organic compounds

(E)-3-Bromo-N-(1,3-oxazolidin-2-yl-idene)benzamide

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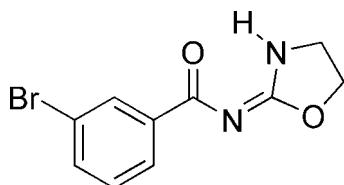Correspondence e-mail: njupanyi@yahoo.com.cn

The five- and six-membered rings in the title compound, $C_{10}H_9BrN_2O_2$, are essentially coplanar. This is consistent with a highly conjugated system, as seen in the short N—C bond distances of 1.308 (6) and 1.317 (5) Å.

Related literature

For related literature, see: Allen (2002); Aoi & Okada (1996); Decken *et al.* (2006); Eisnor *et al.* (2006); Meyers (2005).

Experimental

Crystal data

$C_{10}H_9BrN_2O_2$	V = 1025.4 (2) Å³
M_r = 269.10	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 8.3877 (9) Å	μ = 3.99 mm⁻¹
b = 12.5593 (14) Å	T = 291 (2) K
c = 10.1907 (12) Å	0.30 × 0.26 × 0.24 mm
β = 107.222 (2)°	

Data collection

Bruker SMART APEX CCD diffractometer	5489 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2000) T_{min} = 0.32, T_{max} = 0.38	2016 independent reflections
	1523 reflections with $I > 2\sigma(I)$
	R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.109	
S = 0.99	
2016 reflections	$\Delta\rho_{max}$ = 0.35 e Å⁻³
139 parameters	$\Delta\rho_{min}$ = −0.68 e Å⁻³

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C6—H6$\cdots$N1	0.93	2.50	2.817 (6)	100

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2220).

References

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supporting information

(*E*)-3-Bromo-*N*-(1,3-oxazolidin-2-ylidene)benzamide

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S1. Comment

2-Oxazolines are an important class of heterocyclic compounds which have been applied to a number of areas of chemical endeavour. For example, asymmetric organic synthesis (Meyers, 2005), polymerization chemistry (Aoi & Okada, 1996), Lewis acid catalysis (Eisnor *et al.*, 2006), and coordination chemistry (Decken *et al.*, 2006). Although their coordination complexes are widespread used, few metal-free species have been characterized crystallographically (Allen, 2002). Herein, the molecular structure of the title compound (I) is described.

The bond lengths and angles determined for (I) have the usual values found for structurally similar molecules (Allen, 2002). The existence of a conjugated system is found in the values of the N1—C8 [1.308 (6) Å] and N2—C8 [1.317 (5) Å] bond distances. The dihedral angle between the aryl rings and the oxazolidine ring is 1.82 (2)° and the C4—C7—N2—C8 torsion angle is 179.10 (4)°, indicating planarity of the model.

S2. Experimental

To a solution of 4-chlorobenzoyl chloride (1 mmol) in CH_2Cl_2 (5 ml) was added ammonium thiocyanate (1.3 mmol) and PEG-400 (0.1 mmol). The mixture was then stirred at room temperature for 1 h. A solution of 2-aminoethanol (0.9 mmol) in CH_2Cl_2 (2 ml) was added. The mixture was continuously stirred for 30 min. After the reaction was completed, water (10 ml) was added and the organic phase was dried over anhydrous sodium sulfate. The solvent was removed under vacuum and the residue was purified by flash chromatography to give 1-(3-bromobenzoyl)-3-(2-hydroxyethyl)thiourea in 95% yield. This compound was reacted with dicyclohexylcarbodiimide under weakly basic conditions in acetonitrile to give (I) in 93% yield. Single crystals suitable for the X-ray diffraction study were obtained by slow evaporation of an acetone/water solution of (I); m. p. 374–375 K.

