

## 2-(4-Chlorophenyl)-5-methyl-7,8,9,10-tetrahydro-6H-cyclohepta[e][1,3]-oxazolo[3,2-a]pyridin-11-ium perchlorate

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### Key indicators

Single-crystal X-ray study  
 T = 293 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$   
 R factor = 0.042  
 wR factor = 0.107  
 Data-to-parameter ratio = 15.9

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

The title compound,  $\text{C}_{19}\text{H}_{19}\text{ClNO}^+\cdot\text{ClO}_4^-$ , was synthesized and characterized by  $^1\text{H}$  NMR and X-ray diffraction techniques.

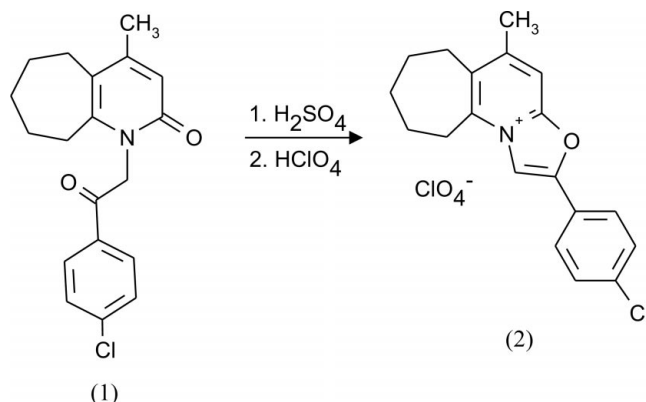
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### Comment

In the course of systematic investigations of the effect of the size of cycloalkane fragments on the reactivity of the corresponding heterocycles based on pyridine, we have previously described the crystal structure of 1-(4-chlorophenacyl)-4-methyl-1,5,6,7,8,9-hexahydro-2H-cyclohepta[b]pyridin-2-one, (1) (Albov *et al.*, 2004a). Following a corresponding study with cyclohexene derivatives (Albov *et al.*, 2004b), we synthesized the title compound (2).



An analysis of bond lengths in the oxazopyridinium ring system of (2) (Fig. 1) shows its aromaticity. The nine-membered bicyclic system is planar to within  $0.0216(9) \text{ \AA}$ : atoms C10, C11, C15 and C16 lie in the same plane, while atoms C12, C13 and C14 are displaced from that plane by  $1.421(3)$ ,  $1.507(3)$  and  $1.322(2) \text{ \AA}$ , respectively. The cycloheptene ring has a chair conformation. The benzene ring is planar to within  $0.0033(9) \text{ \AA}$ , with atoms C11 and C3 in the same plane. The dihedral angle between the oxazopyridinium and benzene fragments is  $3.77(7)^\circ$ , indicating that there is considerable conjugation between these aromatic fragments.

All these results will be compared with the crystal structures of five- and eight-membered cycloalkane derivatives which are in progress, as well as with published (Albov *et al.*, 2004b) six-membered cycloalkane derivatives.

### Experimental

For the preparation of (2), 1.80 g of (1) was dissolved in 15 ml of sulfuric acid and kept overnight. The solution was then poured into 100 ml of a 3% aqueous solution of perchloric acid. A white precipi-

pitite formed and the mixture was kept overnight again for complete precipitation. The product was filtered off and washed with water and acetone (yield 2.15 g, 96%). It was recrystallized from acetonitrile (m.p. 573 K with explosion).  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz, p.p.m.): 1.70 (m, 2H, 13-CH<sub>2</sub>), 1.83 (m, 2H, 12-CH<sub>2</sub>), 1.99 (m, 2H, 14-CH<sub>2</sub>), 2.67 (s, 3H, 10-CH<sub>3</sub>), 3.05 (t, 2H, 11-CH<sub>2</sub>), 3.44 (t, 2H, 15-CH<sub>2</sub>), 7.65, 8.00 (dd, 4H, Ar), 8.08 (s, 1H, 6-CH), 9.58 (s, 1H, 2-CH).

