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## Structure Reports

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## 2,4-Dibromo-6-[(hydroxyimino)methyl]-phenol

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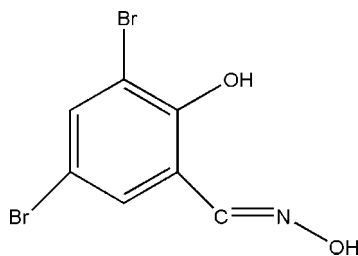
Received 13 July 2011; accepted 17 July 2011

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.013$  Å;  $R$  factor = 0.063;  $wR$  factor = 0.160; data-to-parameter ratio = 13.3.

In the title compound,  $\text{C}_7\text{H}_5\text{Br}_2\text{NO}_2$ , intramolecular  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bonds are observed. In the crystal structure, intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules into dimers.

## Related literature

For details of the preparation, see: Dey *et al.* (2003).



## Experimental

## Crystal data

$\text{C}_7\text{H}_5\text{Br}_2\text{NO}_2$   
 $M_r = 294.94$   
Triclinic,  $P\bar{1}$

$a = 4.2590$  (5) Å  
 $b = 8.6742$  (7) Å  
 $c = 12.0831$  (11) Å

$\alpha = 74.171$  (1)°  
 $\beta = 82.248$  (2)°  
 $\gamma = 79.028$  (1)°  
 $V = 419.98$  (7) Å<sup>3</sup>  
 $Z = 2$

Mo  $K\alpha$  radiation  
 $\mu = 9.60$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.80 \times 0.42 \times 0.18$  mm

## Data collection

Rigaku R-Axis RAPID CCD area-detector diffractometer  
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)  
 $T_{\min} = 0.048$ ,  $T_{\max} = 0.277$

2162 measured reflections  
1453 independent reflections  
987 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.037$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.063$   
 $wR(F^2) = 0.160$   
 $S = 1.05$   
1453 reflections

109 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 1.50$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -1.30$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^i$	0.82	2.10	2.775 (8)	140
$\text{O2}-\text{H2}\cdots\text{N1}$	0.82	1.88	2.601 (10)	147

Symmetry code: (i)  $-x + 2, -y, -z + 3$ .

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP III* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2311).

## References

- Burnett, M. N. & Johnson, C. K. (1996). *ORTEP III*. Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, U.S.  
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**supplementary materials**

*Acta Cryst.* (2011). E67, o2099 [ doi:10.1107/S1600536811028741 ]

## 2,4-Dibromo-6-[(hydroxyimino)methyl]phenol

Y.-H. Luo, J. Xu, M.-L. Pan and J.-F. Li

### Comment

The derivatives of salicylaldehyde are important chemical materials, because they are excellent ligands for transition metals. As part of our interest in these ligands, we report here the crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1, where the dash line indicates the intramolecular O—H $\cdots$ N hydrogen bond.

All the non-H atoms of the title compound are located almost in one plane, as the atoms O1, O2 and N1 are shifted *ca* 0.1204 Å, 0.0727 Å and 0.0402 Å out of the benzene ring plane, respectively.

The title compound formed dimer *via* intermolecular O—H $\cdots$ O hydrogen bonds and the dimers packed *via*  $\pi\cdots\pi$  stacking interactions (3.4367 Å) (Fig. 2).

### Experimental

3,5-dibromosalicylaldehyde were synthesized as follows: 0.2 mol (13.9 g) hydroxylamine hydrochloride in company with 0.2 mol (8 g) NaOH were dissolved in 50 ml ethanol solution in a 250 ml round bottomed flask and stirred to homogeneous. After that, an ethanol solution (30 ml) with 0.2 mol (40 g) 3,5-dibromosalicylaldehyde was added dropwise to this solution at 70 °C and refluxed for about 2 h. After cooling and filtering, crude compound of 3,5-dibromosalicylaldehyde was gained. Pure compound of it was obtained by crystallizing from 20 ml ethanol solution (Dey, *et al.*, 2003).

Crystals of 3,5-dibromosalicylaldehyde suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

### Refinement

All H atoms attached to C atoms and O atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (CH) and O—H = 0.82 Å with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ .

