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4-Amino-6-chloro-8-*p*-tolylpteridin-7(8*H*)-one dichloromethane hemisolvate

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The title compound, $C_{13}H_{10}ClN_5O\cdot0.5CH_2Cl_2$, crystallizes with two independent 4-amino-6-chloro-8-*p*-tolylpteridin-7(8*H*)-one molecules and one dichloromethane molecule in the asymmetric unit. The bond lengths and angles in the molecules are within normal ranges. Intermolecular N—H···N hydrogen bonds link the two independent molecules into hydrogen-bonded dimers. The crystal packing is further stabilized by van der Waals forces.

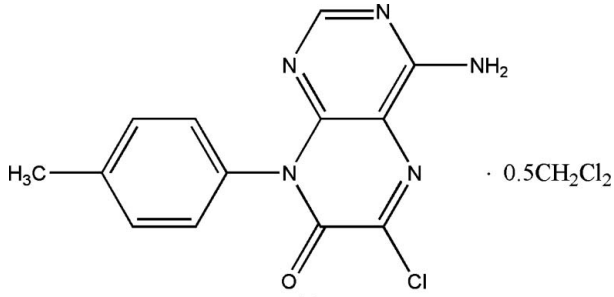
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.054
wR factor = 0.145
Data-to-parameter ratio = 13.2

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

Comment

Pteridine-like molecules are reported to be adenosine kinase inhibitors with modest potency (Perner *et al.*, 2003). In order to find novel compounds with high potency and high membrane permeability, several C6- and N8-substituted 4-aminopteridin-7(8*H*)-one compounds have been designed. Compound 4-amino-6-chloro-8-*p*-tolylpteridin-7(8*H*)-one is one of the key intermediates. In this paper, we report the crystal structure of the title compound, (I).



(I)

Compound (I) crystallizes with two independent 4-amino-6-chloro-8-*p*-tolylpteridin-7(8*H*)-one molecules (Fig. 1) and one dichloromethane molecule in the asymmetric unit. The bond lengths and angles in the molecules are within normal ranges (Allen *et al.*, 1987). Atoms Cl1, O1 and N5 lie in the mean plane (P1) of the C1–C6/N1–N4 7,8-dihydropteridine ring, the largest deviation being 0.062 (2) Å for atom N5. The dihedral angle between plane P1 and the C7–C12 benzene ring is 80.86 (3)°. Atoms Cl2, O2, N10 and C20 lie in the mean plane (P2) of the C14–C19/N6–N9 7,8-dihydropteridine ring, the largest deviation being 0.039 (4) Å for atom N10. The dihedral angle between plane P2 and the C20–C25 benzene ring is 71.67 (2)°.

Intermolecular N—H···N hydrogen bonds (Table 1) link two independent molecules into hydrogen-bonded dimers. The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

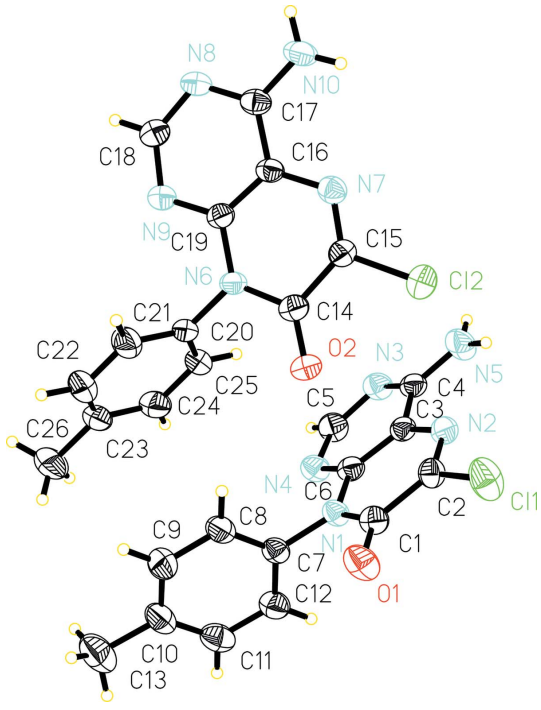


Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 40% probability level. The solvent molecule has been omitted for clarity.

