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4,6-Dibromo-2-[(*E*)-(4-[(*E*)-3,5-dibromo-2-hydroxybenzylidene]amino}butyl)-iminomethyl]phenol

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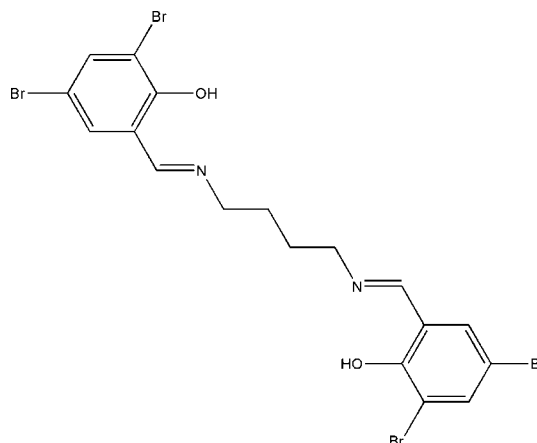
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.035; wR factor = 0.074; data-to-parameter ratio = 18.3.

The asymmetric unit of the title compound, $\text{C}_{18}\text{H}_{16}\text{Br}_4\text{N}_2\text{O}_2$, comprises half the molecule, which is located adjacent to an inversion centre at the mid-point of the central C—C bond of the butane-1,4-diamine segment. There are two intramolecular O—H···N hydrogen bonds making $S(6)$ ring motifs. In the crystal, molecules are linked by pairs of weak C—H···Br interactions into chains along [101], which include $R_2^2(8)$ ring motifs. These chains are further linked by C—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For standard bond lengths, see: Allen *et al.*, (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related Schiff base ligands, see: Kargar *et al.* (2011); Kia *et al.* (2010).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{16}\text{Br}_4\text{N}_2\text{O}_2$
 $M_r = 611.97$
 Orthorhombic, *Pbcn*
 $a = 15.9537$ (12) Å
 $b = 12.8784$ (10) Å
 $c = 9.5566$ (6) Å

$V = 1963.5$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 8.21$ mm⁻¹
 $T = 291$ K
 $0.35 \times 0.14 \times 0.12$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.161$, $T_{\max} = 0.439$

15023 measured reflections
 2164 independent reflections
 1381 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.069$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.074$
 $S = 0.99$
 2164 reflections

118 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.74$ e Å⁻³
 $\Delta\rho_{\min} = -0.57$ 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	0.72	1.92	2.574 (4)	151
C6—H6···O1 ⁱ	0.93	2.56	3.469 (5)	165
C4—H3···Br2 ⁱⁱ	0.93	2.96	3.849 (4)	161

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINTE* (Bruker, 2005); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2461).

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4,6-Dibromo-2-[(*E*)-(4-[(*E*)-3,5-dibromo-2-hydroxybenzylidene]amino)butyl)-iminomethyl]phenol

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S1. Comment

In continuation of our work on the crystal structures of Schiff base ligands (Kargar *et al.*, 2011; Kia *et al.*, 2010), we synthesized and analysed the crystal structure of the title compound.

The asymmetric unit of the title compound, Fig. 1, comprises half of a potential tetradentate Schiff base ligand. The molecule is located about an inversion centre which is situated at the centre of the central C9-C9A bond of the 1,4-butane-diamine segment. The bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges. The intramolecular O—H \cdots N hydrogen bonds make *S*(6) ring motifs (Table 1 and Fig. 1; Bernstein *et al.*, 1995).

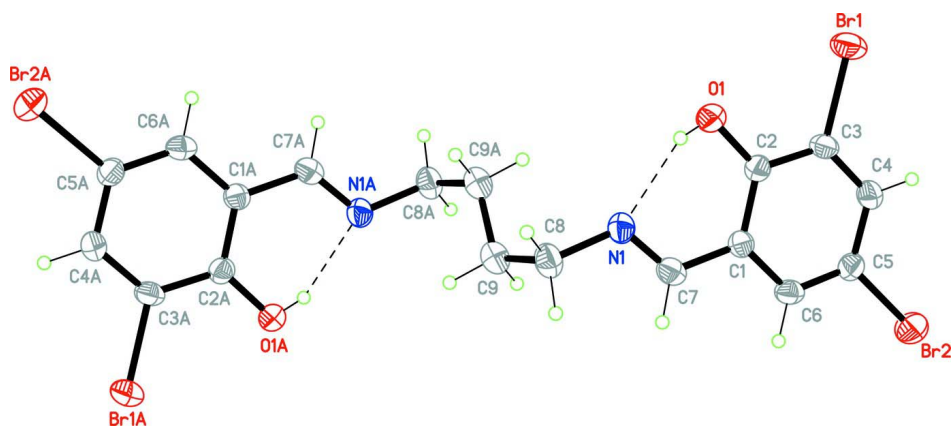
In the crystal, molecules are linked by pairs of weak C—H \cdots Br interactions to form chains along direction [101] which include *R*²₂(8) ring motifs (Table 1 and Fig. 2). These chains are further linked by C—H \cdots O interactions (Table 1) to form a three-dimensional network.

