

2,4-Dibromo-6-(4-bromophenylimino-methyl)phenol

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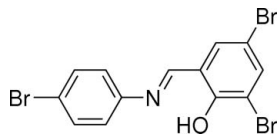
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 Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.014$ Å; R factor = 0.066; wR factor = 0.199; data-to-parameter ratio = 14.2.

In the title compound, $\text{C}_{13}\text{H}_8\text{Br}_3\text{NO}$, the two aromatic substituents lie *trans* to each other across the $\text{C}=\text{N}$ bond. The molecule is almost planar, with a dihedral angle of 3.6 (5)° between the aromatic rings. Intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonding generates an $S(6)$ ring motif, while short intermolecular $\text{C}-\text{H}\cdots\text{O}$, $\text{C}-\text{H}\cdots\text{Br}$ and $\text{Br}\cdots\text{Br}$ contacts [$\text{Br}\cdots\text{Br}$ distance 3.669 (2) Å] link the molecules into a two-dimensional network.

Related literature

For the background to Schiff base chemistry, see: Yeap *et al.* (2003) and for related structures, see: Zheng *et al.* (2005); Özek *et al.* (2007); Guo (2007).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_8\text{Br}_3\text{NO}$
 $M_r = 433.90$

 Triclinic, $P\bar{1}$
 $a = 7.985$ (3) Å

 $b = 8.594$ (3) Å

 $c = 11.020$ (4) Å

 $\alpha = 87.801$ (6)°

 $\beta = 76.688$ (7)°

 $\gamma = 65.180$ (5)°

 $V = 666.5$ (4) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 9.07$ mm⁻¹
 $T = 294$ (2) K

 $0.24 \times 0.16 \times 0.12$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

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

 $T_{\min} = 0.202$, $T_{\max} = 0.339$

 3322 measured reflections
 2309 independent reflections
 1471 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.065$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.199$
 $S = 0.98$

2309 reflections

163 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.86$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.27$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.93	2.574 (10)	135
$\text{C10}-\text{H10}\cdots\text{O1}^{\dagger}$	0.93	2.61	3.341 (13)	136
$\text{C10}-\text{H10}\cdots\text{Br1}^{\dagger}$	0.93	2.99	3.866 (10)	157

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: SJ2397).

References

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supplementary materials

Acta Cryst. (2007). E63, o4558 [doi:10.1107/S1600536807054335]

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Comment

Schiff bases are important in diverse fields of chemistry and biochemistry owing to their biological activity, photochromism and related properties (Yeap *et al.*, 2003). In view of the importance and also the usefulness of these compounds, chemists are prompted to generate the new derivatives by introducing different substituents into the existing skeleton of the molecule (Zheng *et al.*, 2005; Özek *et al.*, 2007; Guo, 2007). Here, we report the structure of the title compound, (I), Fig. 1, a new Schiff base, which was prepared by reaction of 3,5-dibromo-2-hydroxybenzaldehyde with 4-bromobenzenamine. The planarity of the molecule is supported by the conjugation of the imino group and the aromatic system, together with a resonance-assisted intramolecular O1—H1 \cdots N1 hydrogen bond (Table 1). The dihedral angle between the aromatic rings is 3.6 (5)°.

In addition to the intramolecular hydrogen bond, some short intermolecular contacts, C10—H10 \cdots Oⁱ (see Table 1 for symmetry codes), C10—H10 \cdots Brⁱ and Br3 \cdots Br2ⁱⁱ ($d[\text{Br2—Br3}] = 3.669(2) \text{ \AA}$, symmetry code: (ii) $1 + x, -1 + y, 1 + z$), were observed in the crystal structure. These link the molecules into a two-dimensional network, Fig. 2.

