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(*E*)-2-Bromobenzaldehyde oxime

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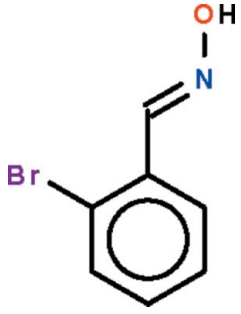
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.022; wR factor = 0.059; data-to-parameter ratio = 15.0.

The configuration of the C=N double bond of the title compound, C_7H_6BrNO, is *E*; the non-H atoms are approximately coplanar (r.m.s. deviation = 0.038 Å). In the crystal, pairs of molecules are linked by a pair of O—H···N hydrogen bonds about a center of inversion, generating hydrogen-bonded dimers.

Related literature

For the synthesis, see: Jin *et al.* (2010). For the spectroscopic differentiation between *E* and *Z* isomers, see: Schnekenburger (1973). For reactions that produce 5-isoxazolpenicillins, see: Wang *et al.* (2007).



Experimental

Crystal data

C_7H_6BrNO M_r = 200.04
Monoclinic, $P2_1/c$
a = 7.7403 (2) Å
b = 4.0012 (1) Å
c = 23.2672 (5) Å
β = 98.810 (2)°
V = 712.09 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 7.25 mm^{-1}
T = 100 K
0.20 × 0.15 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.325, T_{max} = 0.531
4949 measured reflections
1421 independent reflections
1411 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.059
S = 1.06
1421 reflections
95 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.86 (3)	1.98 (3)	2.802 (2)	159 (3)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5606).

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supplementary materials

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(*E*)-2-Bromobenzaldehyde oxime

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Comment

(2-Bromophenyl)methanoxime can be converted to 5-isoxazolpenicillins (Wang *et al.*, 2007); the compound exists into a *E* and a *Z* configuration with respect to the carbon-nitrogen double-bond; mixtures can be differentiated by their UV spectra (Schnekenburger, 1973). A recent study reported the synthesis of the *E* isomer (Scheme I) without the use of a metal-salt catalyst (Jin *et al.*, 2010). Zinc chloride is used in this study to give the compound in high yield. The non-H atoms are co-planar (Fig. 1); two molecules are linked by an O–H···N bond about a center-of-inversion to generate a hydrogen-bonded dimer (Table 1).

Experimental

2-Bromobenzaldehyde (1.0 mmol, 184 mg), 50% hydroxylamine (3.0 mmol, 0.18 ml) and hydrated zinc chloride (0.2 mmol) were heated at 373 K for half an hour. The progress of reaction was monitored by TLC (ethyl acetate / *n*- hexane 1/3). The product was purified by column chromatography on silica gel, with ethanyl acetate/*n*-hexane (1/4) as co-solvent. Colorless were obtained by using ethyl acetate as solvent for recrystallization, m.p. 363 K (yield 90%).

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 Å, U_{iso}(H) 1.2U_{eq}(C)] and were included in the refinement in the riding model approximation.

The hydroxy H-atom was located in a difference Fouier map and was refined.

Figures

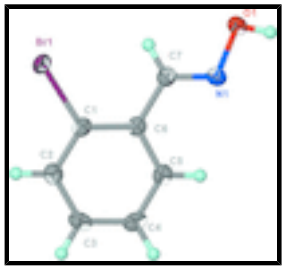

Fig. 1. Anisotropic displacement ellipsoid plot (Barbour, 2001) of C_7H_6BrNO at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

(*E*)-2-Bromobenzaldehyde oxime

Crystal data

C_7H_6BrNO	$F(000) = 392$

M_r = 200.04	D_x = 1.866 Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, λ = 1.54184 Å
Hall symbol: -P 2ybc	Cell parameters from 3601 reflections
a = 7.7403 (2) Å	θ = 3.8–74.0°
b = 4.0012 (1) Å	μ = 7.25 mm^{-1}
c = 23.2672 (5) Å	T = 100 K
β = 98.810 (2)°	Block, colorless
V = 712.09 (3) Å^3	0.20 × 0.15 × 0.10 mm
Z = 4	

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	1421 independent reflections
Radiation source: SuperNova (Cu) X-ray Source	1411 reflections with $I > 2\sigma(I)$
Mirror	R_{int} = 0.017
Detector resolution: 10.4041 pixels mm^{-1}	θ_{max} = 74.2°, θ_{min} = 3.9°
ω scans	h = −8→9
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)	k = −4→4
T_{min} = 0.325, T_{max} = 0.531	l = −28→26
4949 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
$R[F^2 > 2\sigma(F^2)]$ = 0.022	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.059	H atoms treated by a mixture of independent and constrained refinement
S = 1.06	$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 0.6889P]$ where $P = (F_o^2 + 2F_c^2)/3$
1421 reflections	$(\Delta/\sigma)_{max}$ = 0.001
95 parameters	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.14837 (3)	0.15858 (5)	0.213462 (8)	0.02034 (10)
O1	−0.1279 (2)	0.8670 (4)	0.04381 (7)	0.0247 (3)
H1	−0.132 (4)	0.989 (9)	0.0131 (14)	0.042 (8)*
N1	0.0488 (2)	0.7706 (5)	0.05189 (7)	0.0192 (3)
C1	0.3131 (3)	0.2643 (5)	0.16293 (8)	0.0181 (4)
C2	0.4830 (3)	0.1467 (5)	0.17939 (9)	0.0207 (4)
H2	0.5131	0.0239	0.2144	0.025*