S2. Experimental

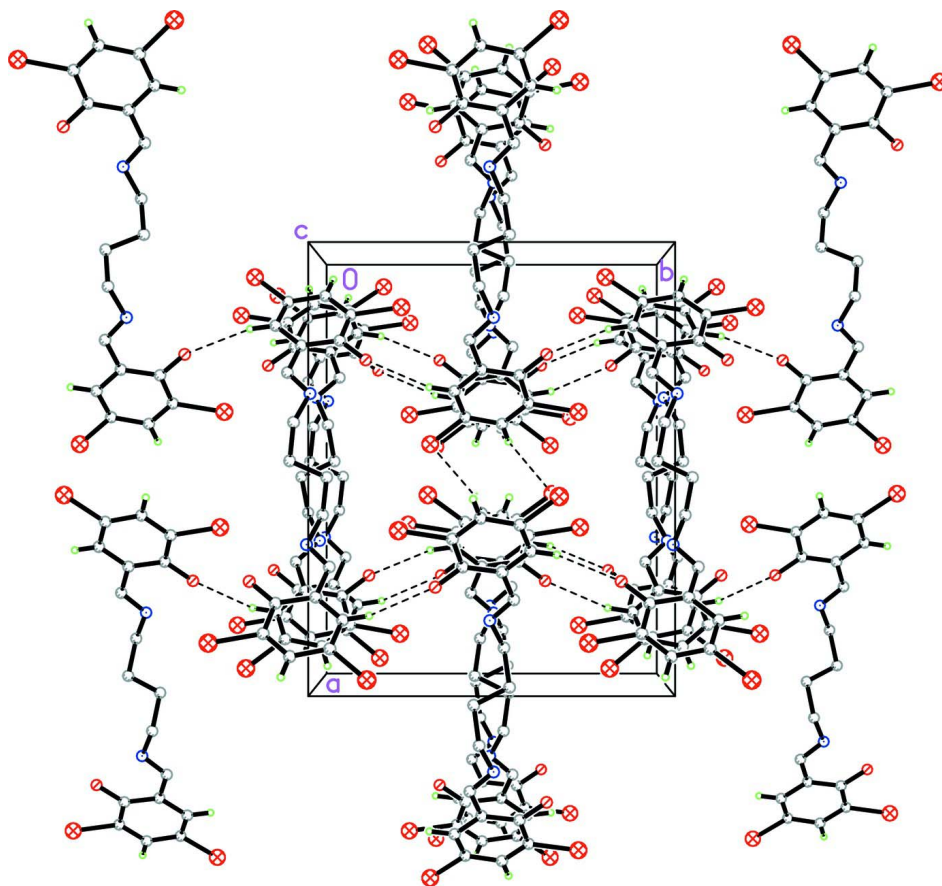
The title compound was synthesized by adding 3,5-dibromosalicylaldehyde (2 mmol) to a solution of butylenediamine (1 mmol) in ethanol (30 ml). The mixture was refluxed with stirring for 30 min. The resultant solution was filtered. Yellow needle-like crystals of the title compound, suitable for *X*-ray structure determination, were obtained by recrystallization from ethanol on slow evaporation of the solvents at room temperature over several days.

S3. Refinement

The OH H atom was located in a difference Fourier map and constrained to ride on the parent O atom with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The C-bound H-atoms were included in calculated positions and treated as riding atoms: C—H = 0.93 and 0.97 Å for CH and CH₂ H-atoms, respectively, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

The molecular structure of the title molecule, showing 40% probability displacement ellipsoids and the atomic numbering [symmetry code for suffix A = -x, -y, -z].

