

Dmitry V. Albov,* Eugenia I.
Turubanova, Victor B. Rybakov,
Eugene V. Babaev and
Leonid A. AslanovDepartment of Chemistry, Moscow State
University, 119992 Moscow, Russian
FederationCorrespondence e-mail:
albov@biocryst.phys.msu.su

Key indicators

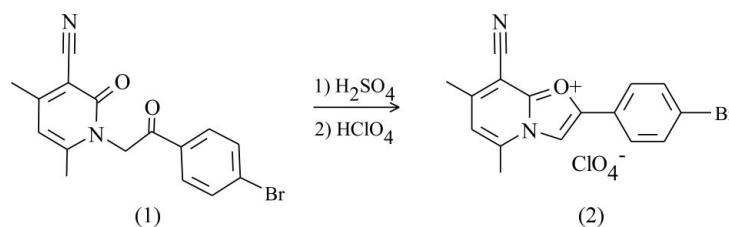
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.045
 wR factor = 0.132
Data-to-parameter ratio = 14.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.2-(4-Bromophenyl)-8-cyano-5,7-dimethyl-
oxazolo[3,2-*a*]pyridin-1-ylum perchlorateThe title compound, $\text{C}_{16}\text{H}_{12}\text{BrN}_2\text{O}^+\cdot\text{ClO}_4^-$, was synthesized,
and characterized by ^1H NMR and X-ray diffraction
techniques.

Received 22 June 2004

Accepted 30 June 2004

Online 9 July 2004

Comment

We have described earlier the crystal structure of *N*-(4-bromophenacyl)-4,6-dimethyl-2-oxo-1,2-dihydropyridine-2-carbonitrile, (1) (Albov *et al.*, 2004). Now, we report here the synthesis and crystal structure of the title compound, (2).

An analysis of bond lengths in the oxazolopyridinium ring system of (2) (Fig. 1) shows its aromaticity. The nine-membered bicyclic system is planar to within 0.018 (2) Å; atoms C61, C71, C91 and C10 lie in the same plane. The benzene ring is planar to within 0.007 (2) Å, with atoms Br1 and C2 in the same plane. The dihedral angle between the oxazolopyridinium and benzene fragments is 2.62 (7)°, indicating that there is considerable conjugation between these aromatic fragments.

Experimental

For the preparation of (2), compound (1) (0.5 g, 1.448 mmol) was dissolved in 20 ml of sulfuric acid and the reaction mixture was allowed to stand for 5 min. It was then poured into a mixture of 100 ml of water and 5 ml of a 70% solution of perchloric acid. The resulting precipitate was filtered off, washed with water and dried in air (yield 0.51 g, 82%; m.p. 512–514 K). The compound was recrystallized from acetonitrile. ^1H NMR (DMSO- d_6 , 400 MHz, p.p.m.): 2.86 (*s*, 3H, 5-CH₃), 3.05 (*s*, 3H, 5-CH₃), 8.0 (*s*, 1H, 6-CH), 7.82–7.84, 7.88–8.02 (*dd*, 4H, Ar), 9.6 (*s*, 1H, 3-CH).

Crystal data

 $\text{C}_{16}\text{H}_{12}\text{BrN}_2\text{O}^+\cdot\text{ClO}_4^-$ $M_r = 427.64$ Triclinic, $P\bar{1}$ $a = 7.150$ (3) Å $b = 7.800$ (2) Å $c = 15.481$ (5) Å $\alpha = 90.68$ (2)° $\beta = 102.62$ (3)° $\gamma = 98.48$ (3)° $V = 832.4$ (5) Å³ $Z = 2$ $D_x = 1.706$ Mg m⁻³Cu $K\alpha$ radiationCell parameters from 25
reflections $\theta = 32$ – 35 ° $\mu = 5.13$ mm⁻¹ $T = 293$ (2) K

