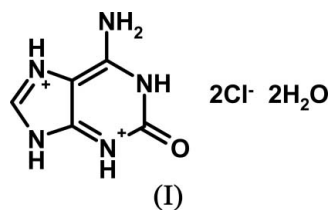


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bats@chemie.uni-frankfurt.de**Key indicators**Single-crystal X-ray study
 $T = 154$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.042
 wR factor = 0.113
Data-to-parameter ratio = 17.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**Isoguanine dihydrochloride dihydrate**

The crystal structure of the title compound, 6-amino-2-oxo-1,2-dihydro-9*H*-purine-3,7-diium dichloride dihydrate, $\text{C}_5\text{H}_7\text{N}_5\text{O}^{2+} \cdot 2\text{Cl}^- \cdot 2\text{H}_2\text{O}$, consists of hydrogen-bonded layers parallel to $(10\bar{1})$. In addition, pairs of layers are connected by an $\text{O}-\text{H} \cdots \text{Cl}$ hydrogen bond and there are also short $\text{Cl} \cdots \text{C}$ and $\text{Cl} \cdots \text{N}$ contacts between the layers.

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Isoguanine is a modified nucleic acid base that is produced by oxidative stress of adenine (Kamiya, 2003). It has interesting biological and chemical properties. It shows high mutagenic potential (Kamiya, 2003), exists in different tautomeric forms (Blas *et al.*, 2004; Rogstad *et al.*, 2003), forms different base aggregates (Gu *et al.*, 2004) and builds a stable complex with isocytosine (Chen *et al.*, 2001).



Isoguanine was unexpectedly formed by heating 2-fluoro-adenine in 1 *N* HCl. We report here the crystal structure of isoguanine dihydrochloride dihydrate, (I). A view of part of the crystal structure of (I), showing the atomic numbering scheme in the asymmetric unit, is shown Fig. 1. All N atoms are protonated. The isoguanine group is essentially planar and the average deviation of the non-H atoms from the mean plane is 0.020 Å. The largest torsion angle in the six-membered ring is 4.4 (3)° about the C2–N3 bond, while the five-membered ring is planar within experimental error. The bond distances in the isoguanine group are very similar to those observed in the crystal structure of isoguanine sulfate (Subramanian & Marsh, 1971), although the cation in that structure is not protonated at N9. The significant differences between the two structures occur for the bond angles in the five-membered ring. The bond angles at N7 and N9 are larger and those at C4 and C8 are smaller in the double-protonated cation in (I).

The molecules are connected by intermolecular hydrogen bonding into layers parallel to the crystallographic $(10\bar{1})$ plane. A part of such a layer is shown in Fig. 1 and details of the hydrogen bonding are given in Table 1. Each layer is also stabilized by a weak $\text{C}-\text{H} \cdots \text{Cl}$ and a weak $\text{C}-\text{H} \cdots \text{O}$ contact, both involving the C8–H8A bond. The distance

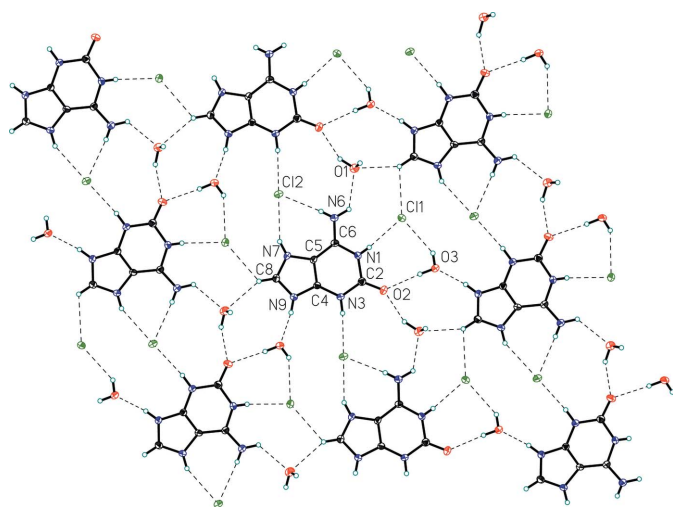


Figure 1
Hydrogen-bonded layer of (I) parallel to the crystallographic $(10\bar{1})$ plane with the atomic numbering scheme shown for the asymmetric unit. The displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as small spheres of arbitrary radii and hydrogen bonds are shown as dashed lines.

