# 2-(4-Bromophenyl)-8-cyano-5,7-dimethyl-oxazolo[3,2-a]pyridin-1-ylium perchlorate

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The title compound, $C_{16}H_{12}BrN_2O^+ \cdot ClO_4^-$, was synthesized, and characterized by $^1$H NMR and X-ray diffraction techniques.

## 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).
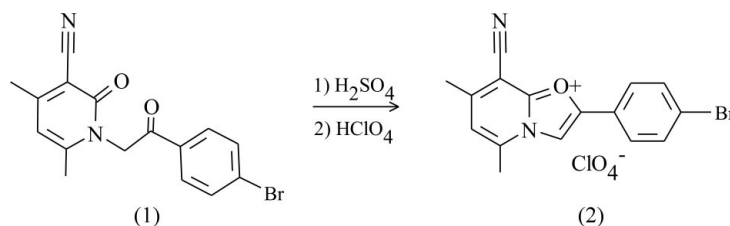


An analysis of bond lengths in the oxazolopyridinium ring system of (2) (Fig. 1) shows its aromaticity. The nine-membered bicyclic system is planar to within 0.018 (2) Å; atoms C61, C71, C91 and C10 lie in the same plane. The benzene ring is planar to within 0.007 (2) Å, with atoms Br1 and C2 in the 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. $^1$H NMR (DMSO-$d_6$, 400 MHz, p.p.m.): 2.86 (*s*, 3H, 5-CH$_3$), 3.05 (*s*, 3H, 5-CH$_3$), 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*

$C_{16}H_{12}BrN_2O^+ \cdot ClO_4^-$

$M_r = 427.64$

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) Å$^3$

$Z = 2$

$D_x = 1.706$ Mg m$^{-3}$

Cu $K\alpha$ radiation

Cell parameters from 25 reflections

$\theta = 32–35°$

$\mu = 5.13$ mm$^{-1}$

$T = 293$ (2) K

Prism, yellow

$0.30 \times 0.30 \times 0.30$ mm

# organic papers
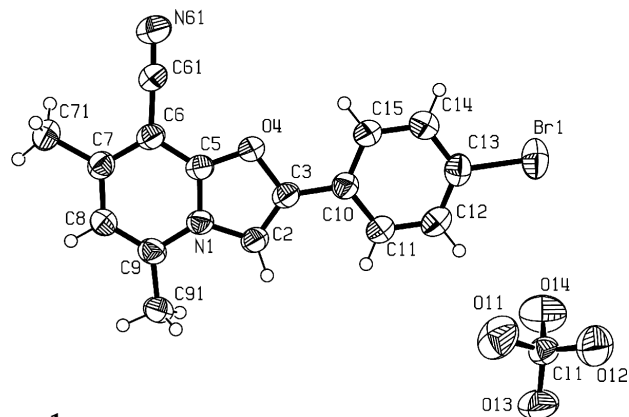
## Data collection

<table>
<tr><td>Enraf–Nonius CAD-4 diffractometer</td><td>$\theta_{max} = 74.8°$</td></tr>
<tr><td>Non-profiled $\omega$ scans</td><td>$h = -8 \rightarrow 8$</td></tr>
<tr><td>Absorption correction: none</td><td>$k = -9 \rightarrow 9$</td></tr>
<tr><td>3288 measured reflections</td><td>$l = 0 \rightarrow 19$</td></tr>
<tr><td>3288 independent reflections</td><td>1 standard reflection</td></tr>
<tr><td>2825 reflections with $I > 2\sigma(I)$</td><td> frequency: 60 min</td></tr>
<tr><td></td><td> intensity decay: 2%</td></tr>
</table>

## Refinement

<table>
<tr><td>Refinement on $F^2$</td><td>$w = 1/[\sigma^2(F_o^2) + (0.0738P)^2$</td></tr>
<tr><td>$R[F^2 > 2\sigma(F^2)] = 0.045$</td><td> $+ 0.5105P]$</td></tr>
<tr><td>$wR(F^2) = 0.132$</td><td> where $P = (F_o^2 + 2F_c^2)/3$</td></tr>
<tr><td>$S = 1.06$</td><td>$(\Delta/\sigma)_{max} < 0.001$</td></tr>
<tr><td>3288 reflections</td><td>$\Delta\rho_{max} = 0.35$ e Å$^{-3}$</td></tr>
<tr><td>228 parameters</td><td>$\Delta\rho_{min} = -0.47$ e Å$^{-3}$</td></tr>
<tr><td>H-atom parameters constrained</td><td></td></tr>
</table>

## Table 1
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| 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) | Cl1—O14 | 1.408 (3) |
| C6—C61 | 1.426 (4) | Cl1—O13 | 1.414 (3) |
| C7—C8 | 1.406 (4) | Cl1—O11 | 1.415 (3) |
| C7—C71 | 1.494 (4) | Cl1—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—Cl1—O13 | 110.2 (2) |
| C6—C7—C8 | 118.7 (3) | O14—Cl1—O11 | 107.2 (2) |
| C6—C7—C71 | 120.4 (3) | O13—Cl1—O11 | 108.8 (2) |
| C8—C7—C71 | 120.9 (3) | O14—Cl1—O12 | 109.8 (2) |
| C9—C8—C7 | 123.1 (3) | O13—Cl1—O12 | 109.9 (2) |
| C8—C9—N1 | 116.5 (3) | O11—Cl1—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 Å) and refined as riding, with $U_{iso}(H) = 1.5U_{eq}$(methyl C) or $1.2U_{eq}$(C) (others).

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

## References

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