Experimental

The title compound, (I), was prepared by reaction of 3,5-dibromo-2-hydroxybenzaldehyde (1.4 g, 5 mmol) with 4-bromobenzenamine (0.95 g 5.5 mol) in 30 ml of 95% ethanol. The mixture was stirred and heated in air at reflux temperature for 30 min, after which 40 ml distilled water was added, the resulting product was separated by filtration (1.8 g, yield 83.1%). The pure product (0.5 g) was heated and dissolved in 15 ml of 95% ethanol. Single crystals were obtained from this solution by slow evaporation over a period of 2 days at room temperature.

Refinement

The H atom involved in the O—H \cdots N hydrogen bond was found in a difference Fourier map, but was fixed during refinement with $d(\text{O—H}) = 0.82 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{O})$. H atoms bound to C atoms were included in the riding model approximation, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C atom})$.

Figures

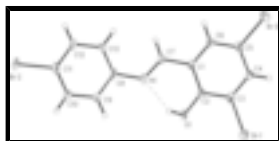


Fig. 1. A view of the structure of (I), showing the atom-numbering Scheme and the O1—H1 \cdots N1 hydrogen bond (dashed line); displacement ellipsoids were drawn at the 30% probability level.



Fig. 2. Packing diagram of (I) viewed down the *c* axis, showing hydrogen bonds and short intermolecular contacts as dashed lines.

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Crystal data

$C_{13}H_8Br_3NO$	$Z = 2$
$M_r = 433.90$	$F_{000} = 412$
Triclinic, $P\bar{1}$	$D_x = 2.162 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 7.985 (3) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.594 (3) \text{ \AA}$	Cell parameters from 1206 reflections
$c = 11.020 (4) \text{ \AA}$	$\theta = 2.9\text{--}26.2^\circ$
$\alpha = 87.801 (6)^\circ$	$\mu = 9.07 \text{ mm}^{-1}$
$\beta = 76.688 (7)^\circ$	$T = 294 (2) \text{ K}$
$\gamma = 65.180 (5)^\circ$	Prism, red
$V = 666.5 (4) \text{ \AA}^3$	$0.24 \times 0.16 \times 0.12 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	2309 independent reflections
Radiation source: fine-focus sealed tube	1471 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.065$
$T = 294(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 9$
$T_{\text{min}} = 0.202, T_{\text{max}} = 0.339$	$k = -6 \rightarrow 10$
3322 measured reflections	$l = -12 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.066$	H-atom parameters constrained
$wR(F^2) = 0.199$	$w = 1/[\sigma^2(F_o^2) + (0.1123P)^2]$
$S = 0.98$	where $P = (F_o^2 + 2F_c^2)/3$
2309 reflections	$(\Delta/\sigma)_{\text{max}} = <0.001$
163 parameters	$\Delta\rho_{\text{max}} = 0.86 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -1.27 \text{ e \AA}^{-3}$
	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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.12496 (17)	0.33749 (14)	0.12113 (9)	0.0560 (4)
Br2	0.69858 (18)	0.14000 (16)	0.98515 (10)	0.0616 (5)
Br3	-0.19458 (18)	1.03572 (14)	0.29251 (11)	0.0597 (5)
O1	0.2910 (9)	0.2930 (9)	0.3421 (6)	0.0451 (18)
H1	0.2990	0.2748	0.4146	0.054*
N1	0.3470 (10)	0.3870 (10)	0.5425 (7)	0.0350 (19)
C1	0.1501 (12)	0.5864 (12)	0.4244 (8)	0.032 (2)
C2	0.1817 (12)	0.4586 (11)	0.3349 (8)	0.030 (2)
C3	0.0952 (13)	0.5088 (13)	0.2346 (8)	0.035 (2)
C4	-0.0163 (12)	0.6781 (11)	0.2222 (8)	0.030 (2)
H4	-0.0730	0.7103	0.1549	0.036*
C5	-0.0428 (12)	0.8008 (12)	0.3121 (9)	0.034 (2)
C6	0.0394 (13)	0.7551 (12)	0.4103 (8)	0.035 (2)
H6	0.0203	0.8394	0.4690	0.042*
C7	0.2360 (13)	0.5403 (13)	0.5293 (9)	0.037 (2)
H7	0.2093	0.6258	0.5894	0.044*
C8	0.4295 (12)	0.3389 (12)	0.6481 (8)	0.032 (2)
C9	0.5304 (14)	0.1652 (12)	0.6570 (9)	0.041 (2)
H9	0.5436	0.0877	0.5950	0.049*
C10	0.6116 (15)	0.1055 (14)	0.7560 (10)	0.049 (3)
H10	0.6817	-0.0116	0.7603	0.059*
C11	0.5887 (13)	0.2199 (13)	0.8488 (9)	0.042 (3)
C12	0.4891 (15)	0.3947 (14)	0.8395 (9)	0.048 (3)
H12	0.4767	0.4728	0.9009	0.057*
C13	0.4093 (13)	0.4518 (12)	0.7400 (8)	0.037 (2)
H13	0.3405	0.5690	0.7348	0.044*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0849 (8)	0.0393 (7)	0.0331 (6)	-0.0047 (5)	-0.0351 (6)	-0.0061 (5)
Br2	0.0917 (9)	0.0532 (8)	0.0349 (7)	-0.0119 (6)	-0.0432 (6)	0.0077 (5)

