

2-(4-Chlorophenyl)-5-methyl-7,8-dihydro-6H-cyclopenta[e][1,3]oxazolo[3,2-a]pyridin-9-ium perchlorate

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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.045
 wR factor = 0.102
Data-to-parameter ratio = 15.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{17}\text{H}_{15}\text{ClNO}^+\cdot\text{ClO}_4^-$, has been synthesized and characterized by ^1H NMR and X-ray diffraction techniques. The bicyclic system is aromatic, with positively charged nitrogen, and is conjugated with the benzene ring.

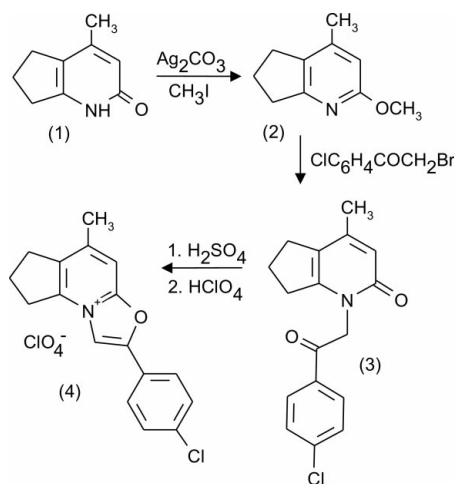
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Comment

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



An analysis of bond lengths in the oxazolopyridinium ring of (4) (Fig. 1 and Table 1) reveals that this bicyclic system is certainly aromatic, with the positive charge located on atom N1. The nine-membered bicyclic system is planar to within 0.0127 (11) Å, with atoms C10, C12, C13 and C14 lying in the same plane. Atom C11 is displaced from this plane by 0.187 (2) Å. The dihedral angle between the oxazolopyridinium and benzene fragments is 4.82 (6)°, indicating that there is considerable conjugation between these aromatic fragments.

All these results will be compared with crystal structures of other six-, seven- and eight-membered cycloalkane derivatives which are in progress.

Experimental

The title compound was prepared according to the method of Albov, Mazina *et al.* (2004) (m.p. 571 K, with explosion). ^1H NMR (DMSO-

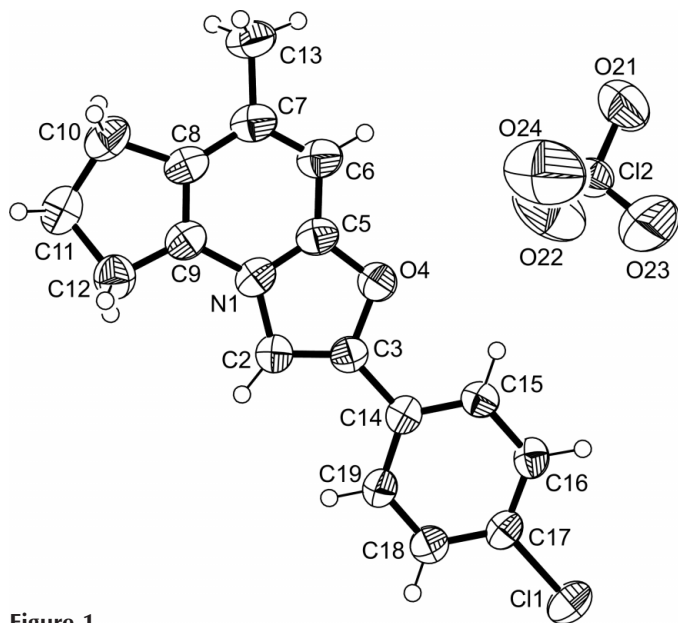


Figure 1
ORTEP-3 (Farrugia, 1997) plot of the molecule and atom-numbering scheme of compound (4). Displacement ellipsoids are drawn at the 50% probability level.

d_6 , 400 MHz, p.p.m.): 2.33 (*m*, 2H, 11-CH₂), 2.62 (*s*, 3H, 13-CH₃), 3.17 (*t*, 2H, 10-CH₂), 3.45 (*t*, 2H, 12-CH₂), 7.64, 8.01 (*dd*, 4H, Ar), 8.04 (*s*, 1H, 6-CH), 9.33 (*s*, 1H, 2-CH) (using the crystallographic numbering scheme of Fig. 1).

Crystal data

C₁₇H₁₅ClNO⁺·ClO₄⁻
M_r = 384.20
 Monoclinic, *P*₂₁/*c*
a = 12.631 (5) Å
b = 8.329 (5) Å
c = 17.982 (8) Å
 β = 119.11 (3)°
V = 1652.8 (14) Å³
Z = 4

D_x = 1.544 Mg m⁻³
 Cu *K*α radiation
 Cell parameters from 25 reflections
 θ = 25–26°
 μ = 3.80 mm⁻¹
T = 293 (2) K
 Prism, colourless
 0.22 × 0.21 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Non-profiled ω scans
 Absorption correction: none
 3411 measured reflections
 3411 independent reflections
 2899 reflections with *I* > 2σ(*I*)

θ_{\max} = 74.8°
h = -15 → 13
k = 0 → 10
l = 0 → 22
 1 standard reflection
 frequency: 30 min
 intensity decay: 2%

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.045
wR(*F*²) = 0.102
S = 0.79
 3411 reflections
 227 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1086P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.24 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.29 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C11–C17	1.7572 (13)	C7–C13	1.499 (2)
O4–C5	1.3432 (16)	C8–C9	1.3461 (19)
O4–C3	1.3959 (16)	C8–C10	1.491 (2)
N1–C5	1.3225 (18)	C9–C12	1.430 (2)
N1–C2	1.4136 (16)	C10–C11	1.538 (3)
N1–C9	1.4153 (16)	C11–C12	1.551 (2)
C2–C3	1.3364 (17)	C12–O23	1.3231 (19)
C3–C14	1.4608 (17)	C12–O24	1.3574 (19)
C5–C6	1.3986 (19)	C12–O22	1.3647 (17)
C6–C7	1.347 (2)	C12–O21	1.4066 (16)
C7–C8	1.422 (2)		
C5–O4–C3	106.04 (10)	C6–C7–C8	119.50 (12)
C5–N1–C2	109.70 (10)	C6–C7–C13	120.19 (14)
C5–N1–C9	118.66 (11)	C8–C7–C13	120.32 (13)
C2–N1–C9	131.62 (11)	C9–C8–C7	122.56 (13)
C3–C2–N1	104.22 (11)	C9–C8–C10	108.31 (13)
C2–C3–O4	110.59 (11)	C7–C8–C10	128.96 (13)
C2–C3–C14	132.91 (11)	C8–C9–N1	117.50 (13)
O4–C3–C14	116.50 (10)	C8–C9–C12	117.82 (13)
N1–C5–O4	109.40 (11)	N1–C9–C12	124.50 (12)
N1–C5–C6	125.25 (13)	C8–C10–C11	103.39 (13)
O4–C5–C6	125.30 (13)	C10–C11–C12	107.23 (13)
C7–C6–C5	116.49 (14)	C9–C12–C11	100.52 (13)

All H atoms were placed in calculated positions and refined as riding atoms, with C–H bond lengths in the range 0.93–0.97 Å. For methyl H atoms, *U*_{iso} values were set equal to 1.5*U*_{eq}(C), and for other H atoms to 1.2*U*_{eq}(C).

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 publication routines (Farrugia, 1999).

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