Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination of piperazine dihydrochloride monohydrate

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Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.149
Data-to-parameter ratio = 28.5

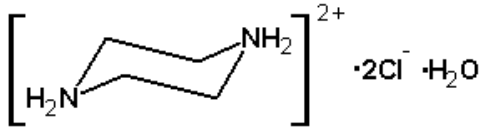
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 16 May 2001
Accepted 12 June 2001
Online 22 June 2001

In the title compound, $C_4H_{12}N_2^{2+}\cdot 2Cl^-\cdot H_2O$, the piperazinum dication has a center of symmetry, while the water molecule is on the twofold axis. The structure exhibits chains of piperazinum dications linked together by N—H···Cl hydrogen bonds with chloride ions. Between the chains, there are weak hydrogen bonds of the O—H···Cl and C—H···Cl types.

Comment

Hydrogen bonding plays an important role in the crystal engineering of organic solids (Desiraju, 1989; Melendez & Hamilton, 1998). There are many examples of one-, two- and three-dimensional motifs formed by hydrogen bonds. The crystal structure of the title compound, (I), has already been reported by Rérat (1960), where a photographic method was used for the intensity measurement and an isotropic refinement of non-H atoms was applied. The refinement of low resultion (0.95 Å) and low quality of data resulted in a poor *R* value of 0.28 and no H atoms were located. We present here a redetermination of this structure using data from a Siemens SMART CCD diffractometer.



(I)

The atomic numbering for (I) is presented in Fig. 1. The structure exhibits hydrogen bonds of the type N—H···Cl,

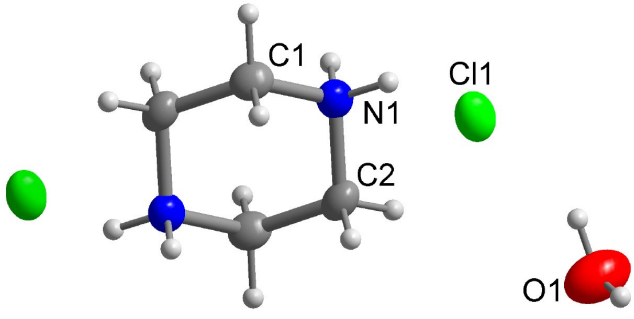


Figure 1
The molecular structure of (I). Displacement ellipsoids are shown at the 50% probability level.

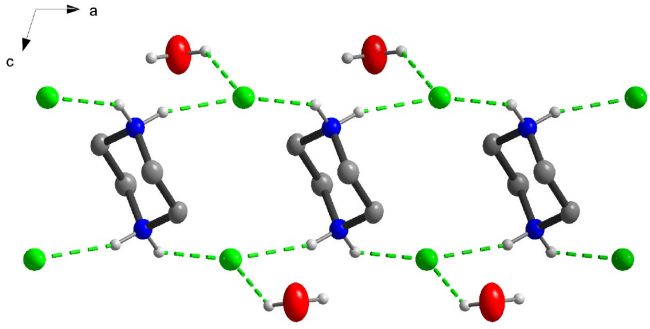


Figure 2
Representation of a hydrogen-bonded chain in the structure of (I). Each chain is involved in hydrogen bonding with water molecules.

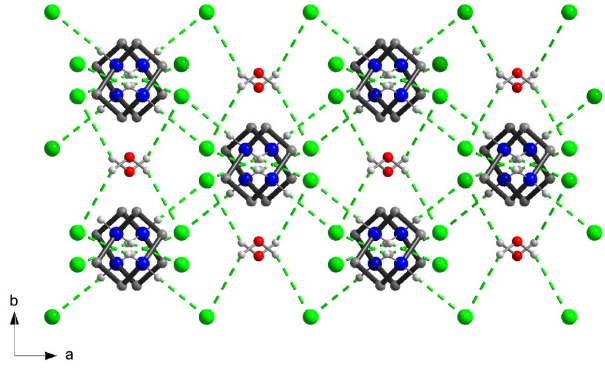


Figure 3
A view along the *c* axis showing how chains are crossed with respect to each other. Water molecules are in the channels and are weakly bonded to the chains.

forming chains of hydrogen-bonded ions along the [110] and [$\bar{1}$10] directions (Fig. 2). The water molecules are on twofold axes and are located in the channels formed by crossed chains and they are involved in weak hydrogen bonds of the O—H···Cl type with the nearest chains (Fig. 3). Thus, the water molecule shows high mobility which is reflected by high anisotropic displacement parameters of the O atom.

Experimental

The title compound was prepared by the reaction of an ethanolic solution of piperazine and HCl (37% in water) in the molar ratio 1:2.5 at room temperature. Crystals of (I) were obtained by slow evaporation of the solution.

Crystal data

$C_4H_{12}N_2^{2+}\cdot 2Cl^-\cdot H_2O$
M_r = 177.07
Monoclinic, *C*2/*c*
a = 10.2233 (3) Å
b = 6.3323 (3) Å
c = 13.5389 (6) Å
β = 107.587 (2)°
V = 835.50 (6) Å^3
Z = 4
D_x = 1.408 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 1781 reflections
θ = 1–32°
μ = 0.71 mm^{-1}
T = 297 (2) K
Thin plate, colorless
0.40 × 0.20 × 0.04 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (Sheldrick, 1996)
T_{min} = 0.764, T_{max} = 0.972
3545 measured reflections
1453 independent reflections
974 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 32.9°
h = −8 → 15
k = −8 → 9
l = −20 → 20

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.149
S = 1.02
1453 reflections
51 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0792P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.487 (3)	C1—C2^{i}	1.510 (3)
N1—C1	1.491 (3)		
C2—N1—C1	110.92 (16)	N1—C2—C1^{i}	110.23 (17)
N1—C1—C2^{i}	111.01 (17)		

Symmetry code: (i) $1-x, 1-y, 1-z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···Cl1	0.90	2.39	3.1412 (19)	141
N1—H1*A*···Cl1^{i}	0.90	2.29	3.1541 (18)	162
O1—H1···Cl1^{ii}	0.86 (2)	2.82 (4)	3.492 (3)	137 (5)
C2—H2*B*···Cl1^{iii}	0.97	2.80	3.475 (2)	128

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

The unique H atom of the water molecule was located from a difference Fourier map and was refined with a fixed isotropic displacement parameter and a restrained bond distance of 0.85 Å, whereas the other H atoms were constrained to idealized geometries using the appropriate riding model.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2000).

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