S3. Refinement

The positional parameters of the N—H atom were refined freely with $U_{iso}(H) = 1.2U_{eq}(N)$; N—H = 0.91 (6) Å. The C-bound H atoms were included in the riding model approximation with C—H = 0.93 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$.
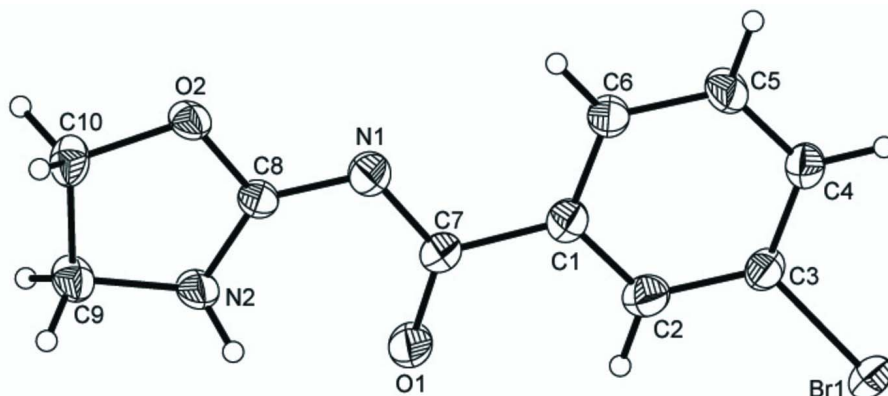
Figure 1

The molecular structure of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids.

(*E*)-3-Bromo-*N*-(1,3-oxazolidin-2-ylidene)benzamide

Crystal data

$C_{10}H_9BrN_2O_2$	$F(000) = 536$
$M_r = 269.10$	$D_x = 1.743$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 2276 reflections
$a = 8.3877$ (9) Å	$\theta = 2.6–27.0°$
$b = 12.5593$ (14) Å	$\mu = 3.99$ mm^{-1}
$c = 10.1907$ (12) Å	$T = 291$ K
$\beta = 107.222$ (2)°	Block, colourless
$V = 1025.4$ (2) Å^3	$0.30 \times 0.26 \times 0.24$ mm
$Z = 4$	

Data collection

Bruker SMART APEX CCD diffractometer	5489 measured reflections
Radiation source: sealed tube	2016 independent reflections
Graphite monochromator	1523 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.043$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$\theta_{max} = 26.0°$, $\theta_{min} = 2.7°$
	$h = -10 \rightarrow 10$
	$k = -15 \rightarrow 12$
$T_{min} = 0.32$, $T_{max} = 0.38$	$l = -8 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.046$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.109$	
$S = 0.99$	H atoms treated by a mixture of independent and constrained refinement
2016 reflections	
139 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.35$ e Å^{-3}
	$\Delta\rho_{min} = -0.68$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.04206 (6)	1.43302 (3)	0.32211 (5)	0.04284 (18)
C1	0.0393 (6)	1.1042 (4)	0.3277 (5)	0.0444 (10)
C2	0.0857 (6)	1.2085 (4)	0.3458 (5)	0.0490 (11)
H2	0.1950	1.2257	0.3946	0.059*
C3	−0.0281 (6)	1.2896 (4)	0.2923 (5)	0.0404 (10)
C4	−0.1893 (6)	1.2665 (4)	0.2181 (5)	0.0455 (11)
H4	−0.2645	1.3206	0.1801	0.055*
C5	−0.2374 (5)	1.1618 (4)	0.2011 (5)	0.0502 (11)
H5	−0.3472	1.1450	0.1532	0.060*
C6	−0.1254 (5)	1.0807 (4)	0.2540 (5)	0.0436 (10)
H6	−0.1600	1.0101	0.2404	0.052*
C7	0.1639 (6)	1.0192 (4)	0.3880 (5)	0.0444 (10)
C8	0.2087 (5)	0.8408 (3)	0.4213 (4)	0.0371 (9)
C9	0.4320 (5)	0.7332 (4)	0.5310 (5)	0.0456 (11)
H9A	0.4701	0.7237	0.6299	0.055*
H9B	0.5225	0.7167	0.4931	0.055*
C10	0.2784 (5)	0.6662 (4)	0.4651 (5)	0.0476 (11)
H10A	0.2988	0.6185	0.3969	0.057*
H10B	0.2479	0.6242	0.5339	0.057*
N1	0.1044 (5)	0.9177 (3)	0.3686 (5)	0.0496 (10)
N2	0.3674 (5)	0.8409 (3)	0.4935 (4)	0.0446 (9)
H2A	0.372 (6)	0.884 (5)	0.566 (5)	0.053*
O1	0.3084 (4)	1.0460 (3)	0.4488 (5)	0.0657 (12)
O2	0.1470 (4)	0.7416 (2)	0.4011 (3)	0.0413 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0564 (3)	0.0335 (3)	0.0415 (3)	−0.0033 (2)	0.01904 (19)	−0.00697 (19)
C1	0.044 (2)	0.037 (2)	0.050 (3)	0.000 (2)	0.012 (2)	−0.003 (2)
C2	0.046 (3)	0.046 (3)	0.052 (3)	−0.006 (2)	0.011 (2)	−0.008 (2)
C3	0.039 (2)	0.033 (2)	0.050 (3)	−0.0011 (18)	0.0145 (19)	−0.0056 (18)
C4	0.042 (2)	0.039 (2)	0.054 (3)	0.0062 (19)	0.012 (2)	−0.003 (2)
C5	0.028 (2)	0.048 (3)	0.068 (3)	0.0001 (19)	0.004 (2)	0.000 (2)
C6	0.032 (2)	0.037 (2)	0.058 (3)	−0.0041 (18)	0.0079 (19)	−0.002 (2)
C7	0.045 (2)	0.034 (2)	0.049 (3)	−0.0020 (19)	0.006 (2)	−0.0057 (19)