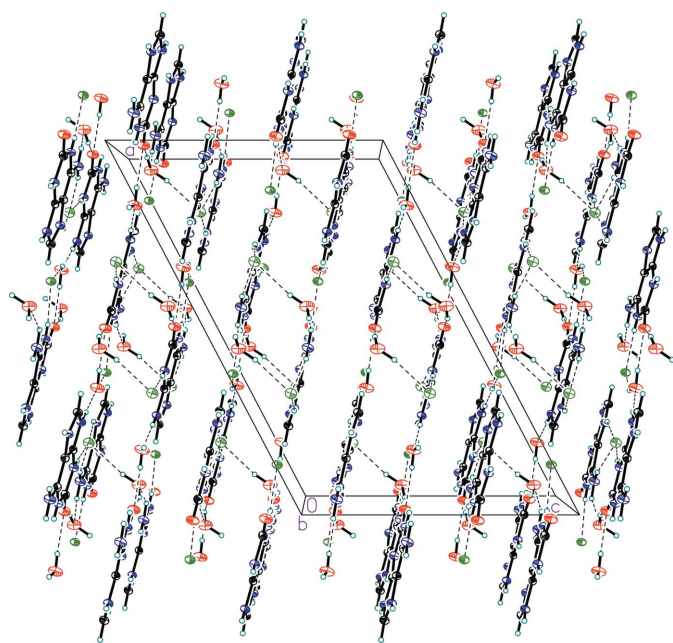


Figure 2
Crystal packing of (I), viewed along b , with hydrogen bonds shown as dashed lines.

between neighboring layers is approximately 3.20 \AA . There are no π - π interactions between the purine groups. Adjacent layers are connected in pairs by symmetry-related hydrogen bonds of type $\text{O1}-\text{H1B}\cdots\text{Cl2}^{\text{iii}}$ [symmetry code: (iii) $1-x, y, \frac{3}{2}-z$], as shown in Fig. 2. The layers also show weak intermolecular contacts between Cl anions and C-N bonds. The shortest contacts are $\text{Cl1}\cdots\text{C5}^{\text{ii}} = 3.311(2) \text{ \AA}$, $\text{Cl1}\cdots\text{N7}^{\text{iii}} = 3.441(2) \text{ \AA}$, $\text{Cl1}\cdots\text{C2}^{\text{vi}} = 3.285(3) \text{ \AA}$, $\text{Cl1}\cdots\text{N3}^{\text{vi}} = 3.342(2) \text{ \AA}$, $\text{Cl2}\cdots\text{C8}^{\text{vii}} = 3.320(3) \text{ \AA}$ and $\text{Cl2}\cdots\text{N9}^{\text{vii}} = 3.484(2) \text{ \AA}$ [symmetry codes: (iii) $1-x, y, \frac{3}{2}-z$; (vi) $1-x,$

$1-y, 1-z$; (vii) $\frac{3}{2}-x, -\frac{1}{2}+y, \frac{3}{2}-z$]. The distances between the Cl atoms and the mid-points of the C-N bonds are $3.30, 3.24$ and 3.34 \AA . The crystal packing in (I) is comparable to that in adenine dihydrochloride (Lewis & Tocher, 2005) and guanine dihydrochloride (Matković-Čalogović & Sanković, 1999). In these crystal structures, hydrogen bonded sheets are also formed. The separation between the sheets is 3.24 and 3.29 \AA respectively. The shortest intermolecular contacts between the sheets are $\text{Cl}\cdots\text{C}$ contacts of 3.25 and 3.30 \AA , respectively. Thus interactions between Cl ions and purine C-N bonds seems to play a significant role in the packing of these crystal structures.