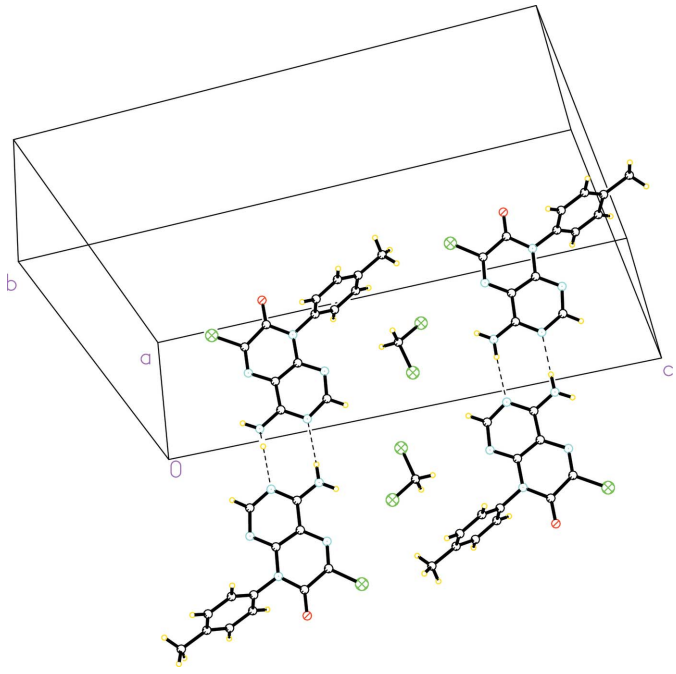


Figure 2
A partial packing diagram, showing the hydrogen-bonded (dashed lines) dimers.

Experimental

4-Amino-8-*p*-tolylpteridine-6,7(5*H*,8*H*)-dione (2.69 g, 0.01 mol), phosphorus pentachloride (4.16 g, 0.02 mol) and phosphorus oxychloride (20 ml) were heated to reflux for 50 min. The mixture was concentrated to 10 ml *in vacuo*. After cooling to room temperature, chloroform (100 ml) was added. Water (100 ml) was then added dropwise while keeping the temperature below 293 K, and the mixture was stirred for 2 h. The organic layer was separated and the aqueous layer was extracted with chloroform. The combined organic layers were dried with anhydrous magnesium sulfate and concentrated *in vacuo*, and the residue was chromatographed on silica gel (ethyl acetate) to give 4-amino-6-chloro-8-*p*-tolylpteridin-7(8*H*)-one in 60% yield. Crystals of (I) suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution of 4-amino-6-chloro-8-*p*-tolylpteridin-7(8*H*)-one in dichloromethane at room temperature over a period of one week.

Crystal data

$C_{13}H_{10}ClN_5O\cdot0.5CH_2Cl_2$
M_r = 330.18
Monoclinic, $P2_1/c$
a = 7.4644 (14) Å
b = 16.138 (3) Å
c = 24.402 (5) Å
β = 97.676 (3)°
V = 2913.1 (10) Å^3
Z = 8
D_x = 1.506 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 298 (2) K
Block, colourless
0.24 × 0.18 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.899, T_{max} = 0.935
14411 measured reflections
5123 independent reflections
3183 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.145
S = 1.01
5123 reflections
389 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.1625P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5B···N8^{i}	0.86	2.08	2.937 (4)	179
N10—H10A···N3^{ii}	0.86	2.23	3.089 (4)	176

Symmetry codes: (i) $-x-1, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x-1, y+\frac{1}{2}, -z+\frac{1}{2}$.

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å and N—H = 0.86 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) for the aryl, methine and N-bound H atoms, and 1.5U_{eq}(C) for the methyl H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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