organic compounds

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

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In the title compound, $C_7H_5Br_2NO_2$, intramolecular O—H···N hydrogen bonds are observed. In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules into dimers.

Related literature

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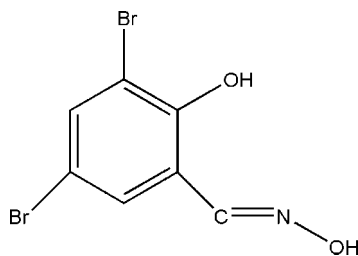
Experimental

Crystal data

$C_7H_5Br_2NO_2$	$a = 4.2590$ (5) Å
$M_r = 294.94$	$b = 8.6742$ (7) Å
Triclinic, $P\bar{1}$	$c = 12.0831$ (11) Å

$\alpha = 74.171$ (1)°	Mo $K\alpha$ radiation
$\beta = 82.248$ (2)°	$\mu = 9.60$ mm^{-1}
$\gamma = 79.028$ (1)°	$T = 293$ K
$V = 419.98$ (7) Å^3	$0.80 \times 0.42 \times 0.18$ mm
$Z = 2$	

Data collection

Rigaku R-AXIS RAPID CCD area-detector diffractometer	2162 measured reflections
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	1453 independent reflections
$T_{min} = 0.048$, $T_{max} = 0.277$	987 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.063$	109 parameters
$wR(F^2) = 0.160$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{max} = 1.50$ e Å^{-3}
1453 reflections	$\Delta\rho_{min} = -1.30$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^i	0.82	2.10	2.775 (8)	140
O2—H2···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: *ORTEPIII* (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). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, U.S.
Dey, M., Rao, C. P., Saarenketo, P. K., Pissanen, K., Kolehmainen, E. & Guionneau, P. (2003). *Polyhedron*, **22**, 3515–3521.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
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supporting information

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

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S1. 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⋯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,0.0727Å and 0.0402Å out of the benzene ring plane, respectively.

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

S2. Experimental

3,5-dibromosalicylaldoxime were synthesized as follows: 0.2 mol (13.9 g) hydroxylamine hydrochloride in companied 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-dibromosalicylicaldehyde was added dropwise to this solution at 70 °C and refluxed for about 2 h. After cooling and filtrating, crude compound of 3,5-di-bromosalicylaldoximewas gained. Pure compound of it was obtained by crystallizing from 20 ml ethanol solution (Dey, *et al.*, 2003).

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

S3. 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 $U_{iso}(H) = 1.2U_{eq}(C)$ and $U_{iso}(H) = 1.5U_{eq}(O)$.
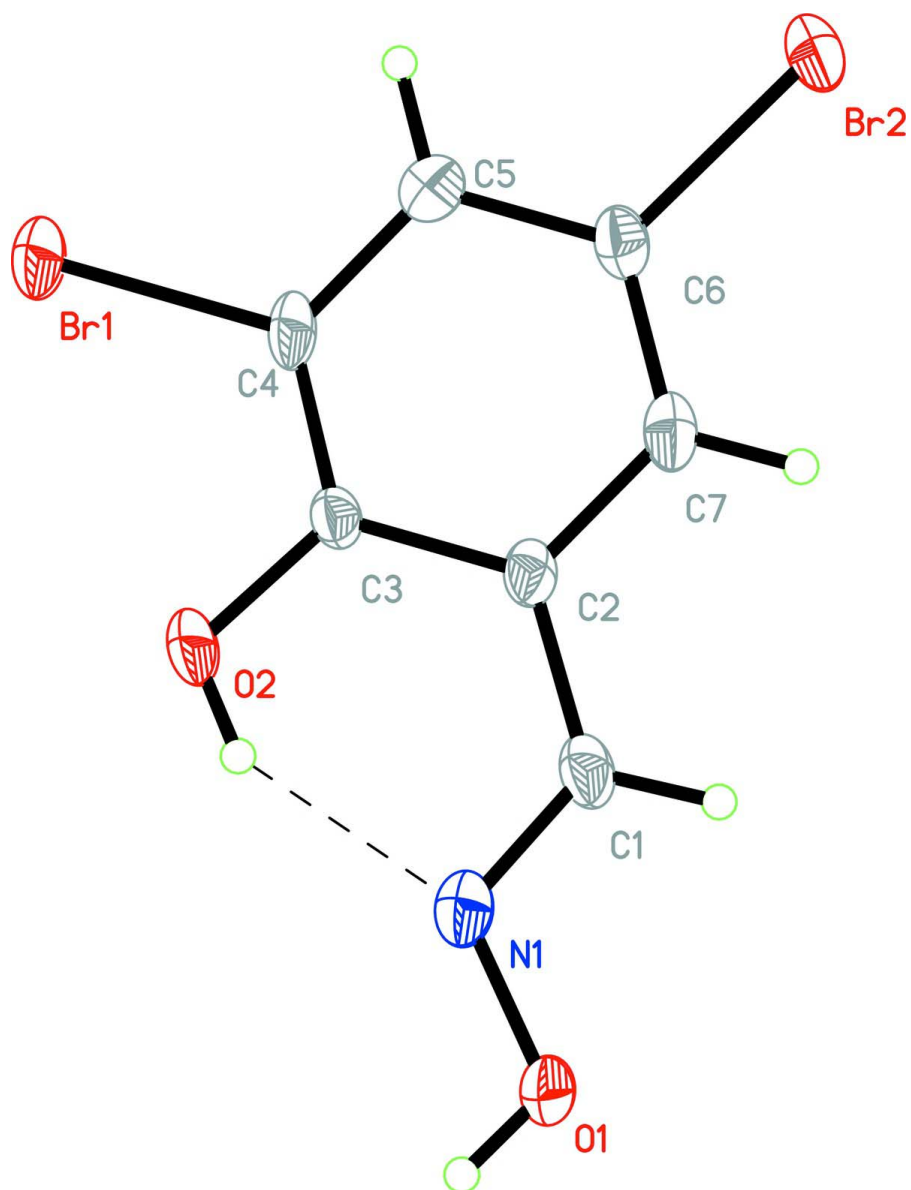
Figure 1

