2-[3-(4-Bromophenyl)-1,2,4-oxadiazol-5-yl]phenol

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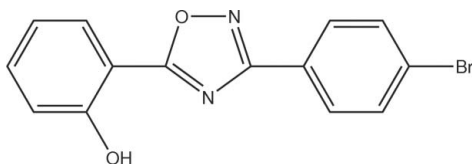Correspondence e-mail: wanghaibo@njut.edu.cn

In the title compound, $C_{14}H_9BrN_2O_2$, the whole molecule is roughly planar, the largest deviation from the mean plane being 0.159 (6) Å for the phenol O atom. The occurrence of an intramolecular O—H···N hydrogen bond might favour this planar conformation. The packing is governed by weak π–π interactions between the oxadiazole and benzene rings of symmetry-related molecules.

Related literature

For related literature, see: Romero (2001); Terashita *et al.* (2002).

Experimental

Crystal data

$C_{14}H_9BrN_2O_2$	$V = 630.2$ (2) Å³
$M_r = 317.14$	$Z = 2$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 6.3950$ (13) Å	$\mu = 3.26$ mm^{-1}
$b = 5.0790$ (10) Å	$T = 293$ (2) K
$c = 19.625$ (4) Å	$0.40 \times 0.10 \times 0.10$ mm
$\beta = 98.65$ (3)°	

Data collection

Enraf–Nonius CAD-4 diffractometer	1381 independent reflections
Absorption correction: ψ scan (North *et al.*, 1968)	881 reflections with $I > 2\sigma(I)$
$T_{min} = 0.356$, $T_{max} = 0.736$	$R_{int} = 0.033$
1507 measured reflections	3 standard reflections every 200 reflections
	intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.114$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
$S = 0.94$	$\Delta\rho_{min} = -0.36$ e Å^{-3}
1381 reflections	Absolute structure: Flack (1983), with no Friedel pairs
173 parameters	Flack parameter: 0.08 (3)
1 restraint	

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N2	0.82	1.93	2.666 (10)	148

Table 2
Main π–π interactions in the title compound.

α is the dihedral angle between the planes, DCC is the length of the CC vector (centroid to centroid), τ is the angle(s) subtended by the plane normal(s) to CC (offset angle). $Cg1$ is the centroid of ring O1/N1/C7/N2/C8, $Cg2$ is the centroid of ring C1–C6, and $Cg3$ is the centroid of ring C9–C14.

Centroid 1	Centroid 2	α°	DCC Å	τ °
$Cg1$	$Cg2^i$	9.19	3.649 (5)	14.7
$Cg1$	$Cg3^{ii}$	5.64	3.661 (5)	22.6

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2275).

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supplementary materials

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Comment

1,2,4-Oxadiazole derivatives possess biological properties such as intrinsic analgesic (Terashita *et al.*, 2002) and antipicornaviral (Romero, 2001) effects. As part of our studies in this area, we report here the synthesis and crystal structure of the title compound, (I).

The whole molecule is roughly planar with the largest deviation from the mean plane being 0.159 (6)\%A. The occurrence of an intramolecular, O—H⋯N hydrogen bond might favor this planar conformation (Fig. 1, Table 1).

The packing is governed by weak π-π interactions between the oxadiazole and the benzene rings of symmetry related molecules (Table 2).

Experimental

4-bromo-*N*-hydroxybenzamidine(10 mmol)was dissolved in ethanol (50 ml) and sodinm ethanolate (30 mmol) was added. Then, methyl 2-hydroxybenzoate (20 mmol)was added to the reaction. The resulting mixture was refluxed for 24 h. After cooling and filtering, the crude title compound was obtained and purified by recrystalization from ethyl acetate. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Refinement

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

Owing to the occurrence of the Br atom, the absolute structure could be determined even if no Friedel pairs were available.

Figures

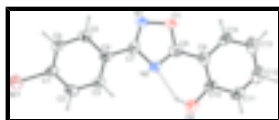Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bond is shown as dashed line.

