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5-(4-Bromophenyl)-1,3-oxazol-2-amine

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The title compound, $C_9H_7BrN_2O$, was synthesized by the hydrazinolysis of 2-(4-bromophenyl)oxazolo[3,2-*a*]pyrimidinium perchlorate. In the molecule, the oxazole and benzene rings make a dihedral angle of 9.68 (7)°. The crystal packing is stabilized by intermolecular N—H···N hydrogen bonds and van der Waals forces.

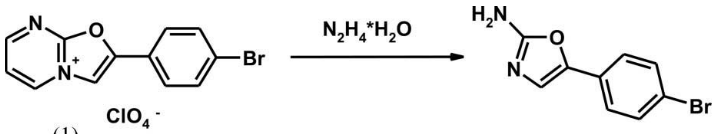
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.091
Data-to-parameter ratio = 14.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

In our previous communication (Rybakov *et al.*, 2006) we have reported the synthesis and crystal structure of 2-(4-bromophenyl)oxazolo[3,2-*a*]pyrimidinium perchlorate, (1). Investigating the reaction of (1) with hydrazine we have found that the cation of (1) underwent selective cleavage of the pyrimidine fragment resulting in the title compound, (2). This type of conversion, unknown in the literature, provides an efficient method of synthesis of 2-aminooxazoles. The title compound, (2), was obtained in 96% yield, which is much better than the known synthetic routes, where (2) has been synthesized either by the reaction of cyanourea with *p*-bromophenacyl bromide (Beiling *et al.*, 1965; Beyer & Schilling, 1966; van Leusen *et al.*, 1981) or by the Curtius rearrangement of the hydrazide of oxazolyl-2-carboxylic acid (Tanaka & Nishiki, 1967). We report here the crystal structure of (2).



(1) (2)

In (2) (Fig. 1), all bonds lengths and angles show normal values (Allen *et al.*, 1987). The oxazole and benzene rings make a dihedral angle of 9.68 (7)°. There is one exocyclic C—N bond involving an amino group (N5). Although amino–imino tautomerism is possible for α-aminoheterocycles, X-ray data confirm the amino form in (2). A search for this amino group in 2-aminooxazoles collected in the Cambridge Structural Database (Version 5.27; Allen, 2002) resulted in only four hits only: 2-amino-6-ethyl-4,5,7,8-tetrahydro-6*H*-oxazolo-(5,4-*d*)azepine (Carpy *et al.*, 1982), 6-allyl-2-amino-5,6,7,8-tetrahydro-4*H*-oxazolo(5,4-*d*)azepine (Luger *et al.*, 1986), ethyl-2-amino-oxazole-5-carboxylate (Kennedy *et al.*, 2001) and 2-amino-4,5-dimethyl-3-(ethoxycarbonylmethyl)-1,3-oxazolium bromide (Peters *et al.*, 1999). The mean exocyclic C—N bond length for these hits is 1.327 (17) Å, while in (2) C5—N5 is 1.2910 (18) Å.

The crystal packing (Fig. 2) is stabilized by intermolecular N—H···N hydrogen bonds (Table 1), which link the molecules into centrosymmetric dimers, and van der Waals forces.

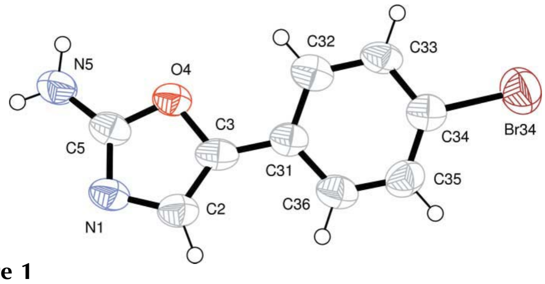


Figure 1
The molecular structure of the title compound, showing the atom numbering scheme and displacement ellipsoids at the 50% probability level.

Experimental

Compound (1) (0.01 mol) was suspended in 50 ml of acetonitrile, and 5 ml of hydrazine hydrate was added with stirring. The reaction mixture was refluxed for 0.5 h, cooled to room temperature and poured into 200 ml of water. The product was isolated by suction and recrystallized from ethanol.

Crystal data

$C_9H_7BrN_2O$
$M_r = 239.07$
Orthorhombic, $Pccn$
$a = 39.344$ (3) Å
$b = 7.5684$ (8) Å
$c = 6.0571$ (8) Å
$V = 1803.6$ (3) Å^3
$Z = 8$
$D_x = 1.761$ Mg m^{-3}
Cu $K\alpha$ radiation
$\mu = 5.88$ mm^{-1}
$T = 293$ (2) K
Plate, yellow
0.12 × 0.12 × 0.02 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.539$, $T_{max} = 0.891$
1860 measured reflections
1860 independent reflections
1644 reflections with $I > 2\sigma(I)$
$\theta_{max} = 75.0°$
1 standard reflection frequency: 60 min intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.091$
$S = 1.03$
1860 reflections
127 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0753P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.78$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0021 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5A···N1^i	0.84 (3)	2.12 (3)	2.947 (2)	165 (3)

Symmetry code: (i) $-x+1, -y+2, -z+2$.

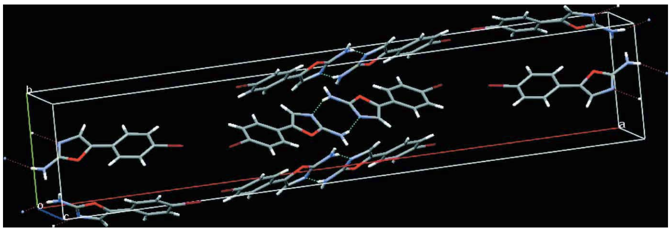


Figure 2
The packing, showing the hydrogen bonds as dashed lines.

The C-bound H atoms were positioned geometrically (C–H = 0.93 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$. The amino H atoms H5$A$ and H5B were initially placed in calculated positions, and then refined isotropically.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; 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: *ORTEP-3 for Windows* (Farrugia, 1997) and *MERCURY* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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