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Redetermination of guaninium chloride dihydrate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.082
Data-to-parameter ratio = 13.4

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

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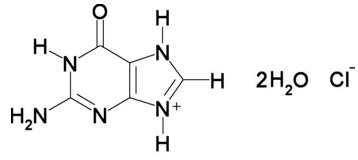
The low-temperature redetermination of guaninium chloride dihydrate, $C_5H_6N_5O^+\cdot Cl^-\cdot 2H_2O$, obtained as part of an experimental polymorph screen on guanine, is reported here.

Comment

The title compound, (I), is a dihydrate salt of guanine, which is one of the two common purine bases found in ribose and deoxyribonucleic acids. The unit cell and space group of (I) were originally reported in 1951 (Broomhead, 1951), with a room-temperature X-ray determination performed 12 years later (Iball & Wilson, 1963, 1965). In this original determination, the intensities were recorded using Weissenberg photographs. All the atoms, including H atoms, were located by means of a difference Fourier synthesis and the structure refined to a final *R* value of 0.073. We have redetermined this crystal structure at 150 K, with a final *R* value of 0.032, to gain more precise data for our theoretical modelling studies.



(I)

In this low-temperature determination, the precision of the unit-cell dimensions was improved by an order of magnitude, and the unit-cell volume decreased by *ca* 14 Å^3, consistent with the determination at low temperature. In general, the

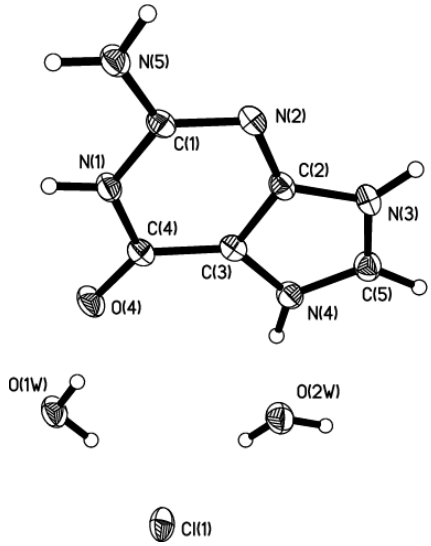


Figure 1
View of (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

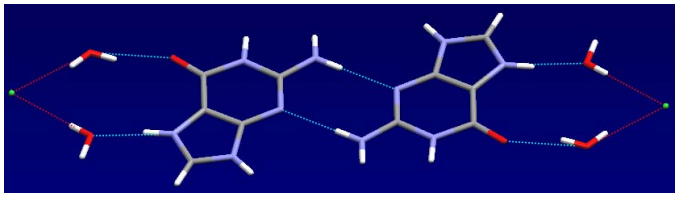


Figure 2
The hydrogen-bonded (dashed lines) planar unit in (I), showing the centrosymmetric dimer linked to four water molecules and two Cl^- ions. The other hydrogen bonds have been omitted for clarity.

metric parameters are not significantly different, the exception being the C1—N2 bond length which is longer in the low-temperature structure, while C1—N5 is shorter in the low-temperature structure, both by *ca* 0.03 Å. The guanine molecule is protonated at N1 and N4, with the C—N bond lengths in the rings ranging from 1.3154 (18) to 1.3892 (18) Å, and the C2—C3, C3—C4 and N5—C1 bond lengths being 1.3797 (18), 1.4202 (19) and 1.3291 (18) Å, respectively.

The packing consists of centrosymmetric dimers, the two components of which are linked by pairs of N—H···N hydrogen bonds. These dimers are linked to four water molecules and two Cl atoms to form a planar unit (Fig. 2). These planar units are linked to one another through O—H···Cl hydrogen bonds within the plane, forming a ribbon structure, and through N—H···Cl and N—H···O hydrogen bonds at an angle of approximately 80° from this plane, forming a complex three-dimensional hydrogen-bonded network (Fig. 3). The two H atoms on the NH_2 group form two very dissimilar hydrogen bonds. A strong bond [N5—H7···N2^{iv} = 3.0162 (17) Å; see Table 1] is formed by one, while the second [N5—H6···Cl1^{i} = 3.4368 (15) Å; see Table 1] is weak.The N—H···N distance within the centrosymmetric dimer is 3.0162 (17) Å, with the N—H···O distances ranging from 2.6463 (17) to 3.0348 (17) Å. The N—H···Cl distances are 3.1281 (13) and 3.4368 (15) Å, and the O—H···Cl distances range from 3.1173 (14) to 3.1576 (13) Å. The O—H···O hydrogen bond involving the carbonyl group is 2.7404 (15) Å.

