) Å
 $b = 6.1676$ (7) Å

$c = 13.693$ (1) Å
 $\beta = 96.461$ (1)°
 $V = 1428.3$ (3) Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 2.88$ mm⁻¹
 $T = 298$ K

0.45 × 0.20 × 0.10 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.357$, $T_{\text{max}} = 0.762$

6906 measured reflections
2492 independent reflections
1799 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.051$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.100$
 $S = 0.99$
2492 reflections

182 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.47$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³

Table 1

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 ¹	0.82	2.10	2.830 (4)	149
O2—H2...N2	0.82	1.91	2.635 (4)	147

Symmetry code: (i) $-x, -y + 3, -z + 1$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008) and DIAMOND (Brandenburg, 1998); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2124).

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supplementary materials

Acta Cryst. (2009). E65, o3154 [doi:10.1107/S1600536809048855]

4-Bromo-2-*{(E)-3-[1-(hydroxyimino)ethyl]phenyliminomethyl}*phenol

L. Xu and L. Wu

Comment

Schiff base ligands containing oxygen and imine nitrogen atoms have attracted much attention due to their variety of applications as well as their strong coordination capability (Dong *et al.*, 2007; Dong *et al.*, 2008; Wang *et al.*, 2009). The oxime compounds frequently exhibit versatility in organic, inorganic, bioinorganic, pigment, analytical, dyes and medical chemistry (Golovnia *et al.*, 2009; Liu *et al.*, 2008; Dong *et al.*, 2009a; Öztürk *et al.*, 2009). Owing to the importance of oxime-type compounds, we report the crystal structure of the title compound (Fig. 1).

In the crystal structure, all bond lengths and bond angles are in normal ranges. The molecule has a crystallographic inversion centre and the oxime unit adopts an E conformation with respect to the O—H group. The aniline ring (C3—C8) and phenol ring (C10—C15) are almost parallel each other, making a dihedral angle of 2.71 (1)°. The torsion angles of O1—N1—C2—C3 and C5—N2—C9—C10 are 178.5 (3) and -178.8 (3)°, respectively. In the crystal structure, a classical intramolecular O—H···N hydrogen bond forms a six-membered ring (Fig. 2 and Table 1). The crystal packing (Fig. 2) is stabilized by intermolecular O—H···N hydrogen bonds between the hydroxy groups and oxime N atoms, with a O1—H1···N1ⁱ (Table 1). In addition, the crystal structure was further stabilized by weak intermolecular Br···Brⁱⁱ (Fig. 2) short interactions with a distance of 3.8768 (5) Å.

Experimental

The title compound was synthesized according to an analogous method reported earlier (Dong *et al.*, 2009b). To an ethanol solution (5 ml) of 3-aminophenylethanone oxime (150.2 mg, 1.00 mmol) was added dropwise an ethanol solution (5 ml) of 5-bromosalicylaldehyde (201.1 mg, 1.00 mmol) then the yellow precipitate was obtained. The mixture solution was stirred at 328–333 K for 1 h. After cooling to room temperature, the precipitate was filtered off, dried *in vacuo* and purified by recrystallization from ethanol of solid. Yield: 54.01%, m. p. 459–461 K. Anal. Calc. for C₁₅H₁₃BrN₂O₂: C, 54.07; H, 3.93; N, 8.41. Found: C, 54.32; H, 4.01; N, 8.81.

Yellow needle-like single crystals suitable for X-ray diffraction studies were obtained by slow evaporation from a solution of dichloromethane at room temperature for about four weeks.

Refinement

Non-H atoms were refined anisotropically. H atoms were treated as riding atoms with distances C—H = 0.96 Å (CH₃), 0.93 Å (CH), O—H = 0.82 Å for (OH). The isotropic displacement parameters for all H atoms were set equal to 1.2 or 1.5 *U*_{eq} of the carrier atom.

Figures

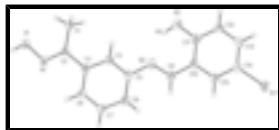


Fig. 1. The molecule structure of the title compound with atom numbering. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

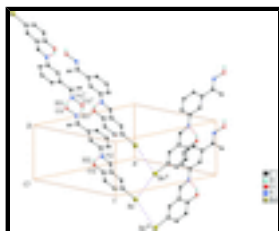


Fig. 2. O—H...N and Br...Br interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) $-x + 1, y + 1/2, -z + 3/2$; (ii) $-x + 1, y - 1/2, -z + 3/2$; (iii) $-x, -y + 3, -z + 1$.]