#### Crystal data

$\text{C}_{19}\text{H}_{19}\text{ClNO}^+\cdot\text{ClO}_4^-$	$D_x = 1.442 \text{ Mg m}^{-3}$
$M_r = 412.25$	Cu $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
$a = 7.894 (3) \text{ \AA}$	$\theta = 33\text{--}35^\circ$
$b = 18.492 (3) \text{ \AA}$	$\mu = 3.35 \text{ mm}^{-1}$
$c = 13.271 (2) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 101.46 (2)^\circ$	Prism, colourless
$V = 1898.6 (8) \text{ \AA}^3$	$0.20 \times 0.20 \times 0.20 \text{ mm}$
$Z = 4$	

#### Data collection

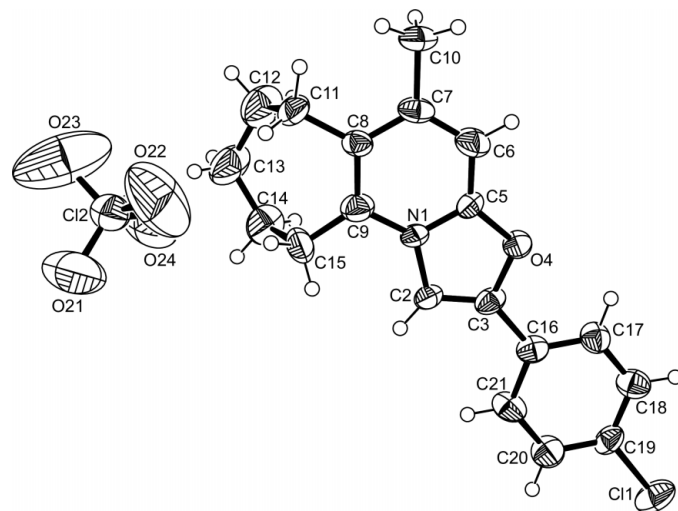
Enraf-Nonius CAD-4 diffractometer	$\theta_{\text{max}} = 75.0^\circ$
Non-profiled $\omega$ scans	$h = -9 \rightarrow 9$
Absorption correction: none	$k = 0 \rightarrow 23$
4010 measured reflections	$l = 0 \rightarrow 16$
3901 independent reflections	1 standard reflection every 200 reflections
3310 reflections with $I > 2\sigma(I)$	intensity decay: 4%
$R_{\text{int}} = 0.004$	

#### Refinement

Refinement on $F^2$	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0892P)^2]$
$wR(F^2) = 0.107$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{\text{max}} = 0.001$
3901 reflections	$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$
246 parameters	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$

**Table 1**  
Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Cl1–C19	1.729 (1)	C8–C9	1.367 (2)
O4–C5	1.337 (1)	C8–C11	1.516 (2)
O4–C3	1.394 (1)	C9–C15	1.476 (2)
N1–C5	1.342 (1)	C11–C12	1.528 (3)
N1–C9	1.384 (1)	C12–C13	1.510 (3)
N1–C2	1.402 (1)	C13–C14	1.501 (3)
C2–C3	1.345 (2)	C14–C15	1.529 (2)
C3–C16	1.455 (2)	Cl2–O21	1.364 (2)
C5–C6	1.382 (2)	Cl2–O22	1.372 (2)
C6–C7	1.369 (2)	Cl2–O23	1.400 (2)
C7–C8	1.427 (2)	Cl2–O24	1.417 (2)
C7–C10	1.517 (2)		
C5–O4–C3	106.7 (1)	C7–C6–C5	117.4 (1)
C5–N1–C9	122.0 (1)	C6–C7–C8	120.0 (1)
C5–N1–C2	107.4 (1)	C6–C7–C10	119.2 (1)
C9–N1–C2	130.6 (1)	C8–C7–C10	120.9 (1)
C3–C2–N1	106.8 (1)	C9–C8–C7	121.1 (1)
C2–C3–O4	108.8 (1)	C9–C8–C11	117.6 (1)
C2–C3–C16	133.7 (1)	C7–C8–C11	121.3 (1)
O4–C3–C16	117.5 (1)	C8–C9–N1	117.1 (1)
O4–C5–N1	110.3 (1)	C8–C9–C15	124.5 (1)
O4–C5–C6	127.3 (1)	N1–C9–C15	118.4 (1)
N1–C5–C6	122.4 (1)	C8–C11–C12	112.6 (1)



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

H atoms were included in calculated positions (C–H = 0.93–0.97  $\text{\AA}$ ) and refined as riding, with  $U_{\text{iso}} = 1.5$  (methyl H) or  $1.2U_{\text{eq}}$ (carrier atom).

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

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