Experimental

As part of an experimental polymorph screen on guanine, (I) was obtained from a solution of guanine in dilute hydrochloric acid which was allowed to evaporate at room temperature (10 ml solution, in 75 × 25 mm vessels), forming block-shaped crystals. If the same guanine solution in dilute hydrochloric acid was allowed to evaporate at a slower rate by virtue of a smaller surface area, small block-like crystals of guaninium chloride monohydrate were obtained.

Crystal data

$C_5H_6N_5O^+\cdot Cl^-\cdot 2H_2O$
M_r = 223.63
Monoclinic, $P2_1/c$
a = 4.8587 (11) Å
b = 13.228 (3) Å
c = 14.612 (3) Å
β = 93.862 (4)°
V = 937.0 (4) Å^3
Z = 4
D_x = 1.585 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2645 reflections
θ = 2.1–28.2°
μ = 0.40 mm^{-1}
T = 150 (2) K
Block, colourless
0.42 × 0.12 × 0.08 mm

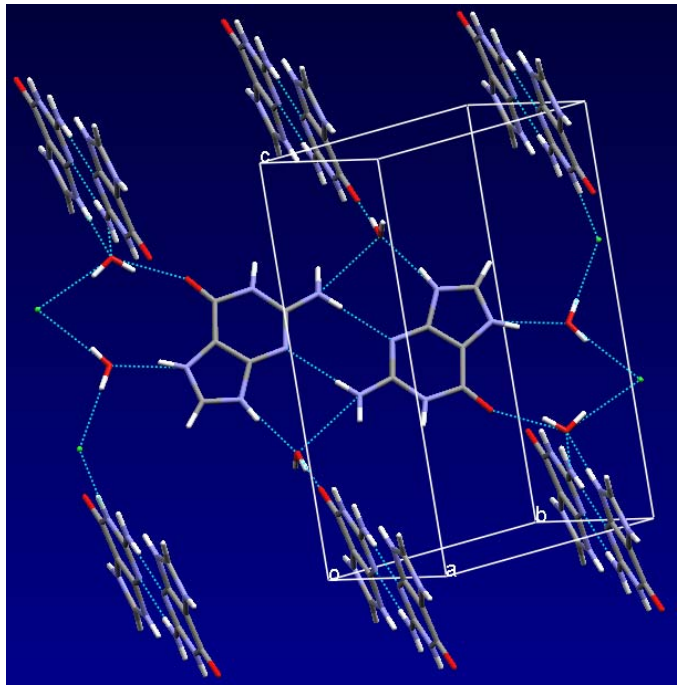


Figure 3
The crystal packing of (I), showing the complex three-dimensional hydrogen-bonding network (dashed lines).

Data collection

Bruker SMART APEX diffractometer
Narrow-frame ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.850, T_{max} = 0.969
7902 measured reflections
2230 independent reflections
1891 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.2°
$h = -6 \rightarrow 6$
$k = -17 \rightarrow 17$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.082
S = 1.04
2230 reflections
167 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.2271P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1^{i}	0.883 (18)	2.261 (19)	3.1281 (13)	167.2 (16)
N3—H3···O1W^{ii}	0.87 (2)	1.83 (2)	2.6867 (16)	167.8 (19)
N4—H4···O2W	0.908 (19)	1.76 (2)	2.6463 (17)	164.2 (18)
N5—H6···O1W^{iii}	0.861 (18)	2.518 (17)	3.0348 (17)	119.5 (13)
N5—H6···Cl1^{i}	0.861 (18)	2.682 (18)	3.4368 (15)	147.2 (14)
N5—H7···N2^{iv}	0.886 (19)	2.131 (19)	3.0162 (17)	176.9 (17)
O2W—H3W···Cl1	0.81 (2)	2.36 (2)	3.1576 (13)	167.9 (19)
O2W—H4W···Cl1^{v}	0.85 (2)	2.27 (2)	3.1173 (14)	169.2 (19)
O1W—H1W···Cl1	0.85 (2)	2.31 (2)	3.1336 (13)	166.0 (17)
O1W—H2W···O4	0.85 (2)	1.93 (2)	2.7404 (15)	160 (2)

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

H atoms were refined independently using an isotropic model.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97*.