4-Bromo-2-*{(E)-3-[1-(hydroxyimino)ethyl]phenyliminomethyl}*phenol

Crystal data

$C_{15}H_{13}BrN_2O_2$

$M_r = 333.18$

Monoclinic, $P2_1/c$

Hall symbol: $-p\ 2ybc$

$a = 17.020$ (2) Å

$b = 6.1676$ (7) Å

$c = 13.693$ (1) Å

$\beta = 96.461$ (1)°

$V = 1428.3$ (3) Å³

$Z = 4$

$F_{000} = 672$

$D_x = 1.549$ Mg m⁻³

Melting point = 459–461 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2173 reflections

$\theta = 2.7$ – 23.8 °

$\mu = 2.88$ mm⁻¹

$T = 298$ K

Needle-like, yellow

$0.45 \times 0.20 \times 0.10$ mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Detector resolution: 10.0 pixels mm⁻¹

$T = 298$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.357, T_{\max} = 0.762$

6906 measured reflections

2492 independent reflections

1799 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.051$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.4$ °

$h = -20 \rightarrow 14$

$k = -7 \rightarrow 7$

$l = -15 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$wR(F^2) = 0.100$	$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2]$
$S = 0.99$	where $P = (F_o^2 + 2F_c^2)/3$
2492 reflections	$(\Delta/\sigma)_{\max} < 0.001$
182 parameters	$\Delta\rho_{\max} = 0.47 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

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.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br	0.46909 (2)	-0.34895 (6)	0.66885 (3)	0.05662 (19)
N1	0.04843 (18)	1.3049 (4)	0.46740 (19)	0.0451 (7)
N2	0.25065 (15)	0.4933 (4)	0.52712 (19)	0.0416 (7)
O1	0.01134 (16)	1.4311 (4)	0.38885 (16)	0.0609 (8)
H1	-0.0096	1.5373	0.4110	0.091*
O2	0.29339 (17)	0.2760 (5)	0.37605 (18)	0.0688 (8)
H2	0.2718	0.3726	0.4044	0.103*
C1	0.0818 (3)	1.0731 (7)	0.3317 (2)	0.0601 (11)
H1A	0.0385	1.1454	0.2940	0.090*
H1B	0.0753	0.9191	0.3247	0.090*
H1C	0.1306	1.1160	0.3084	0.090*
C2	0.0833 (2)	1.1340 (5)	0.4385 (2)	0.0355 (8)
C3	0.12600 (18)	0.9934 (5)	0.5170 (2)	0.0328 (7)
C4	0.16790 (19)	0.8077 (5)	0.4921 (2)	0.0361 (8)
H4	0.1683	0.7723	0.4261	0.043*
C5	0.2089 (2)	0.6749 (5)	0.5637 (2)	0.0372 (8)
C6	0.2088 (2)	0.7269 (6)	0.6638 (2)	0.0457 (9)
H6	0.2355	0.6402	0.7123	0.055*
C7	0.1674 (2)	0.9129 (6)	0.6892 (2)	0.0541 (10)
H7	0.1670	0.9486	0.7551	0.065*
C8	0.1272 (2)	1.0439 (6)	0.6175 (2)	0.0439 (9)
H8	0.1007	1.1664	0.6362	0.053*
C9	0.2887 (2)	0.3533 (5)	0.5848 (2)	0.0402 (8)
H9	0.2886	0.3675	0.6524	0.048*
C10	0.33200 (19)	0.1729 (5)	0.5462 (2)	0.0370 (8)

