organic compounds

(E)-4-Bromo-2-[2-(hydroxymethyl)-phenyliminomethyl]phenol

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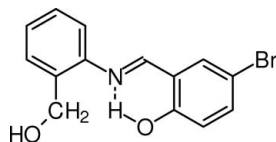Correspondence e-mail: yavuzk@omu.edu.tr

The title compound, $C_{14}H_{12}BrNO_2$, adopt the phenol–imine tautomeric form. The dihedral angle between the aromatic rings is 27.49 (2)°. In the structure, there are O—H⋯O intermolecular hydrogen bonds and O—H⋯N and C—H⋯O intramolecular hydrogen bonds. The phenol H atom forms a strong intramolecular hydrogen bond with the imine N atom.

Related literature

For related literature, see: Özek *et al.*, 2007; Moustakali-Mavridis, Hadjoudis & Mavridis (1978).

Experimental

Crystal data

$C_{14}H_{12}BrNO_2$	V = 1264.5 (4) Å^3
M_r = 306.16	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 6.4356 (13) Å	μ = 3.24 mm^{-1}
b = 4.6361 (5) Å	T = 293 (2) K
c = 42.693 (8) Å	$0.59 \times 0.32 \times 0.03$ mm
β = 96.904 (16)°	

Data collection

Stoe IPDSII diffractometer	8887 measured reflections
Absorption correction: integration (X-$RED32$; Stoe & Cie, 2002)	2418 independent reflections
T_{min} = 0.435, T_{max} = 0.827	1281 reflections with $I > 2\sigma(I)$
	R_{int} = 0.158

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.083	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.164	
S = 0.99	
2418 reflections	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
167 parameters	$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1⋯N1	0.84 (6)	1.86 (6)	2.638 (7)	155 (7)
C12—H12⋯O2	0.93	2.46	2.807 (8)	102
O2—H2⋯O2^i	0.82	1.97	2.765 (10)	163

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2117).

References

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Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

supplementary materials

(*E*)-4-Bromo-2-[2-(hydroxymethyl)phenyliminomethyl]phenol

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Comment

The H atom in the compound is located on atom O1 thus confirming a preference for the phenol-imine tautomer in the solid state, it is consisted with the related structure (Özek *et al.*, 2007). It is known that schiff bases may exhibit thermo-chromism depending on the planarity or non-planarity, respectively (Moustakali-Mavridis *et al.*, 1978). The title compound stabilezed by inter and intramolecular hydrogen bonds, nameyl, O2—H2···O2 (symmetry code: −*x* + 1,-*y*,-*z*), O1—H1···N1 and C12—H12···O2.

Experimental

The compound (*E*)-2-[(2-Hydroxymethylphenylimino)methyl]-4-bromophenol was prepared by reflux a mixture of a solution containing 5-bromosalicylaldehyde (0.05 g 0.25 mmol) in 20 ml e thanol and a solution containing 2-Hydroxymethyl-aniline (0.03 g 0.25 mmol) in 20 ml e thanol. The reaction mixture was stirred for 1 h under reflux. The crystals of (*E*)-2-[(2-Hydroxymethylphenylimino)methyl]-4-bromophenol suitable for X-ray analysis were obtained from ethylalcohol by slow evaporation (yield % 61; m.p.387–389°K).

Refinement

For the title compound(I), the structure was solved by direct methods and refined by full-matrix least-square techniques. All H atoms were located geometrically and refined using a riding model, except for atom H1 bonded to atom O1, fixing the aromatic C—H distance at 0.93 Å, the C—H_2 distance 0.97 Å. The intensity data collected for the title structure are generally weak, so the *R*-int lies outside the normal range.
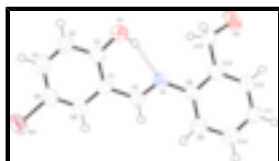
Figures

Fig. 1. An *ORTEP* view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.

(*E*)-4-Bromo-2-[2-(hydroxymethyl)phenyliminomethyl]phenol

Crystal data

$C_{14}H_{12}BrNO_2$	$F_{000} = 616$
$M_r = 306.16$	$D_x = 1.608$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å

supplementary materials

Hall symbol: -P 2ybc

$a = 6.4356\ (13)$ Å

$b = 4.6361\ (5)$ Å

$c = 42.693\ (8)$ Å

$\beta = 96.904\ (16)°$

$V = 1264.5\ (4)$ Å^3

$Z = 4$

Cell parameters from 7218 reflections

$\theta = 3.2–29.3°$

$\mu = 3.24$ mm^{-1}

$T = 293\ (2)$ K

Plate, brown

$0.59 \times 0.32 \times 0.03$ mm

Data collection

STOE IPDS 2 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 6.67 pixels mm^{-1}

$T = 293(2)$ K

ω scan

Absorption correction: integration (X-RED32; Stoe & Cie, 2002)