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supporting information

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Cell parameters from 2645 reflections
$\theta = 2.1$–28.2°
$\mu = 0.40$ mm^{-1}
$T = 150$ K
Block, colourless
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Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω rotation scans with narrow frames
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
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7902 measured reflections
2230 independent reflections
1891 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 28.2°$, $\theta_{min} = 2.1°$
$h = -6\rightarrow6$
$k = -17\rightarrow17$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.082$
$S = 1.04$
2230 reflections
167 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.2271P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	1.11784 (8)	0.97938 (3)	0.33430 (2)	0.02731 (12)
O4	0.3146 (2)	0.70459 (7)	0.30721 (7)	0.0229 (2)
O1W	0.6924 (2)	0.83072 (9)	0.23556 (7)	0.0255 (2)
O2W	0.7162 (2)	0.90818 (9)	0.48276 (9)	0.0284 (3)
N1	−0.0342 (2)	0.60277 (9)	0.34807 (8)	0.0180 (2)
N2	−0.2267 (2)	0.59985 (8)	0.49343 (7)	0.0180 (2)
N3	−0.0027 (2)	0.72248 (9)	0.59658 (8)	0.0196 (3)
N4	0.2943 (2)	0.78712 (9)	0.50781 (8)	0.0192 (2)
N5	−0.3795 (3)	0.49067 (9)	0.37818 (9)	0.0214 (3)
C1	−0.2146 (3)	0.56538 (10)	0.40844 (9)	0.0170 (3)
C2	−0.0421 (3)	0.67400 (10)	0.51360 (9)	0.0171 (3)
C3	0.1454 (3)	0.71508 (10)	0.45727 (9)	0.0172 (3)
C4	0.1597 (3)	0.67858 (10)	0.36631 (9)	0.0177 (3)
C5	0.2011 (3)	0.78988 (11)	0.59023 (9)	0.0209 (3)
H1	−0.036 (4)	0.5739 (13)	0.2936 (13)	0.032 (5)*
H3	−0.095 (4)	0.7137 (14)	0.6449 (15)	0.041 (5)*
H4	0.429 (4)	0.8279 (14)	0.4882 (13)	0.037 (5)*
H5	0.268 (3)	0.8347 (13)	0.6416 (12)	0.025 (4)*
H6	−0.373 (3)	0.4697 (13)	0.3226 (13)	0.024 (4)*
H7	−0.500 (4)	0.4649 (13)	0.4145 (12)	0.030 (5)*
H3W	0.798 (4)	0.9297 (15)	0.4400 (14)	0.040 (6)*
H4W	0.747 (4)	0.9454 (16)	0.5301 (16)	0.044 (6)*
H1W	0.795 (4)	0.8675 (16)	0.2709 (13)	0.038 (5)*
H2W	0.585 (5)	0.7996 (17)	0.2697 (16)	0.053 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0360 (2)	0.0291 (2)	0.01704 (18)	−0.00440 (15)	0.00350 (14)	0.00212 (13)
O4	0.0246 (5)	0.0255 (5)	0.0198 (5)	−0.0048 (4)	0.0099 (4)	−0.0002 (4)
O1W	0.0278 (6)	0.0296 (6)	0.0200 (5)	−0.0062 (5)	0.0088 (4)	−0.0019 (4)
O2W	0.0274 (6)	0.0339 (6)	0.0244 (6)	−0.0098 (5)	0.0061 (4)	−0.0052 (5)
N1	0.0195 (6)	0.0197 (6)	0.0154 (6)	−0.0012 (4)	0.0049 (4)	−0.0013 (4)
N2	0.0189 (5)	0.0184 (6)	0.0173 (6)	0.0006 (4)	0.0056 (4)	0.0006 (4)
N3	0.0228 (6)	0.0210 (6)	0.0156 (6)	0.0011 (5)	0.0056 (5)	−0.0007 (4)
N4	0.0190 (6)	0.0197 (6)	0.0193 (6)	−0.0010 (5)	0.0034 (4)	−0.0012 (4)
N5	0.0225 (6)	0.0226 (6)	0.0198 (6)	−0.0049 (5)	0.0069 (5)	−0.0022 (5)
C1	0.0165 (6)	0.0165 (6)	0.0183 (6)	0.0026 (5)	0.0039 (5)	0.0024 (5)
C2	0.0180 (6)	0.0172 (6)	0.0164 (6)	0.0035 (5)	0.0042 (5)	0.0009 (5)
C3	0.0166 (6)	0.0171 (6)	0.0182 (6)	0.0006 (5)	0.0032 (5)	0.0007 (5)

