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Key indicators

Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.032
 wR factor = 0.082
Data-to-parameter ratio = 13.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Redetermination of guaninium chloride
dihydrateThe low-temperature redetermination of guaninium chloride dihydrate, $\text{C}_5\text{H}_6\text{N}_5\text{O}^+\cdot\text{Cl}^- \cdot 2\text{H}_2\text{O}$, obtained as part of an experimental polymorph screen on guanine, is reported here.

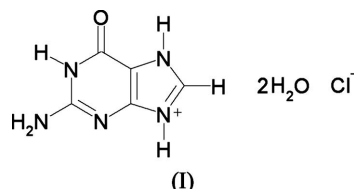
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Comment

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.



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 Å³, consistent with the determination at low temperature. In general, the

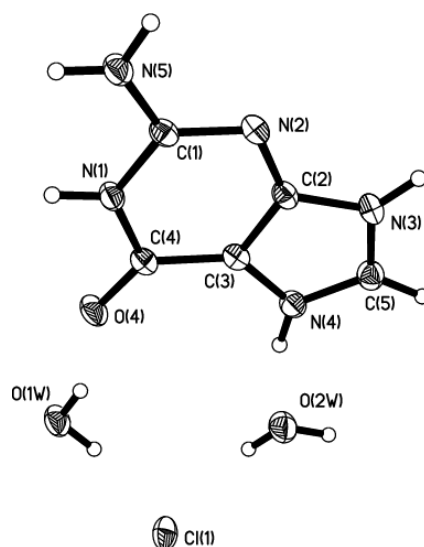
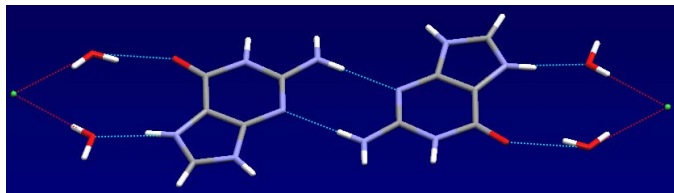


Figure 1
View of (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.


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.

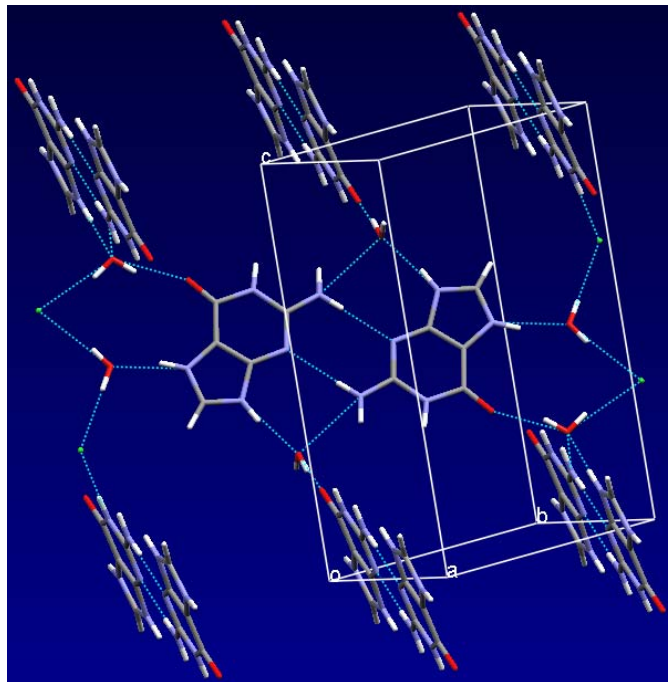
The packing consists of centrosymmetric dimers, the two components of which are linked by pairs of N–H···N hydrogen bonds. These dimers are linked to four water molecules and two Cl atoms to form a planar unit (Fig. 2). These planar units are linked to one another through O–H···Cl hydrogen bonds within 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 NH₂ 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···Cl1ⁱ = 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 ₅ H ₆ N ₅ O ⁺ ·Cl ⁻ ·2H ₂ O	<i>D</i> _x = 1.585 Mg m ⁻³
<i>M</i> _r = 223.63	Mo <i>K</i> α radiation
Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Cell parameters from 2645 reflections
<i>a</i> = 4.8587 (11) Å	<i>θ</i> = 2.1–28.2°
<i>b</i> = 13.228 (3) Å	<i>μ</i> = 0.40 mm ⁻¹
<i>c</i> = 14.612 (3) Å	<i>T</i> = 150 (2) K
<i>β</i> = 93.862 (4)°	Block, colourless
<i>V</i> = 937.0 (4) Å ³	0.42 × 0.12 × 0.08 mm
<i>Z</i> = 4	


Figure 3

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

Data collection

Bruker SMART APEX diffractometer	2230 independent reflections
Narrow-frame <i>ω</i> scans	1891 reflections with <i>I</i> > 2σ(<i>I</i>)
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	<i>R</i> _{int} = 0.024
<i>T</i> _{min} = 0.850, <i>T</i> _{max} = 0.969	<i>θ</i> _{max} = 28.2°
7902 measured reflections	<i>h</i> = -6 → 6
	<i>k</i> = -17 → 17
	<i>l</i> = -19 → 19

Refinement

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

Table 1

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
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* – ½, ½ – *z*; (ii) *x* – 1, ¾ – *y*, ½ + *z*; (iii) –*x*, *y* – ½, ½ – *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: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; 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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'Control and Prediction of the Organic Solid State'. For more information on this work, please visit <http://www.cposs.org.uk>.

References

- Broomhead, J. (1951). *Acta Cryst.* **4**, 92–100.
Bruker (2000). *SMART*, *SAINTE* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M. K., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.
Iball, J. & Wilson, H. R. (1963). *Nature (London)*, 4886, 1193–1195.
Iball, J. & Wilson, H. R. (1965). *Proc. R. Soc. London Ser. A*, **288**, 418–439.
Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.