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4-Bromo-2-[(E)-(2-{2-[(2-[(E)-5-bromo-2-hydroxybenzylidene]amino}phenyl)-sulfanyl]ethylsulfanyl}phenyl)imino-methyl]phenol

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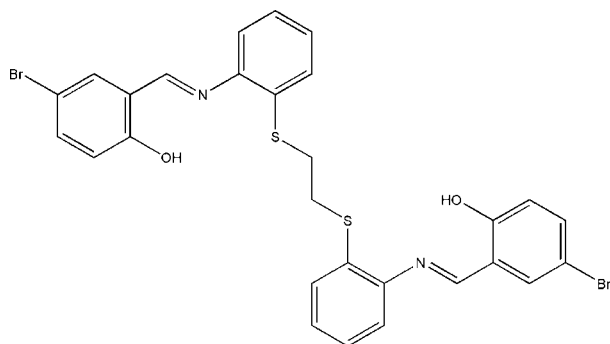
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.052; wR factor = 0.106; data-to-parameter ratio = 17.7.

The asymmetric unit of the title compound, $\text{C}_{28}\text{H}_{22}\text{Br}_2\text{N}_2\text{O}_2\text{S}_2$, comprises half of a Schiff base ligand, the whole molecule being generated by a crystallographic inversion center located at the mid-point of the C–C bond of the central methylene segment. Intramolecular O–H...N and O–H...S hydrogen bonds make $S(6)$ and $S(5)$ ring motifs, respectively. In the crystal, there are no significant intermolecular interactions.

Related literature

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



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Experimental

Crystal data

$\text{C}_{28}\text{H}_{22}\text{Br}_2\text{N}_2\text{O}_2\text{S}_2$
 $M_r = 642.42$
 Monoclinic, $P2_1/c$
 $a = 13.9124$ (18) Å
 $b = 5.4112$ (7) Å
 $c = 17.409$ (2) Å
 $\beta = 92.444$ (7)°

$V = 1309.4$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 3.28$ mm⁻¹
 $T = 296$ 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)
 $T_{\min} = 0.393$, $T_{\max} = 0.694$

10754 measured reflections
 2879 independent reflections
 1277 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.093$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.106$
 $S = 0.93$
 2879 reflections

163 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.38$ e Å⁻³
 $\Delta\rho_{\min} = -0.55$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------|-------|-------------|-------------|---------------|
| O1–H1...N1 | 0.90 | 1.81 | 2.641 (5) | 151 |
| O1–H1...S1 | 0.90 | 2.74 | 3.436 (4) | 135 |

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); 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); software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

HK thanks PNU for financial support. MNT thanks GC University of Sargodha, Pakistan for the research facility.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2486).

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supporting information

Acta Cryst. (2012). E68, o2635 [doi:10.1107/S1600536812034071]

4-Bromo-2-[(*E*)-(2-{2-[(2-[(*E*)-5-bromo-2-hydroxybenzylidene]amino}phenyl)-sulfanyl]ethylsulfanyl}phenyl)iminomethyl]phenol

Hadi Kargar, Reza Kia and Muhammad Nawaz Tahir

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 determined the X-ray crystal structure of the title compound.

The asymmetric unit of the title compound, Fig. 1, comprises half of a Schiff base ligand. The whole molecule is generated by a crystallographic inversion center located in the middle of the C14—C14ⁱ bond of the methylene segment [Symmetry code: (i) $-x, -y+1, -z+1$]. The bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges. Intramolecular O—H \cdots N and O—H \cdots S hydrogen bonds make *S*(6) and *S*(5) ring motifs, respectively (Table 1; Bernstein *et al.*, 1995).

There are no significant intermolecular interactions in the crystal structure.

S2. Experimental

The title compound was synthesized by adding 5-bromosalicylaldehyde (2 mmol) to a solution of 2-(2-(2-aminophenylthio)ethylthio)benzenamine (1 mmol) in ethanol (30 ml). The mixture was refluxed with stirring for half an hour. The resultant solution was filtered. Light-yellow needle-like crystals of the title compound, suitable for X-ray structure analysis, were obtained by slow evaporation of a solution in ethanol at room temperature over several days.

