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5-(8-Hydroxyquinolyl)guanidinium dichloride

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.045
wR factor = 0.139
Data-to-parameter ratio = 15.4

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

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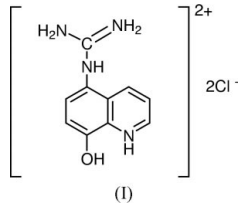
The title compound, $C_{10}H_{12}N_4O^{2+}\cdot 2Cl^-$, was synthesized by the reaction of 5-amino-8-hydroxyquinoline and cyanamide. The plane of the guanidinium group is perpendicular to the quinoline ring system, with a dihedral angle of 83.52 (10)°. N—H···Cl and O—H···Cl hydrogen bonds form a three-dimensional network.

Comment

Guanidine is found in many natural products (Manimala & Anslyn, 2002). Guanidine compounds, especially those containing a quinolyl ring, and their salts are useful in the treatment of gastrointestinal motility disorders (Kuhla *et al.*, 1986). These important compounds are therefore of interest from a structural viewpoint. In this paper, we report the crystal structure of the title compound, (I).



(I)

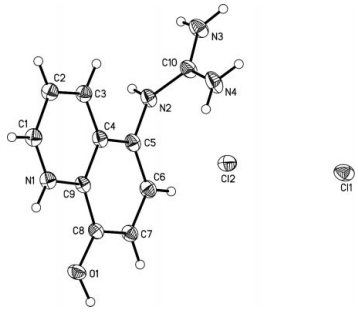


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Atom N1 and the guanidine group are protonated (Fig. 1 and Table 1). The plane O1/C7/C8/C9 is parallel to the quinoline plane (N1/C1–C9), forming a dihedral angle of 0.38 (19)°, while the plane of the guanidinium moiety (N2/N3/N4/C10) is perpendicular to the quinoline plane, with a dihedral angle of 83.52 (10)°. Because of the conjugation in the guanidinium moiety, the N2—C10, N3—C10 and N4—C10 bond distances are 1.363 (4) Å, 1.326 (4) Å and 1.316 (4) Å, respectively. The two Cl^- ions are bound to the H atoms of the guanidinium and hydroxy groups through hydrogen bonds, forming a three-dimensional network (Fig. 2 and Table 2).

Experimental

The title compound was synthesized according to the method described by Hughes & Liu (1976). A mixture of 5-amino-8-hydroxyquinoline, concentrated hydrochloric acid, cyanamide and ethyl alcohol was heated at reflux for 3 h, with stirring. The reaction mixture was evaporated to give a residue. This residue was dissolved in hot methanol and then left to stand at 273 K for 7 h to yield the title compound. Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{10}H_{12}N_4O^{2+}\cdot 2Cl^-$
$M_r = 275.14$
Monoclinic, $C2/c$
$a = 10.715$ (2) Å
$b = 11.333$ (2) Å
$c = 22.720$ (5) Å
$\beta = 103.30$ (3)°
$V = 2685.0$ (10) Å^3
$Z = 8$
$D_x = 1.361$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 2.1$–25.3°
$\mu = 0.47$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.30 × 0.25 × 0.22 mm

Data collection

Siemens *P*4 diffractometer
$2\theta/\omega$ scans
Absorption correction: ψ scan (*XPREP* in *SHELXTL*; Bruker, 2000)
$T_{min} = 0.799$, $T_{max} = 0.902$
2501 measured reflections
2366 independent reflections
1885 reflections with $I > 2\sigma(I)$
$R_{int} = 0.012$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = -26 \rightarrow 26$
3 standard reflections
every 97 reflections
intensity decay: 2.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.139$
$S = 1.05$
2366 reflections
154 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.09P)^2 + 1.95P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.71$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.300 (4)	C9—N1	1.426 (4)
C3—C4	1.388 (4)	C10—N4	1.316 (4)
C4—C9	1.413 (4)	C10—N3	1.326 (4)
C5—N2	1.423 (4)	C10—N2	1.363 (4)
C8—O1	1.349 (3)		
C3—C4—C5	124.4 (3)	C8—C9—C4	124.5 (3)
C4—C5—N2	119.3 (3)	C8—C9—N1	119.1 (2)
O1—C8—C7	123.6 (3)	N4—C10—N2	118.8 (3)

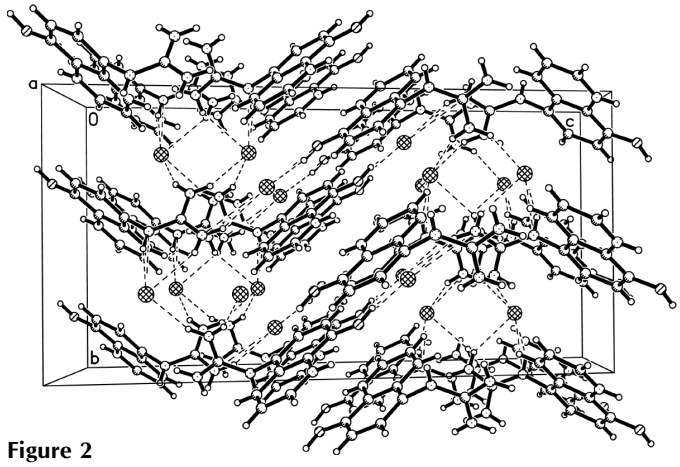


Figure 2
The crystal structure of (I). Dashed lines indicate hydrogen bonds.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Cl1^{i}	0.86	2.43	3.250 (3)	160
N3—H3B···Cl1^{i}	0.86	2.58	3.362 (3)	152
N3—H3A···Cl2^{ii}	0.86	2.50	3.277 (3)	150
N4—H4A···Cl1^{ii}	0.86	2.61	3.293 (3)	137
N4—H4B···Cl1^{iii}	0.86	2.54	3.320 (3)	152
O1—H1C···Cl2^{iv}	0.96	2.08	3.033 (2)	170

Symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}+y, z$; (ii) $1-x, y, \frac{1}{2}-z$; (iii) $1+x, y, z$; (iv) $\frac{3}{2}-x, \frac{3}{2}-y, 1-z$.

All H atoms were positioned geometrically (N—H = 0.86, O—H = 0.96 and C—H = 0.93 Å) and treated as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *XSCANS* (Bruker, 2000); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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