Experimental

The title compound was prepared by heating a solution of commercially available 2-fluoroadenine (Aldrich) in 1 N HCl . Single crystals were obtained by slow evaporation of the solvent.

Crystal data

$\text{C}_5\text{H}_7\text{N}_5\text{O}_2^+ \cdot 2\text{Cl}^- \cdot 2\text{H}_2\text{O}$
 $M_r = 260.09$
 Monoclinic, $C2/c$
 $a = 19.896(3) \text{ \AA}$
 $b = 9.120(3) \text{ \AA}$
 $c = 13.077(3) \text{ \AA}$
 $\beta = 117.713(16)^\circ$
 $V = 2100.7(10) \text{ \AA}^3$

$Z = 8$
 $D_x = 1.645 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.62 \text{ mm}^{-1}$
 $T = 154(2) \text{ K}$
 Parallelepiped, colorless
 $0.44 \times 0.26 \times 0.11 \text{ mm}$

Data collection

Siemens SMART 1K CCD
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2000)
 $T_{\text{min}} = 0.846, T_{\text{max}} = 0.935$

13739 measured reflections
 3248 independent reflections
 2438 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.047$
 $\theta_{\text{max}} = 31.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.113$
 $S = 1.04$
 3248 reflections
 181 parameters

All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.52 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.33 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{N1}-\text{H1A}\cdots\text{Cl1}$ | 0.73 (3) | 2.29 (3) | 3.017 (2) | 172 (3) |
| $\text{N3}-\text{H3A}\cdots\text{Cl2}^{\text{i}}$ | 0.87 (3) | 2.27 (3) | 3.130 (2) | 173 (2) |
| $\text{N6}-\text{H6A}\cdots\text{Cl2}$ | 0.77 (3) | 2.39 (3) | 3.154 (3) | 170 (3) |
| $\text{N6}-\text{H6B}\cdots\text{O1}$ | 0.89 (3) | 2.19 (3) | 2.818 (3) | 128 (2) |
| $\text{N7}-\text{H7A}\cdots\text{Cl2}$ | 0.84 (3) | 2.49 (3) | 3.253 (2) | 151 (3) |
| $\text{N9}-\text{H9A}\cdots\text{O3}^{\text{ii}}$ | 0.83 (3) | 1.82 (3) | 2.637 (3) | 171 (3) |
| $\text{O1}-\text{H1B}\cdots\text{Cl2}^{\text{iii}}$ | 0.95 (3) | 2.30 (4) | 3.240 (3) | 169 (3) |
| $\text{O1}-\text{H1C}\cdots\text{O2}^{\text{iv}}$ | 0.92 (4) | 1.98 (4) | 2.827 (3) | 153 (4) |
| $\text{O3}-\text{H3B}\cdots\text{O2}$ | 0.85 (3) | 2.07 (3) | 2.919 (3) | 176 (3) |
| $\text{O3}-\text{H3C}\cdots\text{Cl1}$ | 0.80 (4) | 2.45 (5) | 3.140 (2) | 145 (4) |
| $\text{C8}-\text{H8A}\cdots\text{Cl1}^{\text{v}}$ | 0.91 (3) | 2.68 (3) | 3.240 (2) | 121 (2) |
| $\text{C8}-\text{H8A}\cdots\text{O1}^{\text{v}}$ | 0.91 (3) | 2.35 (3) | 3.161 (3) | 147 (2) |

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

H atoms were located in a difference Fourier map and were refined with individual isotropic displacement parameters ($X-H$ distances

are given in Table 1). The crystal was twinned. The twin relations are $h_{\text{twin}} = -h - 1.41l$, $k_{\text{twin}} = -k$ and $l_{\text{twin}} = l$. Reflections from the twin domain were considered to coincide with reflections of the main domain if their positions differ by less than 0.25 reciprocal lattice units. The twin fraction refined to 0.174 (2).

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINTE* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXL97*.

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