S3. Refinement

The O-bound H atom was located in a difference Fourier map and constrained to refine on the parent 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 $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

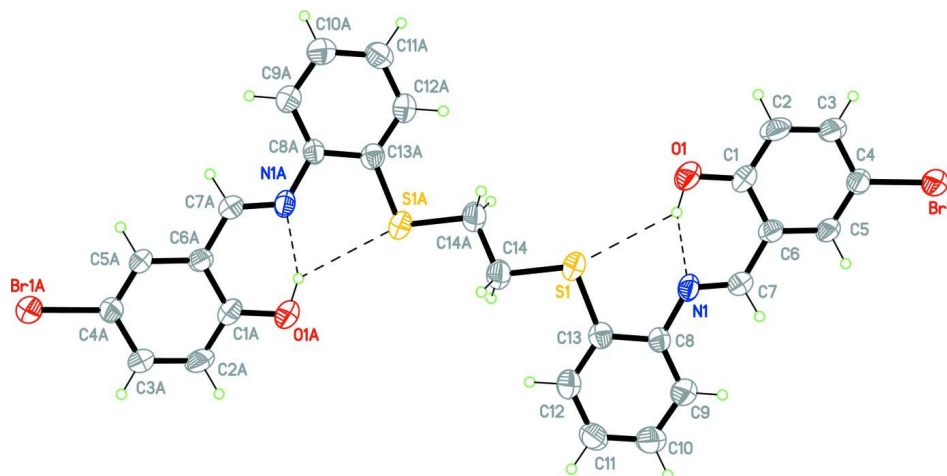


Figure 1

The molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atomic numbering [symmetry code for A: $-x, -y + 1, -z + 1$]. Dashed lines show the intramolecular interactions

4-Bromo-2-[(E)-(2-{2-[(2-[(E)-5-bromo-2-hydroxybenzylidene]amino]phenyl)sulfanyl]ethylsulfanyl}phenyl)iminomethyl]phenol

Crystal data

$C_{28}H_{22}Br_2N_2O_2S_2$

$M_r = 642.42$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 13.9124$ (18) Å

$b = 5.4112$ (7) Å

$c = 17.409$ (2) Å

$\beta = 92.444$ (7)°

$V = 1309.4$ (3) Å³

$Z = 2$

$F(000) = 644$

$D_x = 1.629$ Mg m⁻³

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

Cell parameters from 2098 reflections

$\theta = 2.5$ – 28.8 °

$\mu = 3.28$ mm⁻¹

$T = 296$ K

Needle, light-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)