**Figure 2**

The crystal packing of the title compound viewed along the *c* axis, showing linking of molecules through C—H...O and weak C—H...Br interactions (dashed lines; see Table 1 for details). Only the H atoms involved in hydrogen bonding are shown.

4,6-Dibromo-2-[(E)-(4-[(E)-3,5-dibromo-2-hydroxybenzylidene]amino)butyl]iminomethyl]phenol

Crystal data

C₁₈H₁₆Br₄N₂O₂ $M_r = 611.97$ Orthorhombic, *Pbcn*

Hall symbol: -P 2n 2ab

 $a = 15.9537$ (12) Å $b = 12.8784$ (10) Å $c = 9.5566$ (6) Å $V = 1963.5$ (2) Å³ $Z = 4$ $F(000) = 1176$ $D_x = 2.070$ Mg m⁻³Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 2045 reflections

 $\theta = 3.3$ – 27.5° $\mu = 8.21$ mm⁻¹ $T = 291$ K

Needle, yellow

 $0.35 \times 0.14 \times 0.12$ mm

Data collection

Bruker SMART APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2005)

 $T_{\min} = 0.161$, $T_{\max} = 0.439$

15023 measured reflections

2164 independent reflections

1381 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.069$ $\theta_{\max} = 27.2^\circ$, $\theta_{\min} = 2.0^\circ$ $h = -20 \rightarrow 20$ $k = -16 \rightarrow 16$ $l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.035$ $wR(F^2) = 0.074$ $S = 0.99$

2164 reflections

118 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0269P)^2 + 1.1226P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.74$ e Å⁻³ $\Delta\rho_{\min} = -0.57$ e Å⁻³

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

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 (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.13066 (3)	-0.24496 (3)	0.17601 (5)	0.0577 (2)
Br2	0.05752 (3)	0.17551 (3)	0.07666 (5)	0.0549 (2)
O1	0.25622 (17)	-0.15166 (19)	0.3764 (3)	0.0456 (9)
N1	0.3337 (2)	-0.0051 (2)	0.5060 (3)	0.0374 (11)
C1	0.2230 (2)	0.0284 (3)	0.3459 (4)	0.0320 (11)
C2	0.2109 (2)	-0.0776 (3)	0.3154 (4)	0.0324 (11)

C3	0.1493 (2)	-0.1035 (3)	0.2181 (4)	0.0359 (11)
C4	0.1036 (2)	-0.0293 (3)	0.1493 (4)	0.0373 (12)
C5	0.1186 (2)	0.0745 (3)	0.1788 (4)	0.0373 (12)
C6	0.1761 (2)	0.1035 (3)	0.2756 (4)	0.0349 (11)
C7	0.2860 (2)	0.0599 (3)	0.4468 (4)	0.0360 (12)
C8	0.3979 (2)	0.0338 (3)	0.6028 (4)	0.0433 (14)
C9	0.4818 (3)	0.0488 (3)	0.5281 (5)	0.0537 (17)
H1	0.28180	-0.12680	0.42830	0.0680*
H3	0.06320	-0.04820	0.08400	0.0450*
H6	0.18440	0.17350	0.29520	0.0420*
H7	0.29150	0.13000	0.46860	0.0430*
H8A	0.40490	-0.01510	0.67920	0.0520*
H8B	0.37970	0.09950	0.64210	0.0520*
H9A	0.47400	0.09730	0.45150	0.0650*
H9B	0.52140	0.07960	0.59310	0.0650*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0699 (3)	0.0317 (2)	0.0716 (3)	-0.0021 (2)	-0.0215 (3)	-0.0097 (2)
Br2	0.0590 (3)	0.0417 (2)	0.0641 (3)	0.0064 (2)	-0.0152 (2)	0.0100 (2)
O1	0.0460 (17)	0.0348 (14)	0.0561 (18)	0.0052 (13)	-0.0166 (14)	-0.0036 (13)
N1	0.0298 (17)	0.0403 (18)	0.042 (2)	-0.0008 (16)	-0.0012 (16)	-0.0022 (15)
C1	0.027 (2)	0.0309 (19)	0.038 (2)	-0.0025 (17)	0.0029 (19)	0.0002 (17)
C2	0.029 (2)	0.0313 (19)	0.037 (2)	0.0010 (17)	0.0004 (18)	0.0008 (17)
C3	0.037 (2)	0.0267 (18)	0.044 (2)	-0.0035 (18)	0.0021 (19)	-0.0057 (17)
C4	0.035 (2)	0.038 (2)	0.039 (2)	-0.0024 (19)	-0.0080 (19)	-0.0016 (18)
C5	0.033 (2)	0.034 (2)	0.045 (2)	0.0023 (18)	-0.0035 (19)	0.0043 (18)
C6	0.038 (2)	0.0258 (18)	0.041 (2)	-0.0018 (17)	0.005 (2)	-0.0003 (17)
C7	0.037 (2)	0.034 (2)	0.037 (2)	-0.0042 (18)	0.003 (2)	-0.0058 (18)
C8	0.038 (2)	0.053 (3)	0.039 (2)	-0.003 (2)	-0.006 (2)	-0.006 (2)
C9	0.043 (3)	0.049 (3)	0.069 (3)	-0.003 (2)	-0.013 (3)	-0.011 (2)