supplementary materials

C11	0.3322 (2)	0.1382 (6)	0.4443 (3)	0.0455 (9)
C12	0.3730 (2)	-0.0397 (6)	0.4104 (3)	0.0554 (10)
H12	0.3726	-0.0625	0.3432	0.067*
C13	0.4141 (2)	-0.1828 (6)	0.4770 (3)	0.0520 (10)
H13	0.4413	-0.3000	0.4544	0.062*
C14	0.4138 (2)	-0.1477 (5)	0.5778 (2)	0.0391 (8)
C15	0.37336 (19)	0.0263 (5)	0.6123 (2)	0.0393 (8)
H15	0.3735	0.0467	0.6797	0.047*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0571 (3)	0.0476 (3)	0.0643 (3)	0.01472 (19)	0.0030 (2)	0.00954 (19)
N1	0.056 (2)	0.0395 (17)	0.0372 (15)	0.0112 (15)	-0.0063 (14)	0.0022 (13)
N2	0.0357 (17)	0.0356 (17)	0.0537 (17)	0.0027 (13)	0.0058 (14)	0.0020 (13)
O1	0.083 (2)	0.0526 (16)	0.0436 (13)	0.0295 (15)	-0.0089 (13)	0.0039 (12)
O2	0.078 (2)	0.079 (2)	0.0479 (14)	0.0289 (17)	0.0001 (15)	0.0108 (14)
C1	0.081 (3)	0.062 (2)	0.0367 (19)	0.028 (2)	0.0017 (19)	-0.0001 (18)
C2	0.036 (2)	0.0329 (19)	0.0373 (17)	-0.0002 (15)	0.0027 (15)	-0.0020 (14)
C3	0.0309 (19)	0.0315 (18)	0.0353 (16)	0.0001 (14)	0.0005 (14)	0.0005 (14)
C4	0.037 (2)	0.037 (2)	0.0338 (16)	-0.0039 (15)	0.0021 (15)	-0.0024 (14)
C5	0.0311 (19)	0.0339 (19)	0.0462 (19)	-0.0012 (14)	0.0031 (15)	0.0022 (15)
C6	0.051 (2)	0.043 (2)	0.043 (2)	0.0113 (18)	0.0038 (17)	0.0141 (16)
C7	0.071 (3)	0.058 (2)	0.0331 (18)	0.014 (2)	0.0074 (18)	0.0027 (17)
C8	0.052 (2)	0.040 (2)	0.0395 (18)	0.0097 (17)	0.0060 (17)	0.0024 (16)
C9	0.040 (2)	0.0346 (19)	0.0455 (19)	-0.0010 (16)	0.0020 (16)	-0.0017 (16)
C10	0.0321 (19)	0.0332 (19)	0.0443 (18)	-0.0013 (15)	-0.0016 (15)	0.0004 (15)
C11	0.037 (2)	0.050 (2)	0.048 (2)	0.0033 (17)	0.0018 (17)	0.0052 (18)
C12	0.057 (3)	0.070 (3)	0.0408 (19)	0.012 (2)	0.0101 (18)	-0.0030 (19)
C13	0.051 (3)	0.051 (2)	0.055 (2)	0.0117 (18)	0.0109 (19)	-0.0053 (18)
C14	0.034 (2)	0.0331 (19)	0.050 (2)	-0.0024 (16)	0.0022 (16)	0.0017 (15)
C15	0.038 (2)	0.038 (2)	0.0411 (18)	0.0003 (16)	0.0013 (16)	-0.0041 (15)

Geometric parameters (\AA , $^\circ$)

Br—C14	1.928 (3)	C4—H4	0.9300
Br—Br ⁱ	3.8768 (5)	C5—C6	1.409 (5)
Br—Br ⁱⁱ	3.8768 (5)	C6—C7	1.410 (5)
N1—C2	1.293 (4)	C6—H6	0.9300
N1—O1	1.417 (3)	C7—C8	1.390 (5)
N2—C9	1.294 (4)	C7—H7	0.9300
N2—C5	1.446 (4)	C8—H8	0.9300
O1—H1	0.8200	C9—C10	1.466 (4)
O2—C11	1.377 (4)	C9—H9	0.9300
O2—H2	0.8200	C10—C15	1.411 (4)
C1—C2	1.507 (4)	C10—C11	1.411 (5)
C1—H1A	0.9600	C11—C12	1.405 (5)
C1—H1B	0.9600	C12—C13	1.399 (5)