C3	0.6078 (3)	0.2117 (6)	0.14383 (10)	0.0226 (4)
H3	0.7241	0.1334	0.1545	0.027*
C4	0.5625 (3)	0.3910 (6)	0.09268 (10)	0.0228 (4)
H4	0.6480	0.4358	0.0684	0.027*
C5	0.3930 (3)	0.5045 (5)	0.07699 (8)	0.0210 (4)
H5	0.3634	0.6248	0.0417	0.025*
C6	0.2639 (3)	0.4463 (5)	0.11190 (8)	0.0175 (4)
C7	0.0852 (3)	0.5747 (5)	0.09540 (8)	0.0188 (4)
H7	−0.0037	0.5116	0.1172	0.023*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02289 (14)	0.02016 (15)	0.01905 (14)	0.00002 (7)	0.00669 (9)	0.00319 (7)
O1	0.0177 (7)	0.0297 (9)	0.0269 (8)	0.0036 (6)	0.0038 (6)	0.0083 (6)
N1	0.0174 (8)	0.0186 (8)	0.0213 (8)	0.0010 (7)	0.0028 (6)	−0.0004 (7)
C1	0.0213 (9)	0.0153 (9)	0.0184 (9)	−0.0018 (8)	0.0054 (7)	−0.0017 (8)
C2	0.0233 (10)	0.0184 (10)	0.0200 (10)	0.0003 (7)	0.0015 (8)	−0.0002 (7)
C3	0.0163 (9)	0.0227 (10)	0.0282 (11)	0.0013 (8)	0.0012 (8)	−0.0048 (8)
C4	0.0217 (10)	0.0243 (11)	0.0237 (10)	−0.0050 (8)	0.0081 (8)	−0.0046 (8)
C5	0.0239 (10)	0.0200 (10)	0.0193 (9)	−0.0017 (8)	0.0041 (7)	0.0001 (8)
C6	0.0194 (9)	0.0146 (9)	0.0183 (9)	−0.0023 (7)	0.0023 (7)	−0.0028 (7)
C7	0.0200 (9)	0.0183 (9)	0.0184 (9)	−0.0020 (8)	0.0038 (7)	−0.0004 (8)

Geometric parameters (Å, °)

Br1—C1	1.9093 (19)	C3—C4	1.388 (3)
O1—N1	1.406 (2)	C3—H3	0.9500
O1—H1	0.86 (3)	C4—C5	1.384 (3)
N1—C7	1.277 (3)	C4—H4	0.9500
C1—C2	1.394 (3)	C5—C6	1.400 (3)
C1—C6	1.395 (3)	C5—H5	0.9500
C2—C3	1.389 (3)	C6—C7	1.471 (3)
C2—H2	0.9500	C7—H7	0.9500
N1—O1—H1	100 (2)	C5—C4—H4	120.0
C7—N1—O1	111.47 (16)	C3—C4—H4	120.0
C2—C1—C6	122.18 (18)	C4—C5—C6	121.63 (19)
C2—C1—Br1	116.55 (15)	C4—C5—H5	119.2
C6—C1—Br1	121.26 (15)	C6—C5—H5	119.2
C3—C2—C1	119.08 (19)	C5—C6—C1	117.05 (18)
C3—C2—H2	120.5	C5—C6—C7	121.12 (18)
C1—C2—H2	120.5	C1—C6—C7	121.83 (18)
C2—C3—C4	120.03 (19)	N1—C7—C6	120.37 (18)
C2—C3—H3	120.0	N1—C7—H7	119.8
C4—C3—H3	120.0	C6—C7—H7	119.8
C5—C4—C3	120.0 (2)		
C6—C1—C2—C3	−0.2 (3)	C2—C1—C6—C5	0.7 (3)
Br1—C1—C2—C3	179.03 (15)	Br1—C1—C6—C5	−178.50 (15)

C1—C2—C3—C4	−0.1 (3)	C2—C1—C6—C7	−178.85 (19)
C2—C3—C4—C5	−0.2 (3)	Br1—C1—C6—C7	2.0 (3)
C3—C4—C5—C6	0.7 (3)	O1—N1—C7—C6	−179.41 (17)
C4—C5—C6—C1	−0.9 (3)	C5—C6—C7—N1	−7.1 (3)
C4—C5—C6—C7	178.62 (19)	C1—C6—C7—N1	172.4 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.86 (3)	1.98 (3)	2.802 (2)	159 (3)

Symmetry codes: (i) −*x*, −*y*+2, −*z*.

Fig. 1