Geometric parameters (Å, °)

Br1—C3	1.889 (4)	C4—C5	1.387 (5)
Br2—C5	1.896 (4)	C5—C6	1.355 (5)
O1—C2	1.331 (5)	C8—C9	1.529 (6)
O1—H1	0.7200	C9—C9 ⁱ	1.485 (6)
N1—C7	1.265 (5)	C4—H3	0.9300
N1—C8	1.468 (5)	C6—H6	0.9300
C1—C2	1.409 (5)	C7—H7	0.9300
C1—C6	1.395 (5)	C8—H8A	0.9700
C1—C7	1.451 (5)	C8—H8B	0.9700
C2—C3	1.393 (5)	C9—H9A	0.9700
C3—C4	1.370 (5)	C9—H9B	0.9700
C2—O1—H1	107.00	C8—C9—C9 ⁱ	113.8 (3)

C7—N1—C8	118.4 (3)	C3—C4—H3	121.00
C2—C1—C6	119.9 (3)	C5—C4—H3	121.00
C2—C1—C7	120.2 (3)	C1—C6—H6	120.00
C6—C1—C7	119.9 (3)	C5—C6—H6	120.00
O1—C2—C3	120.3 (3)	N1—C7—H7	119.00
C1—C2—C3	117.8 (3)	C1—C7—H7	119.00
O1—C2—C1	121.9 (3)	N1—C8—H8A	109.00
Br1—C3—C2	118.9 (3)	N1—C8—H8B	109.00
C2—C3—C4	121.9 (4)	C9—C8—H8A	109.00
Br1—C3—C4	119.1 (3)	C9—C8—H8B	109.00
C3—C4—C5	118.9 (3)	H8A—C8—H8B	108.00
Br2—C5—C6	120.7 (3)	C8—C9—H9A	109.00
C4—C5—C6	121.4 (3)	C8—C9—H9B	109.00
Br2—C5—C4	117.9 (3)	H9A—C9—H9B	108.00
C1—C6—C5	120.0 (4)	C9 ⁱ —C9—H9A	109.00
N1—C7—C1	121.9 (3)	C9 ⁱ —C9—H9B	109.00
N1—C8—C9	111.1 (3)		
C8—N1—C7—C1	177.7 (3)	O1—C2—C3—C4	-177.5 (3)
C7—N1—C8—C9	-94.4 (4)	C1—C2—C3—Br1	-179.4 (3)
C6—C1—C2—O1	177.7 (3)	C1—C2—C3—C4	2.5 (5)
C6—C1—C2—C3	-2.3 (5)	Br1—C3—C4—C5	-178.9 (3)
C7—C1—C2—O1	-0.3 (5)	C2—C3—C4—C5	-0.8 (5)
C7—C1—C2—C3	179.7 (3)	C3—C4—C5—Br2	177.6 (3)
C2—C1—C6—C5	0.4 (5)	C3—C4—C5—C6	-1.2 (5)
C7—C1—C6—C5	178.4 (3)	Br2—C5—C6—C1	-177.4 (3)
C2—C1—C7—N1	2.7 (5)	C4—C5—C6—C1	1.4 (5)
C6—C1—C7—N1	-175.2 (3)	N1—C8—C9—C9 ⁱ	-62.9 (5)
O1—C2—C3—Br1	0.6 (5)	C8—C9—C9 ⁱ —C8 ⁱ	-180.0 (3)

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

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H1...N1	0.72	1.92	2.574 (4)	151
C6—H6...O1 ⁱⁱ	0.93	2.56	3.469 (5)	165
C4—H3...Br2 ⁱⁱⁱ	0.93	2.96	3.849 (4)	161

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