2-[3-(4-Bromophenyl)-1,2,4-oxadiazol-5-yl]phenol

Crystal data

$C_{14}H_9BrN_2O_2$	$F_{000} = 316$
$M_r = 317.14$	$D_x = 1.671 \text{ Mg m}^{-3}$

supplementary materials

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 6.3950$ (13) Å

$b = 5.0790$ (10) Å

$c = 19.625$ (4) Å

$\beta = 98.65$ (3)°

$V = 630.2$ (2) Å^3

$Z = 2$

Melting point: 431 K

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

Cell parameters from 25 reflections

$\theta = 9–13°$

$\mu = 3.26$ mm^{-1}

$T = 293$ (2) K

Block, colorless

$0.40 \times 0.10 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{min} = 0.356$, $T_{max} = 0.736$

1507 measured reflections

1381 independent reflections

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

$R_{int} = 0.033$

$\theta_{max} = 26.0°$

$\theta_{min} = 1.1°$

$h = -7 \rightarrow 7$

$k = 0 \rightarrow 6$

$l = 0 \rightarrow 24$

3 standard reflections

every 200 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.114$

$S = 0.94$

1381 reflections

173 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.32$ e Å^{-3}

$\Delta\rho_{min} = -0.36$ e Å^{-3}

Extinction correction: none

Absolute structure: Flack parameter (Flack, 1983), no Friedel pairs

Flack parameter: 0.08 (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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.90540 (15)	1.0994 (3)	0.05102 (5)	0.0686 (4)
N1	0.3401 (11)	0.2222 (17)	0.2187 (4)	0.066 (2)
N2	0.6748 (9)	0.134 (2)	0.2756 (3)	0.0451 (19)
O1	0.3297 (9)	0.0163 (13)	0.2682 (3)	0.063 (2)
O2	0.9421 (10)	−0.1051 (16)	0.3737 (4)	0.079 (2)
H2	0.9028	−0.0021	0.3421	0.118*
C1	0.7898 (13)	0.8640 (18)	0.1054 (4)	0.050 (2)
C2	0.9179 (14)	0.750 (2)	0.1593 (4)	0.063 (3)
H2A	1.0587	0.8009	0.1702	0.075*
C3	0.8340 (12)	0.554 (2)	0.1981 (4)	0.051 (3)
H3	0.9214	0.4755	0.2347	0.061*
C4	0.6330 (13)	0.4778 (19)	0.1838 (4)	0.050 (2)
C5	0.4992 (12)	0.600 (4)	0.1278 (4)	0.067 (2)
H5	0.3560	0.5602	0.1175	0.080*
C6	0.5969 (14)	0.787 (2)	0.0884 (4)	0.066 (3)
H6	0.5182	0.8557	0.0487	0.079*
C7	0.5431 (15)	0.2755 (19)	0.2250 (4)	0.056 (2)
C8	0.5339 (14)	−0.021 (2)	0.2972 (4)	0.056 (2)
C9	0.5753 (15)	−0.2226 (19)	0.3509 (4)	0.055 (2)
C10	0.7744 (13)	−0.2542 (19)	0.3866 (5)	0.058 (2)
C11	0.8120 (15)	−0.445 (2)	0.4368 (5)	0.068 (3)
H11	0.9477	−0.4610	0.4613	0.082*
C12	0.6581 (18)	−0.610 (3)	0.4521 (5)	0.080 (3)
H12	0.6902	−0.7389	0.4857	0.095*
C13	0.442 (2)	−0.585 (2)	0.4156 (5)	0.075 (3)
H13	0.3330	−0.6938	0.4248	0.089*
C14	0.4125 (12)	−0.391 (3)	0.3672 (4)	0.058 (2)
H14	0.2769	−0.3672	0.3433	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0825 (6)	0.0546 (5)	0.0718 (5)	−0.0017 (9)	0.0210 (4)	−0.0010 (9)
N1	0.042 (4)	0.068 (6)	0.085 (6)	−0.011 (4)	−0.005 (4)	0.010 (5)
N2	0.042 (3)	0.037 (5)	0.054 (4)	0.011 (5)	0.001 (3)	−0.002 (5)
O1	0.041 (3)	0.072 (6)	0.074 (4)	−0.012 (3)	−0.003 (3)	0.006 (4)
O2	0.070 (4)	0.073 (5)	0.087 (5)	−0.002 (4)	−0.010 (4)	0.015 (4)
C1	0.042 (4)	0.051 (6)	0.054 (5)	0.011 (5)	0.000 (4)	−0.012 (5)
C2	0.052 (5)	0.064 (7)	0.071 (6)	−0.008 (5)	0.006 (5)	−0.015 (6)