C4	0.0181 (6)	0.0176 (6)	0.0177 (6)	0.0019 (5)	0.0042 (5)	0.0019 (5)
C5	0.0226 (7)	0.0209 (7)	0.0194 (7)	0.0025 (5)	0.0020 (5)	−0.0017 (5)

Geometric parameters (Å, º)

O4—C4	1.2321 (16)	N3—C2	1.3736 (18)
O1W—H1W	0.85 (2)	N3—H3	0.87 (2)
O1W—H2W	0.85 (2)	N4—C5	1.3154 (18)
O2W—H3W	0.81 (2)	N4—C3	1.3802 (18)
O2W—H4W	0.85 (2)	N4—H4	0.908 (19)
N1—C1	1.3769 (17)	N5—C1	1.3291 (18)
N1—C4	1.3892 (18)	N5—H6	0.861 (18)
N1—H1	0.883 (18)	N5—H7	0.886 (19)
N2—C1	1.3278 (18)	C2—C3	1.3797 (18)
N2—C2	1.3481 (17)	C3—C4	1.4202 (19)
N3—C5	1.3405 (19)	C5—H5	0.995 (17)
H1W—O1W—H2W	106.1 (19)	N2—C1—N5	120.19 (12)
H3W—O2W—H4W	110.7 (19)	N2—C1—N1	123.09 (12)
C1—N1—C4	126.10 (12)	N5—C1—N1	116.72 (12)
C1—N1—H1	117.0 (12)	N2—C2—N3	125.72 (12)
C4—N1—H1	116.8 (12)	N2—C2—C3	127.75 (12)
C1—N2—C2	112.57 (11)	N3—C2—C3	106.52 (12)
C5—N3—C2	108.01 (12)	C2—C3—N4	107.22 (12)
C5—N3—H3	124.6 (13)	C2—C3—C4	120.02 (12)
C2—N3—H3	127.4 (13)	N4—C3—C4	132.73 (12)
C5—N4—C3	107.99 (12)	O4—C4—N1	120.35 (12)
C5—N4—H4	124.9 (12)	O4—C4—C3	129.18 (13)
C3—N4—H4	127.1 (12)	N1—C4—C3	110.47 (11)
C1—N5—H6	119.5 (11)	N4—C5—N3	110.26 (12)
C1—N5—H7	119.8 (11)	N4—C5—H5	126.3 (10)
H6—N5—H7	120.6 (16)	N3—C5—H5	123.5 (9)
C2—N2—C1—N5	178.71 (12)	N3—C2—C3—C4	178.63 (11)
C2—N2—C1—N1	−0.62 (18)	C5—N4—C3—C2	−0.28 (15)
C4—N1—C1—N2	1.1 (2)	C5—N4—C3—C4	−178.36 (14)
C4—N1—C1—N5	−178.29 (12)	C1—N1—C4—O4	178.54 (12)
C1—N2—C2—N3	−178.52 (12)	C1—N1—C4—C3	−0.98 (18)
C1—N2—C2—C3	0.35 (19)	C2—C3—C4—O4	−178.82 (13)
C5—N3—C2—N2	178.93 (13)	N4—C3—C4—O4	−0.9 (3)
C5—N3—C2—C3	−0.14 (15)	C2—C3—C4—N1	0.65 (17)
N2—C2—C3—N4	−178.79 (13)	N4—C3—C4—N1	178.53 (14)
N3—C2—C3—N4	0.25 (14)	C3—N4—C5—N3	0.19 (16)
N2—C2—C3—C4	−0.4 (2)	C2—N3—C5—N4	−0.03 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1^{i}	0.883 (18)	2.261 (19)	3.1281 (13)	167.2 (16)
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Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) *x*−1, −*y*+3/2, *z*+1/2; (iii) −*x*, *y*−1/2, −*z*+1/2; (iv) −*x*−1, −*y*+1, −*z*+1; (v) −*x*+2, −*y*+2, −*z*+1.