$T_{\min} = 0.393$, $T_{\max} = 0.694$

10754 measured reflections

2879 independent reflections

1277 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.093$

$\theta_{\max} = 27.1$ °, $\theta_{\min} = 1.5$ °

$h = -17 \rightarrow 17$

$k = -6 \rightarrow 4$

$l = -22 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.106$

$S = 0.93$

2879 reflections

163 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-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.38 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.55 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 | 0.55873 (4) | 1.84843 (11) | 0.63714 (3) | 0.0707 (3) |
| S1 | 0.08346 (11) | 0.7463 (2) | 0.57293 (8) | 0.0622 (4) |
| O1 | 0.2265 (3) | 1.2258 (6) | 0.51696 (19) | 0.0684 (11) |
| H1 | 0.2092 | 1.1303 | 0.5566 | 0.103* |
| N1 | 0.2327 (3) | 0.9977 (8) | 0.6512 (2) | 0.0479 (10) |
| C1 | 0.2994 (4) | 1.3675 (9) | 0.5463 (3) | 0.0502 (13) |
| C2 | 0.3336 (4) | 1.5574 (10) | 0.5026 (3) | 0.0633 (16) |
| H2 | 0.3048 | 1.5889 | 0.4544 | 0.076* |
| C3 | 0.4094 (4) | 1.7013 (9) | 0.5286 (3) | 0.0577 (15) |
| H3 | 0.4322 | 1.8277 | 0.4981 | 0.069* |
| C4 | 0.4522 (3) | 1.6562 (9) | 0.6015 (3) | 0.0492 (13) |
| C5 | 0.4185 (3) | 1.4704 (9) | 0.6453 (3) | 0.0483 (13) |
| H5 | 0.4480 | 1.4404 | 0.6934 | 0.058* |
| C6 | 0.3408 (3) | 1.3230 (8) | 0.6206 (2) | 0.0439 (12) |
| C7 | 0.3043 (4) | 1.1325 (9) | 0.6696 (3) | 0.0510 (13) |
| H7 | 0.3352 | 1.1076 | 0.7174 | 0.061* |
| C8 | 0.1967 (3) | 0.8149 (9) | 0.7006 (3) | 0.0436 (12) |
| C9 | 0.2299 (4) | 0.7718 (10) | 0.7752 (3) | 0.0672 (16) |
| H9 | 0.2797 | 0.8671 | 0.7968 | 0.081* |
| C10 | 0.1892 (4) | 0.5876 (10) | 0.8175 (3) | 0.0692 (17) |
| H10 | 0.2109 | 0.5615 | 0.8681 | 0.083* |
| C11 | 0.1171 (4) | 0.4423 (10) | 0.7861 (3) | 0.0616 (15) |
| H11 | 0.0911 | 0.3157 | 0.8148 | 0.074* |
| C12 | 0.0836 (4) | 0.4857 (10) | 0.7114 (3) | 0.0595 (15) |
| H12 | 0.0338 | 0.3893 | 0.6903 | 0.071* |
| C13 | 0.1228 (3) | 0.6697 (9) | 0.6678 (3) | 0.0447 (12) |
| C14 | 0.0071 (4) | 0.4938 (9) | 0.5431 (2) | 0.0567 (14) |
| H14A | 0.0368 | 0.3379 | 0.5582 | 0.068* |
| H14B | -0.0543 | 0.5063 | 0.5671 | 0.068* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|------------|-------------|--------------|-------------|
| Br1 | 0.0767 (5) | 0.0794 (5) | 0.0547 (4) | -0.0273 (3) | -0.0120 (3) | 0.0093 (3) |
| S1 | 0.0652 (11) | 0.0659 (11) | 0.0537 (9) | -0.0180 (7) | -0.0170 (7) | -0.0023 (7) |
| O1 | 0.059 (3) | 0.091 (3) | 0.053 (2) | -0.020 (2) | -0.0163 (19) | 0.0036 (18) |
| N1 | 0.042 (3) | 0.054 (3) | 0.047 (2) | -0.011 (2) | -0.006 (2) | -0.007 (2) |
| C1 | 0.049 (3) | 0.059 (4) | 0.042 (3) | 0.000 (3) | -0.008 (2) | -0.008 (3) |
| C2 | 0.060 (4) | 0.085 (5) | 0.043 (3) | 0.002 (3) | -0.014 (3) | 0.016 (3) |
| C3 | 0.060 (4) | 0.070 (4) | 0.042 (3) | -0.008 (3) | -0.008 (3) | 0.010 (3) |
| C4 | 0.048 (3) | 0.057 (4) | 0.042 (3) | -0.009 (3) | 0.001 (2) | -0.003 (3) |
| C5 | 0.046 (4) | 0.060 (4) | 0.038 (3) | -0.007 (3) | -0.009 (2) | 0.003 (3) |
| C6 | 0.042 (3) | 0.052 (4) | 0.037 (3) | 0.001 (3) | -0.001 (2) | 0.003 (3) |
| C7 | 0.056 (4) | 0.057 (4) | 0.039 (3) | -0.007 (3) | -0.006 (3) | 0.003 (3) |
| C8 | 0.039 (3) | 0.047 (4) | 0.045 (3) | -0.008 (3) | 0.001 (2) | -0.001 (3) |
| C9 | 0.064 (4) | 0.084 (5) | 0.052 (4) | -0.023 (3) | -0.009 (3) | 0.007 (3) |
| C10 | 0.068 (5) | 0.083 (5) | 0.056 (4) | -0.011 (3) | -0.002 (3) | 0.013 (3) |
| C11 | 0.060 (4) | 0.061 (4) | 0.064 (4) | -0.008 (3) | 0.004 (3) | 0.013 (3) |
| C12 | 0.048 (4) | 0.067 (4) | 0.062 (4) | -0.014 (3) | -0.007 (3) | 0.001 (3) |
| C13 | 0.036 (3) | 0.047 (3) | 0.051 (3) | 0.003 (3) | -0.002 (2) | 0.000 (3) |
| C14 | 0.048 (3) | 0.060 (4) | 0.062 (3) | -0.009 (3) | -0.003 (3) | -0.005 (3) |