supplementary materials

Br3	0.0896 (9)	0.0281 (7)	0.0566 (8)	-0.0077 (6)	-0.0431 (6)	0.0098 (5)
O1	0.061 (4)	0.039 (4)	0.023 (4)	-0.001 (3)	-0.026 (3)	0.003 (3)
N1	0.044 (4)	0.033 (5)	0.028 (4)	-0.011 (4)	-0.022 (3)	0.006 (4)
C1	0.044 (5)	0.034 (5)	0.018 (5)	-0.014 (4)	-0.015 (4)	0.007 (4)
C2	0.039 (5)	0.024 (5)	0.017 (4)	-0.001 (4)	-0.011 (4)	0.000 (4)
C3	0.047 (5)	0.047 (7)	0.017 (5)	-0.020 (5)	-0.016 (4)	0.005 (4)
C4	0.045 (5)	0.030 (5)	0.014 (4)	-0.010 (4)	-0.017 (4)	0.006 (4)
C5	0.044 (5)	0.026 (5)	0.039 (6)	-0.015 (4)	-0.020 (4)	0.016 (4)
C6	0.060 (6)	0.027 (6)	0.025 (5)	-0.018 (5)	-0.022 (4)	0.005 (4)
C7	0.053 (6)	0.035 (6)	0.032 (5)	-0.020 (5)	-0.024 (4)	0.003 (4)
C8	0.037 (5)	0.045 (6)	0.014 (4)	-0.014 (4)	-0.014 (4)	0.007 (4)
C9	0.062 (6)	0.029 (6)	0.036 (6)	-0.015 (5)	-0.030 (5)	0.001 (4)
C10	0.067 (7)	0.031 (6)	0.046 (6)	-0.006 (5)	-0.037 (5)	0.002 (5)
C11	0.050 (6)	0.043 (6)	0.026 (5)	-0.007 (5)	-0.021 (4)	0.005 (4)
C12	0.077 (7)	0.041 (7)	0.032 (6)	-0.022 (6)	-0.034 (5)	0.013 (5)
C13	0.053 (5)	0.021 (5)	0.028 (5)	-0.004 (4)	-0.017 (4)	-0.003 (4)

Geometric parameters (Å, °)