Prism, yellow

0.30 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4
diffractometer
Non-profiled ω scans
Absorption correction: none
3288 measured reflections
3288 independent reflections
2825 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 74.8^\circ$
 $h = -8 \rightarrow 8$
 $k = -9 \rightarrow 9$
 $l = 0 \rightarrow 19$
1 standard reflection
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.132$
 $S = 1.06$
3288 reflections
228 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0738P)^2 + 0.5105P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.47 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Br1—C13	1.895 (3)	C8—C9	1.370 (4)
N1—C5	1.348 (3)	C9—C91	1.476 (4)
N1—C9	1.383 (4)	C10—C11	1.393 (4)
N1—C2	1.398 (4)	C10—C15	1.394 (4)
C2—C3	1.338 (4)	C11—C12	1.373 (5)
C3—O4	1.405 (3)	C12—C13	1.375 (5)
C3—C10	1.450 (4)	C13—C14	1.385 (5)
O4—C5	1.328 (4)	C14—C15	1.373 (5)
C5—C6	1.390 (4)	C61—N61	1.140 (4)
C6—C7	1.396 (4)	C11—O14	1.408 (3)
C6—C61	1.426 (4)	C11—O13	1.414 (3)
C7—C8	1.406 (4)	C11—O11	1.415 (3)
C7—C71	1.494 (4)	C11—O12	1.418 (3)
C5—N1—C9	122.0 (3)	C8—C9—C91	126.6 (3)
C5—N1—C2	107.8 (2)	N1—C9—C91	116.9 (3)
C9—N1—C2	130.2 (2)	C11—C10—C15	118.9 (3)
C3—C2—N1	106.7 (2)	C11—C10—C3	119.4 (3)
C2—C3—O4	108.8 (3)	C15—C10—C3	121.7 (3)
C2—C3—C10	133.9 (3)	C12—C11—C10	120.6 (3)
O4—C3—C10	117.3 (2)	C11—C12—C13	119.5 (3)
C5—O4—C3	106.7 (2)	C12—C13—C14	121.0 (3)
O4—C5—N1	110.0 (2)	C12—C13—Br1	119.4 (3)
O4—C5—C6	127.7 (3)	C14—C13—Br1	119.6 (3)
N1—C5—C6	122.3 (3)	C15—C14—C13	119.4 (3)
C5—C6—C7	117.4 (3)	C14—C15—C10	120.5 (3)
C5—C6—C61	119.8 (3)	N61—C61—C6	178.0 (4)
C7—C6—C61	122.8 (3)	O14—C11—O13	110.2 (2)
C6—C7—C8	118.7 (3)	O14—C11—O11	107.2 (2)
C6—C7—C71	120.4 (3)	O13—C11—O11	109.8 (2)
C8—C7—C71	120.9 (3)	O14—C11—O12	109.8 (3)
C9—C8—C7	123.1 (3)	O13—C11—O12	109.9 (2)
C8—C9—N1	116.5 (3)	O11—C11—O12	109.9 (3)

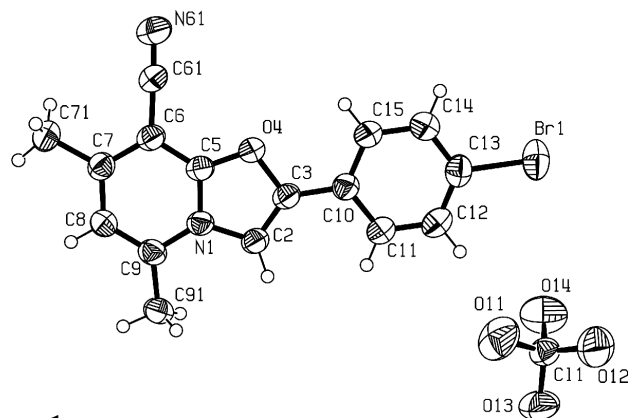


Figure 1
ORTEP-3 (Farrugia, 1997) plot of (2), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

H atoms were included in calculated positions ($C-H = 0.93-0.96 \text{ \AA}$) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$ or $1.2U_{\text{eq}}(\text{C})$ (others).

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* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors are indebted to the Russian Foundation for Basic Research for covering the licence fee for use of the Cambridge Structural Database (Allen, 2002) (project No. 02-07-90322).

References

Albov, D. V., Turubanova, E. I., Rybakov, V. B., Babaev, E. V. & Aslanov, L. A. (2004). *Acta Cryst.* **E60**, o1222–o1223.
Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.