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.
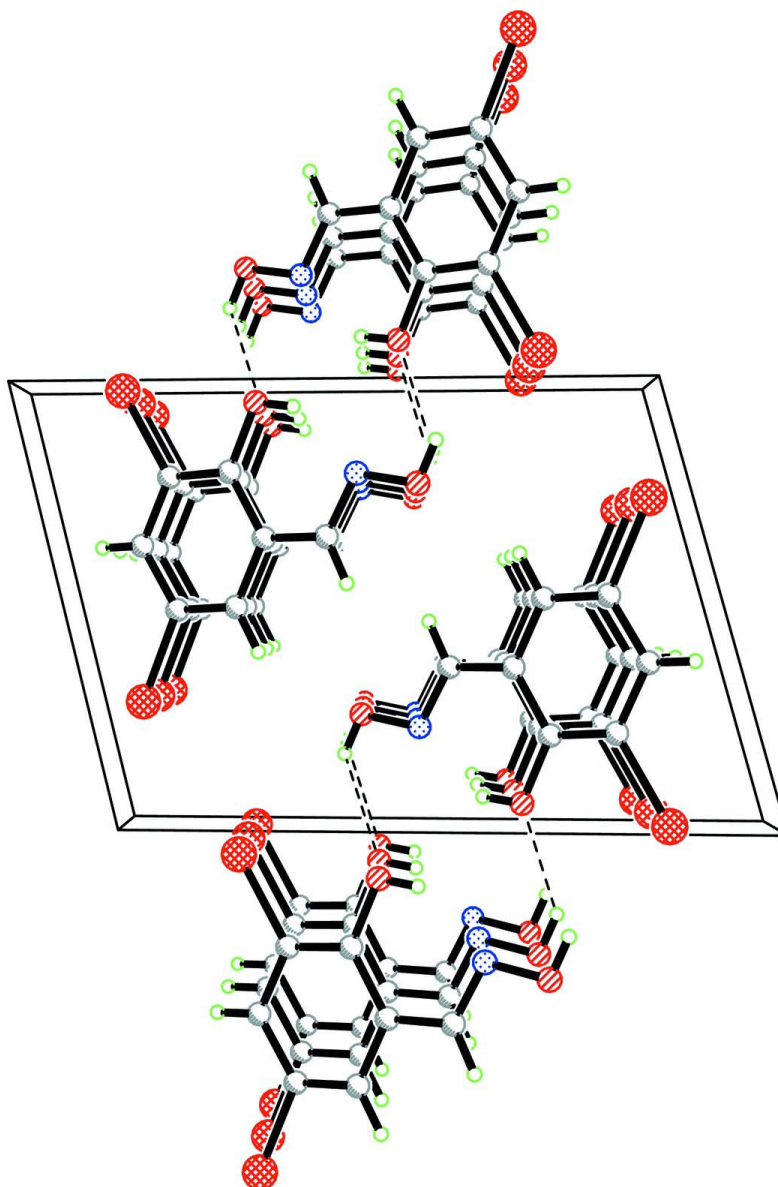
Figure 2

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

$C_7H_5Br_2NO_2$	$\gamma = 79.028\ (1)°$
$M_r = 294.94$	$V = 419.98\ (7)$ Å^3
Triclinic, $P\overline{1}$	$Z = 2$
Hall symbol: -P 1	$F(000) = 280$
$a = 4.2590\ (5)$ Å	$D_x = 2.332$ Mg m^{-3}
$b = 8.6742\ (7)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$c = 12.0831\ (11)$ Å	Cell parameters from 808 reflections
$\alpha = 74.171\ (1)°$	$\theta = 2.5–26.6°$
$\beta = 82.248\ (2)°$	$\mu = 9.60$ mm^{-1}

T = 293 K

Prism, white

0.80 × 0.42 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID CCD area-detector diffractometer	2162 measured reflections
Radiation source: fine-focus sealed tube	1453 independent reflections
Graphite monochromator	987 reflections with $I > 2\sigma(I)$
Detector resolution: 8.192 pixels mm^{-1}	R_{int} = 0.037
φ and ω scans	θ_{max} = 25.0°, θ_{min} = 2.5°
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	$h = -5 \rightarrow 5$
	$k = -9 \rightarrow 10$
T_{min} = 0.048, T_{max} = 0.277	$l = -13 \rightarrow 14$

Refinement

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

	x	y	z	U_{iso}*/U_{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)
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 (Å²)

	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 (Å, º)

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, º)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2[i]	0.82	2.10	2.775 (8)	140
O2—H2···N1	0.82	1.88	2.601 (10)	147

Symmetry code: (i) −x+2, −y, −z+3.