Br1—C3	1.870 (10)	C5—C6	1.354 (12)
Br2—C11	1.876 (9)	C6—H6	0.9300
Br3—C5	1.904 (9)	C7—H7	0.9300
O1—C2	1.332 (11)	C8—C13	1.365 (13)
O1—H1	0.8200	C8—C9	1.379 (13)
N1—C7	1.268 (12)	C9—C10	1.371 (13)
N1—C8	1.431 (10)	C9—H9	0.9300
C1—C6	1.370 (13)	C10—C11	1.373 (14)
C1—C2	1.406 (13)	C10—H10	0.9300
C1—C7	1.437 (12)	C11—C12	1.387 (15)
C2—C3	1.397 (12)	C12—C13	1.367 (13)
C3—C4	1.372 (13)	C12—H12	0.9300
C4—C5	1.390 (13)	C13—H13	0.9300
C4—H4	0.9300		
C2—O1—H1	109.4	N1—C7—H7	118.9
C7—N1—C8	122.6 (8)	C1—C7—H7	118.9
C6—C1—C2	119.8 (8)	C13—C8—C9	119.3 (8)
C6—C1—C7	120.1 (9)	C13—C8—N1	124.5 (8)
C2—C1—C7	120.1 (8)	C9—C8—N1	116.2 (8)
O1—C2—C3	119.2 (8)	C10—C9—C8	120.8 (9)
O1—C2—C1	122.6 (8)	C10—C9—H9	119.6
C3—C2—C1	118.2 (8)	C8—C9—H9	119.6
C4—C3—C2	121.3 (8)	C9—C10—C11	119.6 (10)
C4—C3—Br1	120.5 (6)	C9—C10—H10	120.2
C2—C3—Br1	118.2 (7)	C11—C10—H10	120.2
C3—C4—C5	118.7 (8)	C10—C11—C12	119.8 (9)
C3—C4—H4	120.6	C10—C11—Br2	120.0 (8)
C5—C4—H4	120.6	C12—C11—Br2	120.1 (8)
C6—C5—C4	121.0 (8)	C13—C12—C11	119.8 (10)
C6—C5—Br3	120.6 (8)	C13—C12—H12	120.1

C4—C5—Br3	118.4 (6)	C11—C12—H12	120.1
C5—C6—C1	120.9 (9)	C8—C13—C12	120.8 (9)
C5—C6—H6	119.5	C8—C13—H13	119.6
C1—C6—H6	119.5	C12—C13—H13	119.6
N1—C7—C1	122.2 (9)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	0.82	1.93	2.574 (10)	135
C10—H10 \cdots O1 ⁱ	0.93	2.61	3.341 (13)	136
C10—H10 \cdots Br1 ⁱ	0.93	2.99	3.866 (10)	157

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

Fig. 1

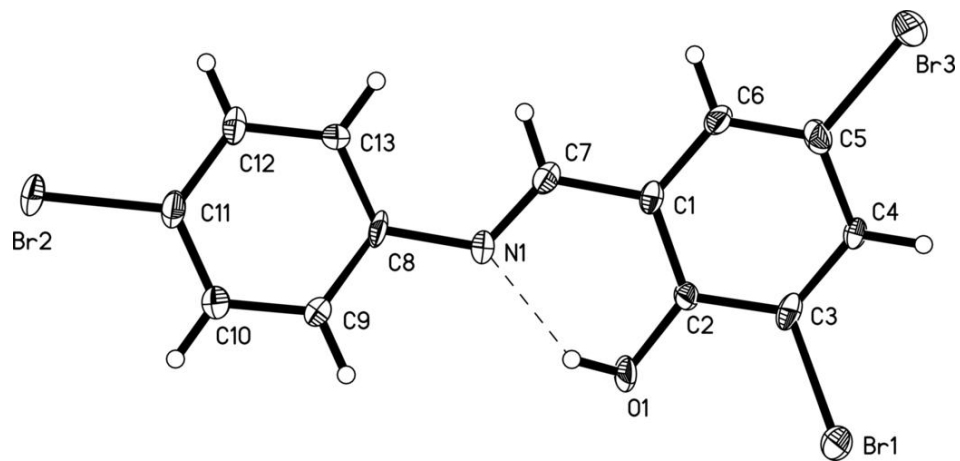


Fig. 2