Figures

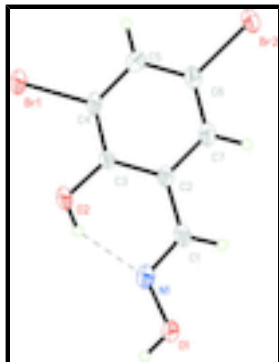


Fig. 1. The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Intramolecular hydrogen bonds are shown as dashed line.

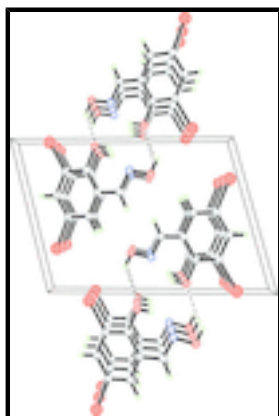


Fig. 2. A packing view down the *a* axis showing the three dimensional network. Intermolecular hydrogen bonds are shown as dashed lines. Intramolecular O—H...N hydrogen bonds have been omitted for the sake of clarity.

**2,4-Dibromo-6-[(hydroxyimino)methyl]phenol**

*Crystal data*

$C_7H_5Br_2NO_2$

$M_r = 294.94$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 4.2590\ (5)\ \text{\AA}$

$b = 8.6742\ (7)\ \text{\AA}$

$c = 12.0831\ (11)\ \text{\AA}$

$\alpha = 74.171\ (1)^\circ$

$\beta = 82.248\ (2)^\circ$

$\gamma = 79.028\ (1)^\circ$

$V = 419.98\ (7)\ \text{\AA}^3$

$Z = 2$

$F(000) = 280$

$D_x = 2.332\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 808 reflections

$\theta = 2.5\text{--}26.6^\circ$

$\mu = 9.60\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Prism, white

$0.80 \times 0.42 \times 0.18\ \text{mm}$

*Data collection*

Rigaku R-Axis RAPID CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

1453 independent reflections

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

$R_{\text{int}} = 0.037$

Detector resolution: 8.192 pixels mm<sup>-1</sup>  
 $\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.5^\circ$   
 $\varphi$  and  $\omega$  scans  
 $h = -5 \rightarrow 5$   
Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2005)  
 $k = -9 \rightarrow 10$   
 $T_{\min} = 0.048$ ,  $T_{\max} = 0.277$   
 $l = -13 \rightarrow 14$   
2162 measured reflections

### Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.063$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.160$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0812P)^2]$
1453 reflections	where $P = (F_o^2 + 2F_c^2)/3$
109 parameters	$(\Delta/\sigma)_{\max} = 0.001$
0 restraints	$\Delta\rho_{\max} = 1.50 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -1.30 \text{ e } \text{\AA}^{-3}$

### 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.3846 (3)	0.03675 (11)	1.18327 (9)	0.0501 (4)
Br2	0.1284 (3)	0.71718 (11)	1.09089 (9)	0.0508 (4)
N1	0.9050 (19)	0.2301 (9)	1.5027 (7)	0.040 (2)
O1	1.0649 (17)	0.2544 (8)	1.5855 (6)	0.0474 (18)
H1	1.1377	0.1664	1.6269	0.071*
O2	0.6873 (17)	0.0731 (7)	1.3820 (6)	0.0469 (18)
H2	0.7721	0.0846	1.4357	0.070*
C1	0.783 (2)	0.3610 (10)	1.4352 (8)	0.037 (2)
H1A	0.8077	0.4594	1.4471	0.045*
C2	0.609 (2)	0.3625 (10)	1.3421 (8)	0.031 (2)
C3	0.575 (2)	0.2192 (10)	1.3157 (7)	0.030 (2)
C4	0.424 (2)	0.2277 (10)	1.2216 (8)	0.036 (2)