C8	0.027 (2)	0.038 (2)	0.044 (2)	−0.0029 (16)	0.0072 (16)	−0.0023 (18)
C9	0.030 (2)	0.045 (2)	0.061 (3)	−0.0017 (18)	0.011 (2)	0.006 (2)
C10	0.038 (2)	0.035 (2)	0.073 (3)	0.0006 (19)	0.020 (2)	0.006 (2)
N1	0.046 (2)	0.036 (2)	0.059 (2)	−0.0029 (18)	0.0049 (18)	0.0021 (18)
N2	0.0314 (19)	0.038 (2)	0.056 (2)	−0.0071 (15)	−0.0002 (17)	0.0013 (18)
O1	0.0422 (19)	0.0362 (19)	0.101 (3)	−0.0062 (15)	−0.0059 (19)	−0.0101 (19)
O2	0.0309 (14)	0.0372 (16)	0.0486 (17)	−0.0080 (12)	0.0005 (12)	0.0050 (13)

Geometric parameters (Å, º)

Br1—C3	1.891 (4)	C7—N1	1.362 (6)
C1—C2	1.364 (7)	C8—N1	1.308 (6)
C1—C6	1.395 (6)	C8—N2	1.317 (5)
C1—C7	1.494 (7)	C8—O2	1.340 (5)
C2—C3	1.392 (7)	C9—N2	1.466 (6)
C2—H2	0.9300	C9—C10	1.519 (6)
C3—C4	1.371 (6)	C9—H9A	0.9700
C4—C5	1.371 (7)	C9—H9B	0.9700
C4—H4	0.9300	C10—O2	1.453 (5)
C5—C6	1.383 (6)	C10—H10A	0.9700
C5—H5	0.9300	C10—H10B	0.9700
C6—H6	0.9300	N2—H2A	0.91 (6)
C7—O1	1.233 (5)		
C2—C1—C6	118.3 (4)	N1—C8—N2	132.1 (4)
C2—C1—C7	119.6 (4)	N1—C8—O2	116.3 (4)
C6—C1—C7	122.1 (4)	N2—C8—O2	111.6 (4)
C1—C2—C3	121.0 (4)	N2—C9—C10	101.3 (3)
C1—C2—H2	119.5	N2—C9—H9A	111.5
C3—C2—H2	119.5	C10—C9—H9A	111.5
C4—C3—C2	120.7 (4)	N2—C9—H9B	111.5
C4—C3—Br1	120.0 (3)	C10—C9—H9B	111.5
C2—C3—Br1	119.3 (3)	H9A—C9—H9B	109.3
C3—C4—C5	118.6 (4)	O2—C10—C9	105.6 (3)
C3—C4—H4	120.7	O2—C10—H10A	110.6
C5—C4—H4	120.7	C9—C10—H10A	110.6
C4—C5—C6	121.1 (4)	O2—C10—H10B	110.6
C4—C5—H5	119.4	C9—C10—H10B	110.6
C6—C5—H5	119.4	H10A—C10—H10B	108.8
C5—C6—C1	120.3 (4)	C8—N1—C7	117.4 (4)
C5—C6—H6	119.9	C8—N2—C9	112.3 (4)
C1—C6—H6	119.9	C8—N2—H2A	104 (3)
O1—C7—N1	126.4 (4)	C9—N2—H2A	115 (4)
O1—C7—C1	118.4 (4)	C8—O2—C10	109.2 (3)
N1—C7—C1	115.3 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···N1	0.93	2.50	2.817 (6)	100