organic papers

Taek Hyeon Kim,[a] Jung Hee Jang[a] and Uk Lee[b]*

[a]Department of Applied Chemistry, Chonnam National University, Gwangju 500-757, Republic of Korea, and [b]Department of Chemistry, Pukyong National University, 599-1 Daeyeon-3dong Nam-ku, Pusan 608-737, Republic of Korea

Correspondence e-mail: uklee@pknu.ac.kr

3-(4-Bromobenzoyl)-N-phenyl-1,3-oxazolidin-2-imine

The title compound, $C_{17}H_{15}BrN_2O_2$, was prepared by the reaction of 2-anilino-4-methyl-2-oxazoline with 4-bromo-benzoyl chloride in the presence of potassium *tert*-butoxide at room temperature. X-ray crystallographic analysis shows that the *endo*-substituted product is formed.

Comment

Alkylation and acylation reactions of 2-amino-2-oxazolines bearing similarly ambident nucleophiles exhibit problems of regioselectivity, occurring either on the endocyclic or exocyclic N atom, depending on the experimental conditions and on the nature of the electrophilic reactants. The reaction of 2-amino-2-thiazolines with some electrophilic compounds has been investigated in detail (Avalos *et al.*, 2000), but 2-amino-2-oxazolines have been less studied (Ganboa *et al.*, 1982; Lee *et al.*, 2002). The reaction of 2-anilino-4-methyl-2-oxazoline with 4-bromobenzoyl chloride furnished regioselectively the *endo*-substituted product 3-(4-bromobenzoyl)-N-phenyl-2-oxazoli-dinimine, (I), and its structure is reported here.
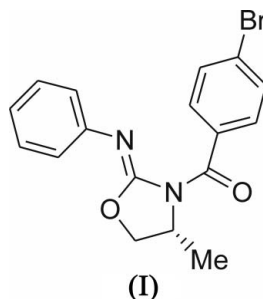
All bond lengths and angles in (I) show normal values (Table 1). The iminooxazoline ring adopts an envelope conformation (Fig. 1), with atom C10 deviating from the N1/C8/C9/O2 plane by 0.149 Å. The N1/C8/C9/O2 mean plane forms dihedral angles of 46.9 (3) and 62.5 (3)° with the C1–C6 and C11–C16 rings, respectively.

Experimental

The title compound was synthesized following the procedure used by Jang *et al.* (2005) for related compounds. To a stirred solution of potassium *tert*-butoxide (0.16 g, 1.43 mmol) and 2-anilino-4-methyl-2-oxazoline (0.27 g, 1.19 mmol) in anhydrous tetrahydrofuran (15 ml) under nitrogen at room temperature, 4-bromobenzoyl chloride (0.34 g, 1.55 mmol) was added dropwise. The solution was stirred for 30 min, then quenched with water (30 ml) and extracted with diethyl ether. The combined extracts were dried over magnesium sulfate, filtered and concentrated. Purification by flash chromatography

afforded the title compound in 71% yield. Single crystals suitable for an X-ray diffraction study were obtained by recrystallization from hexane (m.p. 393–395 K).

Crystal data

C$_{17}$H$_{15}$BrN$_2$O$_2$
$M_r = 359.22$
Triclinic, $P\bar{1}$
$a = 5.867$ (1) Å
$b = 10.638$ (2) Å
$c = 13.982$ (3) Å
$\alpha = 69.44$ (3)°
$\beta = 78.09$ (3)°
$\gamma = 86.28$ (3)°
$V = 799.5$ (3) Å^3

$Z = 2$
$D_x = 1.492$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 34 reflections
$\theta = 9.6$–$10.5°$
$\mu = 2.58$ mm^{-1}
$T = 298$ (2) K
Plate, colourless
$0.30 \times 0.20 \times 0.01$ mm

Data collection

Stoe STADI-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: numerical (X-SHAPE; Stoe & Cie, 1996)
$T_{min} = 0.578$, $T_{max} = 0.895$
3646 measured reflections
3646 independent reflections
2237 reflections with $I > 2\sigma(I)$

$\theta_{max} = 27.5°$
$h = 0 \to 7$
$k = -13 \to 13$
$l = -17 \to 18$
3 standard reflections
 frequency: 60 min
 intensity decay: 3.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.196$
$S = 1.13$
3646 reflections
199 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0712P)^2 + 1.6199P]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.98$ e Å^{-3}
$\Delta\rho_{min} = -0.76$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C1	1.898 (5)	N1—C10	1.396 (7)
O1—C7	1.212 (7)	N1—C8	1.473 (7)
O2—C10	1.359 (6)	N2—C10	1.253 (7)
O2—C9	1.455 (7)	N2—C11	1.411 (7)
N1—C7	1.390 (7)		
C10—O2—C9	108.9 (4)	C10—N1—C8	109.1 (4)
C7—N1—C10	129.6 (4)	C10—N2—C11	121.5 (5)
C7—N1—C8	120.2 (4)		

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: STADI4 (Stoe & Cie, 1996); cell refinement: STADI4; data reduction: X-RED (Stoe & Cie, 1996); program(s)
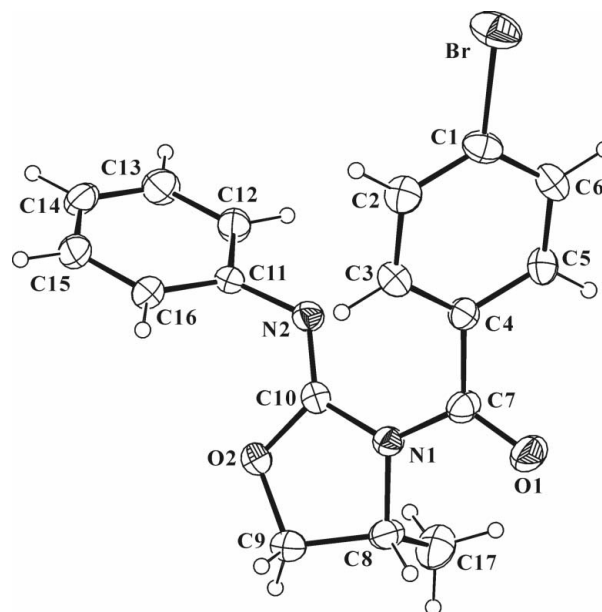

Figure 1
An ORTEP-3 (Farrugia, 1997) view of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids.

used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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