C3	0.045 (5)	0.051 (10)	0.053 (5)	0.009 (5)	−0.002 (4)	−0.007 (5)
C4	0.042 (5)	0.047 (5)	0.060 (5)	0.005 (4)	0.002 (4)	−0.005 (5)
C5	0.049 (4)	0.069 (6)	0.081 (6)	−0.002 (9)	0.009 (4)	0.014 (10)
C6	0.060 (6)	0.080 (8)	0.053 (5)	−0.004 (6)	−0.009 (5)	0.015 (6)
C7	0.076 (7)	0.040 (5)	0.049 (5)	0.009 (5)	0.006 (5)	−0.008 (5)
C8	0.060 (6)	0.058 (6)	0.051 (5)	−0.003 (5)	0.009 (5)	−0.018 (5)
C9	0.072 (6)	0.038 (5)	0.060 (5)	0.002 (5)	0.028 (5)	−0.009 (5)
C10	0.044 (5)	0.045 (6)	0.083 (6)	0.020 (5)	0.005 (5)	−0.003 (6)
C11	0.061 (6)	0.062 (10)	0.077 (6)	0.022 (6)	−0.002 (5)	0.001 (6)
C12	0.120 (10)	0.058 (7)	0.066 (7)	0.032 (7)	0.030 (7)	0.000 (6)
C13	0.113 (9)	0.047 (6)	0.073 (7)	0.006 (7)	0.042 (7)	−0.002 (6)
C14	0.053 (5)	0.062 (6)	0.060 (5)	0.034 (8)	0.010 (4)	0.005 (9)

Geometric parameters (Å, °)

Br1—C1	1.831 (9)	C4—C7	1.477 (13)
N1—C7	1.313 (11)	C5—C6	1.425 (16)
N1—O1	1.435 (9)	C5—H5	0.9300
N2—C8	1.314 (12)	C6—H6	0.9300
N2—C7	1.401 (12)	C8—C9	1.463 (13)
O1—C8	1.357 (10)	C9—C10	1.367 (12)
O2—C10	1.367 (10)	C9—C14	1.421 (15)
O2—H2	0.8200	C10—C11	1.377 (14)
C1—C6	1.290 (11)	C11—C12	1.361 (15)
C1—C2	1.366 (12)	C11—H11	0.9300
C2—C3	1.408 (13)	C12—C13	1.462 (15)
C2—H2A	0.9300	C12—H12	0.9300
C3—C4	1.331 (11)	C13—C14	1.362 (16)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.431 (13)	C14—H14	0.9300
C7—N1—O1	103.4 (7)	N1—C7—C4	123.5 (9)
C8—N2—C7	99.7 (7)	N2—C7—C4	120.2 (8)
C8—O1—N1	104.3 (7)	N2—C8—O1	116.3 (9)
C10—O2—H2	109.5	N2—C8—C9	126.3 (9)
C6—C1—C2	120.7 (10)	O1—C8—C9	117.3 (9)
C6—C1—Br1	120.1 (7)	C10—C9—C14	118.1 (9)
C2—C1—Br1	118.8 (7)	C10—C9—C8	120.3 (9)
C1—C2—C3	119.1 (8)	C14—C9—C8	121.5 (9)
C1—C2—H2A	120.5	C9—C10—O2	122.4 (9)
C3—C2—H2A	120.5	C9—C10—C11	119.9 (10)
C4—C3—C2	121.7 (9)	O2—C10—C11	117.7 (9)
C4—C3—H3	119.1	C12—C11—C10	122.5 (10)
C2—C3—H3	119.1	C12—C11—H11	118.8
C3—C4—C5	118.9 (9)	C10—C11—H11	118.8
C3—C4—C7	122.0 (8)	C11—C12—C13	119.9 (10)
C5—C4—C7	119.1 (8)	C11—C12—H12	120.0
C6—C5—C4	116.4 (8)	C13—C12—H12	120.0
C6—C5—H5	121.8	C14—C13—C12	115.3 (11)
C4—C5—H5	121.8	C14—C13—H13	122.3

C1—C6—C5	122.9 (8)	C12—C13—H13	122.3
C1—C6—H6	118.6	C13—C14—C9	124.2 (9)
C5—C6—H6	118.6	C13—C14—H14	117.9
N1—C7—N2	116.2 (9)	C9—C14—H14	117.9

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O2—H2⋯N2	0.82	1.93	2.666 (10)	148

Main π–π interactions in (I).
α is the dihedral angle between the planes, DCC is the length of the CC vector (centroid to centroid), τ is the angle(s) subtended by the plane normal(s) to CC (offset angle). Cg1 is the centroid of ring O1, N1, C7, N2, C8, Cg2 is the centroid of ring C1 to C6 and Cg3 is the centroid of ring C9 to C14.

Centroid 1	Centroid 2	α°	DCC Å	τ °
*Cg*1	*Cg*2[i]	9.19	3.649 (5)	14.7
*Cg*1	*Cg*3[ii]	5.64	3.661 (5)	22.6

Symmetry codes: (i) $x, y − 1, z$; (ii) $x, 1 + y, z$

Fig. 1