C1—H1C	0.9600	C12—H12	0.9300
C2—C3	1.504 (4)	C13—C14	1.398 (5)
C3—C8	1.409 (4)	C13—H13	0.9300
C3—C4	1.411 (4)	C14—C15	1.387 (4)
C4—C5	1.401 (4)	C15—H15	0.9300
C14—Br—Br ⁱ	86.56 (10)	C8—C7—C6	121.3 (3)
C14—Br—Br ⁱⁱ	164.58 (10)	C8—C7—H7	119.4
Br ⁱ —Br—Br ⁱⁱ	105.395 (19)	C6—C7—H7	119.4
C2—N1—O1	113.3 (3)	C7—C8—C3	120.9 (3)
C9—N2—C5	122.5 (3)	C7—C8—H8	119.6
N1—O1—H1	109.5	C3—C8—H8	119.6
C11—O2—H2	109.5	N2—C9—C10	121.6 (3)
C2—C1—H1A	109.5	N2—C9—H9	119.2
C2—C1—H1B	109.5	C10—C9—H9	119.2
H1A—C1—H1B	109.5	C15—C10—C11	118.7 (3)
C2—C1—H1C	109.5	C15—C10—C9	119.3 (3)
H1A—C1—H1C	109.5	C11—C10—C9	122.0 (3)
H1B—C1—H1C	109.5	O2—C11—C12	118.3 (3)
N1—C2—C3	116.9 (3)	O2—C11—C10	121.5 (3)
N1—C2—C1	122.8 (3)	C12—C11—C10	120.1 (3)
C3—C2—C1	120.2 (3)	C13—C12—C11	120.4 (3)
C8—C3—C4	117.6 (3)	C13—C12—H12	119.8
C8—C3—C2	121.6 (3)	C11—C12—H12	119.8
C4—C3—C2	120.8 (3)	C14—C13—C12	119.3 (3)
C5—C4—C3	122.0 (3)	C14—C13—H13	120.4
C5—C4—H4	119.0	C12—C13—H13	120.4
C3—C4—H4	119.0	C15—C14—C13	120.9 (3)
C4—C5—C6	119.5 (3)	C15—C14—Br	120.2 (2)
C4—C5—N2	115.8 (3)	C13—C14—Br	118.9 (2)
C6—C5—N2	124.6 (3)	C14—C15—C10	120.5 (3)
C5—C6—C7	118.7 (3)	C14—C15—H15	119.7
C5—C6—H6	120.6	C10—C15—H15	119.7
C7—C6—H6	120.6		
O1—N1—C2—C3	178.5 (3)	N2—C9—C10—C15	179.2 (3)
O1—N1—C2—C1	-1.9 (5)	N2—C9—C10—C11	-2.2 (5)
N1—C2—C3—C8	0.8 (5)	C15—C10—C11—O2	-179.6 (3)
C1—C2—C3—C8	-178.8 (3)	C9—C10—C11—O2	1.8 (5)
N1—C2—C3—C4	-177.9 (3)	C15—C10—C11—C12	0.1 (5)
C1—C2—C3—C4	2.5 (5)	C9—C10—C11—C12	-178.5 (3)
C8—C3—C4—C5	0.6 (5)	O2—C11—C12—C13	179.2 (4)
C2—C3—C4—C5	179.4 (3)	C10—C11—C12—C13	-0.5 (6)
C3—C4—C5—C6	-0.1 (5)	C11—C12—C13—C14	0.4 (6)
C3—C4—C5—N2	-178.6 (3)	C12—C13—C14—C15	0.0 (5)
C9—N2—C5—C4	-177.6 (3)	C12—C13—C14—Br	178.9 (3)
C9—N2—C5—C6	4.0 (5)	Br ⁱ —Br—C14—C15	-39.2 (3)
C4—C5—C6—C7	-0.3 (5)	Br ⁱⁱ —Br—C14—C15	102.3 (4)
N2—C5—C6—C7	178.1 (3)	Br ⁱ —Br—C14—C13	141.9 (3)

supplementary materials

C5—C6—C7—C8	0.0 (6)	Br ⁱⁱ —Br—C14—C13	-76.7 (5)
C6—C7—C8—C3	0.6 (6)	C13—C14—C15—C10	-0.4 (5)
C4—C3—C8—C7	-0.9 (5)	Br—C14—C15—C10	-179.3 (2)
C2—C3—C8—C7	-179.6 (3)	C11—C10—C15—C14	0.3 (5)
C5—N2—C9—C10	-178.8 (3)	C9—C10—C15—C14	179.0 (3)

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 ⁱⁱⁱ —N1 ⁱⁱⁱ	0.82	2.10	2.830 (4)	149
O2—H2 ⁱⁱⁱ —N2	0.82	1.91	2.635 (4)	147

Symmetry codes: (iii) $-x, -y+3, -z+1$.

Fig. 1

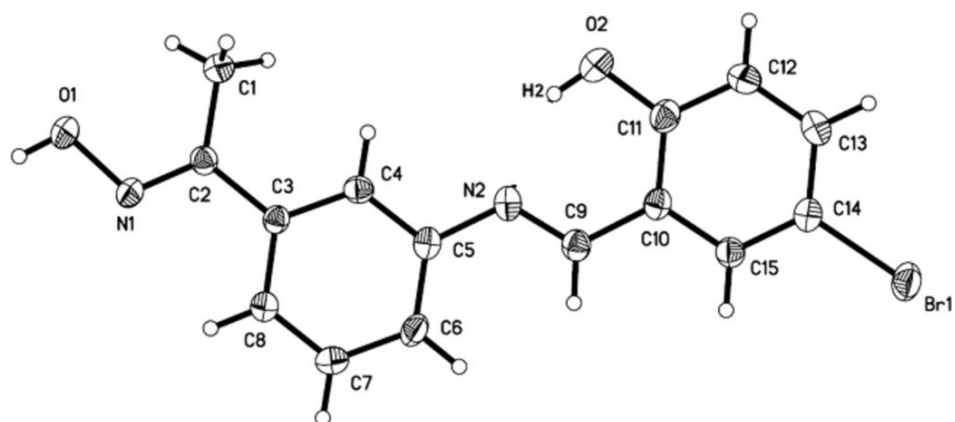


Fig. 2