$T_{min} = 0.435$, $T_{max} = 0.827$

8887 measured reflections

2418 independent reflections

1281 reflections with $I > 2\sigma(I)$

$R_{int} = 0.158$

$\theta_{max} = 26.0°$

$\theta_{min} = 3.2°$

$h = -7 \rightarrow 7$

$k = -5 \rightarrow 5$

$l = -52 \rightarrow 52$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.083$

$wR(F^2) = 0.164$

$S = 0.99$

2418 reflections

167 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.37$ e Å^{-3}

$\Delta\rho_{min} = -0.45$ e Å^{-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$^2 > 2$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	$U_{iso}*/U_{eq}$
C1	0.3539 (8)	0.5546 (12)	0.15615 (15)	0.0442 (15)
C2	0.5621 (8)	0.6418 (13)	0.15407 (17)	0.0499 (16)
C3	0.6531 (9)	0.8446 (15)	0.17555 (18)	0.0569 (19)
H3	0.7895	0.9046	0.1741	0.068*
C4	0.5485 (10)	0.9571 (15)	0.19847 (19)	0.0622 (18)
H4	0.6127	1.0933	0.2124	0.075*
C5	0.3446 (10)	0.8679 (14)	0.20100 (17)	0.0527 (17)
C6	0.2488 (9)	0.6722 (13)	0.17980 (16)	0.0471 (15)
H6	0.1113	0.6174	0.1813	0.056*
C7	0.2448 (8)	0.3595 (12)	0.13302 (16)	0.0422 (14)
H7	0.1063	0.3119	0.1348	0.051*
C8	0.2096 (8)	0.0736 (12)	0.08767 (15)	0.0413 (14)
C9	0.0395 (9)	−0.0889 (12)	0.09518 (17)	0.0499 (16)
H9	0.0003	−0.0846	0.1154	0.060*
C10	−0.0702 (9)	−0.2571 (13)	0.07177 (19)	0.0546 (18)
H10	−0.1835	−0.3665	0.0765	0.066*
C11	−0.0134 (9)	−0.2637 (13)	0.04185 (18)	0.0540 (18)
H11	−0.0887	−0.3762	0.0264	0.065*
C12	0.1539 (9)	−0.1049 (12)	0.03464 (17)	0.0496 (16)
H12	0.1916	−0.1122	0.0143	0.060*
C13	0.2691 (8)	0.0679 (11)	0.05727 (16)	0.0419 (15)
C14	0.4530 (8)	0.2478 (13)	0.04907 (17)	0.0497 (16)
H14A	0.4368	0.4441	0.0562	0.060*
H14B	0.5813	0.1712	0.0603	0.060*
N1	0.3306 (7)	0.2525 (10)	0.11061 (12)	0.0424 (12)
O1	0.6734 (7)	0.5367 (11)	0.13205 (13)	0.0666 (14)
O2	0.4709 (8)	0.2513 (10)	0.01635 (13)	0.0743 (15)
H2	0.4878	0.0863	0.0102	0.111*
Br1	0.19493 (12)	1.02961 (17)	0.232551 (19)	0.0751 (4)
H1	0.589 (9)	0.427 (13)	0.1214 (17)	0.05 (2)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.042 (3)	0.045 (3)	0.045 (4)	0.000 (3)	0.001 (3)	0.004 (3)
C2	0.043 (3)	0.059 (4)	0.048 (5)	0.002 (3)	0.008 (3)	0.000 (4)
C3	0.044 (3)	0.067 (4)	0.059 (5)	−0.005 (3)	0.004 (3)	−0.017 (4)
C4	0.064 (4)	0.060 (4)	0.059 (5)	−0.007 (4)	−0.006 (4)	−0.001 (4)
C5	0.065 (4)	0.049 (4)	0.043 (4)	0.012 (3)	0.002 (3)	0.001 (3)
C6	0.049 (3)	0.048 (3)	0.045 (4)	0.004 (3)	0.007 (3)	0.001 (3)
C7	0.040 (3)	0.045 (3)	0.043 (4)	−0.007 (3)	0.011 (3)	0.003 (3)
C8	0.038 (3)	0.043 (3)	0.044 (4)	0.002 (2)	0.008 (3)	0.003 (3)
C9	0.046 (3)	0.054 (4)	0.051 (4)	−0.004 (3)	0.011 (3)	0.002 (3)
C10	0.046 (3)	0.052 (4)	0.065 (5)	−0.009 (3)	0.004 (3)	0.005 (4)

C11	0.053 (3)	0.057 (4)	0.050 (5)	−0.010 (3)	−0.005 (3)	−0.006 (4)
C12	0.056 (3)	0.049 (3)	0.043 (4)	0.002 (3)	0.004 (3)	−0.007 (3)
C13	0.039 (3)	0.039 (3)	0.048 (4)	0.004 (2)	0.006 (3)	−0.001 (3)
C14	0.049 (3)	0.049 (3)	0.052 (5)	−0.001 (3)	0.011 (3)	−0.003 (3)
N1	0.045 (2)	0.046 (3)	0.036 (3)	0.001 (2)	0.005 (2)	0.000 (3)
O1	0.051 (2)	0.083 (3)	0.069 (4)	−0.013 (3)	0.022 (2)	−0.023 (3)
O2	0.108 (4)	0.071 (3)	0.050 (4)	−0.024 (3)	0.039 (3)	−0.014 (3)
Br1	0.1005 (6)	0.0744 (5)	0.0545 (5)	0.0018 (4)	0.0262 (4)	−0.0133 (4)