## supplementary materials

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C5	0.287 (2)	0.3756 (11)	1.1518 (8)	0.040 (2)
H5	0.1827	0.3798	1.0881	0.048*
C6	0.314 (2)	0.5157 (10)	1.1813 (8)	0.036 (2)
C7	0.477 (2)	0.5089 (11)	1.2733 (8)	0.038 (2)
H7	0.4994	0.6049	1.2896	0.045*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.0685 (8)	0.0273 (6)	0.0611 (8)	-0.0064 (5)	-0.0103 (6)	-0.0208 (5)
Br2	0.0661 (8)	0.0263 (6)	0.0574 (8)	0.0040 (5)	-0.0175 (5)	-0.0084 (5)
N1	0.044 (5)	0.031 (4)	0.048 (5)	-0.005 (4)	-0.002 (4)	-0.016 (4)
O1	0.063 (5)	0.026 (3)	0.058 (5)	0.004 (3)	-0.025 (4)	-0.017 (3)
O2	0.067 (5)	0.019 (3)	0.055 (4)	0.003 (3)	-0.015 (4)	-0.012 (3)
C1	0.031 (5)	0.024 (5)	0.055 (6)	0.003 (4)	-0.005 (4)	-0.012 (5)
C2	0.022 (5)	0.026 (5)	0.048 (6)	0.002 (4)	-0.002 (4)	-0.017 (4)
C3	0.038 (5)	0.021 (4)	0.030 (5)	-0.001 (4)	-0.003 (4)	-0.008 (4)
C4	0.037 (5)	0.022 (5)	0.051 (6)	-0.003 (4)	0.004 (5)	-0.019 (4)
C5	0.043 (6)	0.040 (6)	0.040 (6)	-0.008 (5)	-0.006 (5)	-0.014 (5)
C6	0.044 (6)	0.026 (5)	0.038 (5)	-0.001 (4)	0.001 (4)	-0.011 (4)
C7	0.036 (6)	0.025 (5)	0.053 (6)	-0.008 (4)	0.003 (5)	-0.013 (4)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Br1—C4	1.879 (8)	C2—C7	1.377 (13)
Br2—C6	1.882 (9)	C2—C3	1.402 (11)
N1—C1	1.268 (12)	C3—C4	1.360 (13)
N1—O1	1.364 (10)	C4—C5	1.397 (13)
O1—H1	0.8200	C5—C6	1.384 (12)
O2—C3	1.339 (10)	C5—H5	0.9300
O2—H2	0.8200	C6—C7	1.371 (13)
C1—C2	1.424 (13)	C7—H7	0.9300
C1—H1A	0.9300		
C1—N1—O1	113.3 (7)	C3—C4—C5	122.1 (8)
N1—O1—H1	109.5	C3—C4—Br1	120.1 (7)
C3—O2—H2	109.5	C5—C4—Br1	117.8 (7)
N1—C1—C2	122.3 (8)	C6—C5—C4	117.4 (9)
N1—C1—H1A	118.9	C6—C5—H5	121.3
C2—C1—H1A	118.9	C4—C5—H5	121.3
C7—C2—C3	118.5 (9)	C7—C6—C5	121.0 (9)
C7—C2—C1	119.4 (8)	C7—C6—Br2	120.3 (7)
C3—C2—C1	122.0 (8)	C5—C6—Br2	118.7 (8)
O2—C3—C4	119.1 (8)	C6—C7—C2	121.2 (8)
O2—C3—C2	121.3 (8)	C6—C7—H7	119.4
C4—C3—C2	119.7 (8)	C2—C7—H7	119.4
O1—N1—C1—C2	179.2 (7)	C2—C3—C4—Br1	178.5 (6)
N1—C1—C2—C7	178.8 (9)	C3—C4—C5—C6	0.6 (14)
N1—C1—C2—C3	-3.0 (14)	Br1—C4—C5—C6	179.4 (6)

C7—C2—C3—O2	-177.4 (8)	C4—C5—C6—C7	2.0 (14)
C1—C2—C3—O2	4.4 (13)	C4—C5—C6—Br2	-179.0 (7)
C7—C2—C3—C4	2.2 (13)	C5—C6—C7—C2	-2.5 (14)
C1—C2—C3—C4	-176.0 (8)	Br2—C6—C7—C2	178.6 (7)
O2—C3—C4—C5	176.9 (8)	C3—C2—C7—C6	0.3 (13)
C2—C3—C4—C5	-2.7 (14)	C1—C2—C7—C6	178.6 (8)
O2—C3—C4—Br1	-1.9 (12)		

*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 $\cdots$ O2 <sup>i</sup>	0.82	2.10	2.775 (8)	140.
O2—H2 $\cdots$ N1	0.82	1.88	2.601 (10)	147.