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

| | | | |
|------------|-----------|----------------------|-----------|
| Br1—C4 | 1.894 (5) | C6—C7 | 1.444 (6) |
| S1—C13 | 1.766 (5) | C7—H7 | 0.9300 |
| S1—C14 | 1.793 (5) | C8—C9 | 1.379 (6) |
| O1—C1 | 1.354 (5) | C8—C13 | 1.397 (6) |
| O1—H1 | 0.9026 | C9—C10 | 1.376 (6) |
| N1—C7 | 1.265 (5) | C9—H9 | 0.9300 |
| N1—C8 | 1.416 (5) | C10—C11 | 1.369 (7) |
| C1—C2 | 1.377 (6) | C10—H10 | 0.9300 |
| C1—C6 | 1.413 (6) | C11—C12 | 1.383 (6) |
| C2—C3 | 1.373 (6) | C11—H11 | 0.9300 |
| C2—H2 | 0.9300 | C12—C13 | 1.379 (6) |
| C3—C4 | 1.399 (6) | C12—H12 | 0.9300 |
| C3—H3 | 0.9300 | C14—C14 ⁱ | 1.508 (8) |
| C4—C5 | 1.358 (6) | C14—H14A | 0.9700 |
| C5—C6 | 1.396 (6) | C14—H14B | 0.9700 |
| C5—H5 | 0.9300 | | |
| C13—S1—C14 | 104.3 (2) | C9—C8—C13 | 120.1 (5) |
| C1—O1—H1 | 104.8 | C9—C8—N1 | 125.2 (5) |
| C7—N1—C8 | 123.0 (4) | C13—C8—N1 | 114.7 (4) |
| O1—C1—C2 | 119.0 (4) | C10—C9—C8 | 119.8 (5) |
| O1—C1—C6 | 121.3 (5) | C10—C9—H9 | 120.1 |
| C2—C1—C6 | 119.7 (5) | C8—C9—H9 | 120.1 |
| C3—C2—C1 | 121.3 (5) | C11—C10—C9 | 120.9 (5) |

| | | | |
|-----------|-----------|----------------------------|-----------|
| C3—C2—H2 | 119.3 | C11—C10—H10 | 119.5 |
| C1—C2—H2 | 119.3 | C9—C10—H10 | 119.5 |
| C2—C3—C4 | 119.4 (5) | C10—C11—C12 | 119.3 (5) |
| C2—C3—H3 | 120.3 | C10—C11—H11 | 120.3 |
| C4—C3—H3 | 120.3 | C12—C11—H11 | 120.3 |
| C5—C4—C3 | 119.7 (5) | C13—C12—C11 | 121.0 (5) |
| C5—C4—Br1 | 120.4 (4) | C13—C12—H12 | 119.5 |
| C3—C4—Br1 | 119.9 (4) | C11—C12—H12 | 119.5 |
| C4—C5—C6 | 122.0 (4) | C12—C13—C8 | 118.8 (4) |
| C4—C5—H5 | 119.0 | C12—C13—S1 | 124.8 (4) |
| C6—C5—H5 | 119.0 | C8—C13—S1 | 116.3 (4) |
| C5—C6—C1 | 117.7 (4) | C14 ⁱ —C14—S1 | 107.8 (4) |
| C5—C6—C7 | 120.8 (4) | C14 ⁱ —C14—H14A | 110.2 |
| C1—C6—C7 | 121.5 (4) | S1—C14—H14A | 110.2 |
| N1—C7—C6 | 123.7 (5) | C14 ⁱ —C14—H14B | 110.2 |
| N1—C7—H7 | 118.1 | S1—C14—H14B | 110.2 |
| C6—C7—H7 | 118.1 | H14A—C14—H14B | 108.5 |

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

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 | 0.90 | 1.81 | 2.641 (5) | 151 |
| O1—H1 \cdots S1 | 0.90 | 2.74 | 3.436 (4) | 135 |