Geometric parameters (Å, °)

C1—C6	1.392 (9)	C8—N1	1.439 (7)
C1—C2	1.412 (8)	C9—C10	1.391 (9)
C1—C7	1.456 (8)	C9—H9	0.9300
C2—O1	1.340 (8)	C10—C11	1.371 (10)
C2—C3	1.392 (9)	C10—H10	0.9300
C3—C4	1.357 (10)	C11—C12	1.370 (8)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.392 (9)	C12—C13	1.397 (8)
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.374 (9)	C13—C14	1.523 (8)
C5—Br1	1.902 (7)	C14—O2	1.415 (8)
C6—H6	0.9300	C14—H14A	0.9700
C7—N1	1.262 (8)	C14—H14B	0.9700
C7—H7	0.9300	O1—H1	0.84 (6)
C8—C9	1.398 (8)	O2—H2	0.8200
C8—C13	1.396 (9)		
C6—C1—C2	118.7 (5)	C10—C9—C8	118.8 (7)
C6—C1—C7	119.9 (5)	C10—C9—H9	120.6
C2—C1—C7	121.3 (6)	C8—C9—H9	120.6
O1—C2—C3	119.3 (5)	C11—C10—C9	120.9 (6)
O1—C2—C1	122.1 (6)	C11—C10—H10	119.6
C3—C2—C1	118.6 (6)	C9—C10—H10	119.6
C4—C3—C2	121.9 (6)	C10—C11—C12	120.2 (6)
C4—C3—H3	119.0	C10—C11—H11	119.9
C2—C3—H3	119.0	C12—C11—H11	119.9
C3—C4—C5	119.7 (6)	C11—C12—C13	121.2 (7)
C3—C4—H4	120.2	C11—C12—H12	119.4
C5—C4—H4	120.2	C13—C12—H12	119.4
C6—C5—C4	119.8 (7)	C12—C13—C8	118.2 (5)
C6—C5—Br1	120.1 (5)	C12—C13—C14	121.1 (6)
C4—C5—Br1	120.0 (5)	C8—C13—C14	120.7 (5)
C5—C6—C1	121.2 (6)	O2—C14—C13	113.0 (5)
C5—C6—H6	119.4	O2—C14—H14A	109.0
C1—C6—H6	119.4	C13—C14—H14A	109.0
N1—C7—C1	122.6 (5)	O2—C14—H14B	109.0
N1—C7—H7	118.7	C13—C14—H14B	109.0
C1—C7—H7	118.7	H14A—C14—H14B	107.8
C9—C8—C13	120.8 (6)	C7—N1—C8	119.5 (5)

C9—C8—N1	121.9 (6)	C2—O1—H1	103 (5)
C13—C8—N1	117.2 (5)	C14—O2—H2	109.5
C6—C1—C2—O1	179.6 (6)	N1—C8—C9—C10	−180.0 (5)
C7—C1—C2—O1	−4.2 (9)	C8—C9—C10—C11	−0.1 (9)
C6—C1—C2—C3	−0.7 (9)	C9—C10—C11—C12	0.4 (9)
C7—C1—C2—C3	175.5 (6)	C10—C11—C12—C13	−0.5 (9)
O1—C2—C3—C4	−179.6 (7)	C11—C12—C13—C8	0.3 (8)
C1—C2—C3—C4	0.8 (10)	C11—C12—C13—C14	−179.1 (5)
C2—C3—C4—C5	0.4 (11)	C9—C8—C13—C12	−0.1 (8)
C3—C4—C5—C6	−1.7 (10)	N1—C8—C13—C12	179.9 (4)
C3—C4—C5—Br1	−178.9 (6)	C9—C8—C13—C14	179.3 (5)
C4—C5—C6—C1	1.7 (9)	N1—C8—C13—C14	−0.7 (7)
Br1—C5—C6—C1	178.9 (5)	C12—C13—C14—O2	8.7 (8)
C2—C1—C6—C5	−0.5 (9)	C8—C13—C14—O2	−170.7 (5)
C7—C1—C6—C5	−176.7 (6)	C1—C7—N1—C8	−176.7 (5)
C6—C1—C7—N1	178.1 (6)	C9—C8—N1—C7	−28.5 (8)
C2—C1—C7—N1	2.0 (9)	C13—C8—N1—C7	151.5 (5)
C13—C8—C9—C10	0.0 (8)		

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H1⋯N1	0.84 (6)	1.86 (6)	2.638 (7)	155 (7)
C12—H12⋯O2	0.93	2.46	2.807 (8)	102
O2—H2⋯O2[i]	0.82	1.97	2.765 (10)	163

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