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

Fig. 1

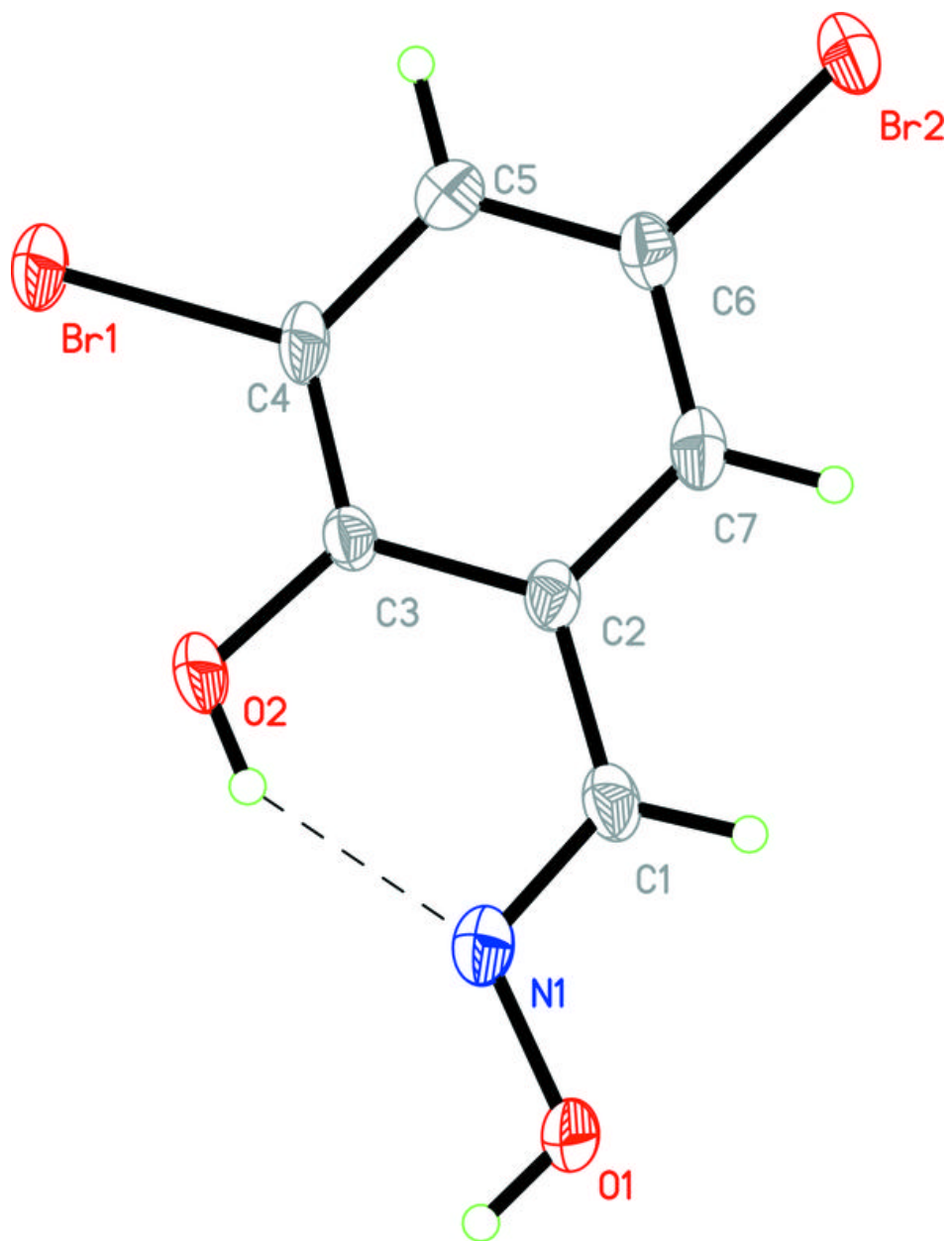




Fig. 2

