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3-Amino-1-propylammonium perchlorate monohydrate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.052
wR factor = 0.177
Data-to-parameter ratio = 14.4

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

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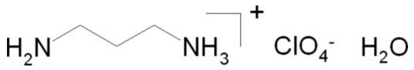
In the title compound, $C_3H_{10.67}N_2 \cdot H_{0.33}ClO_4 \cdot H_2O$ or $C_3H_{11}N_2^+ \cdot ClO_4^- \cdot H_2O$, the lone H atom is disordered over both N atoms and one perchlorate O atom. An extensive three-dimensional hydrogen-bonding network is formed by N—H···O, O—H···O and C—H···O hydrogen bonds.

Comment

Crystal structures of 1,3-propanediammonium diperchlorate (Pritchard *et al.*, 1992; Brooker & de Geest, 1999) and 3-amino-1-propylammonium perchlorate (Aurangzeb *et al.*, 1993) have been reported previously. Here, we present the crystal structure of the title compound, (I), which is a monohydrate of the latter compound.



(I)

In compound (I) (Fig. 1), the lone H atom is disordered over both N atoms and one perchlorate O atom. This was deduced from the residual electron-density map and the Cl1—O4 bond length [1.585 (3) Å], which is longer than the other Cl—O distances [1.510 (3)–1.526 (3) Å]. Some elongation of the Cl—O bond lengths in the perchlorate can be attributed to an extensive three-dimensional hydrogen-bonding network formed by the N—H···O, O—H···O and C—H···O intermolecular hydrogen bonds (Table 1), which stabilize the crystal packing (Fig. 2).

Experimental

Single crystals of the title compound, (I), were grown from the mother liquor of the condensation reaction between 2,6-diformyl-4-

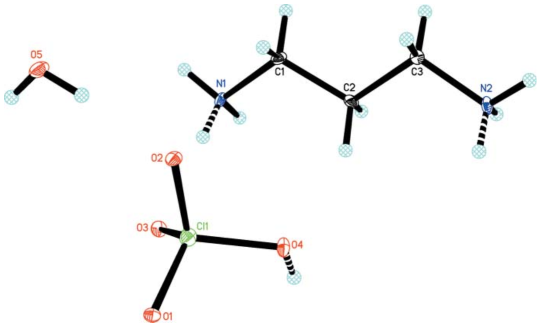


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme and with 30% probability displacement ellipsoids. Dashed lines show the chemical bonds with the disordered lone H atom.

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chlorophenol (0.185 g, 1.0 mmol) and 1,3-propanediamine (0.074 g, 1.0 mmol) in the presence of $Zn(ClO_4)_2 \cdot 6H_2O$ (0.745 g, 2.0 mmol), under reflux in ethanol (50 ml) for 2 h, in the process of preparing Schiff base macrocyclic complexes (Huang *et al.*, 2006).

Crystal data

$C_3H_{10.67}N_2^+ \cdot ClH_{0.33}O_4^- \cdot H_2O$
$M_r = 192.60$
Monoclinic, $P2_1/c$
$a = 6.9398\ (11)$ Å
$b = 16.634\ (3)$ Å
$c = 7.8543\ (12)$ Å
$\beta = 113.731\ (2)°$
$V = 830.0\ (2)$ Å^3

$Z = 4$
$D_x = 1.541$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.44$ mm^{-1}
$T = 120\ (2)$ K
Block, colourless
0.22 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.935$, $T_{\max} = 0.957$

4167 measured reflections
1466 independent reflections
1132 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.056$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.177$
$S = 1.13$
1466 reflections
102 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0882P)^2 + 0.8777P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.56$ e Å^{-3}
$\Delta\rho_{\min} = -0.85$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O1$^{\rm i}$	0.91	1.83	2.741 (4)	179
N1—H1D···O1$^{\rm ii}$	0.91	1.87	2.777 (4)	172
N1—H1E···O3	0.91	1.96	2.863 (4)	171
N2—H2D···O1$^{\rm iii}$	0.91	2.02	2.879 (4)	157
N2—H2D···O2$^{\rm iii}$	0.91	2.45	3.143 (5)	133
N2—H2C···O2$^{\rm iv}$	0.91	1.81	2.721 (4)	175
O5—H5A···O2	0.85	1.86	2.705 (4)	177
O5—H5B···O3$^{\rm v}$	0.83	2.01	2.834 (4)	175
C2—H2A···O4	0.99	2.44	3.366 (5)	155

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

All H atoms were placed in geometrically idealized positions, with C—H = 0.99, N—H = 0.91 and O—H = 0.83–0.85 Å, and refined as riding, with $U_{\rm iso}$(H) = $1.5_{\rm eq}$(N,O) or $U_{\rm iso}$(H) = $1.2_{\rm eq}$(C).

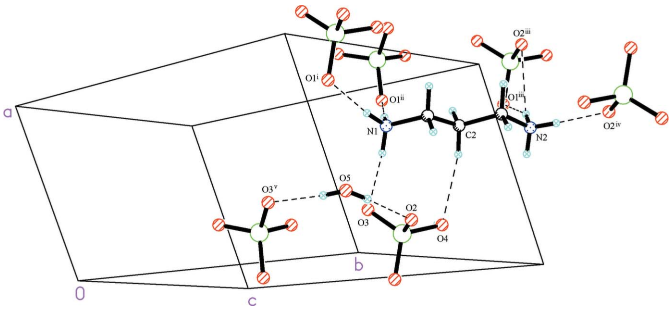

Figure 2
A portion of the crystal packing of (I), showing hydrogen bonds as dashed lines. [Symmetry codes: (i) $1 + x, \frac{3}{2} - y, \frac{1}{2} + z$; (ii) $1 + x, y, z$; (iii) $1 + x, y, 1 + z$; (iv) $1 - x, 2 - y, 2 - z$; (v) $x, \frac{3}{2} - y, -\frac{1}{2} + z$.]

In order to position the lone H atom correctly, we first used the approach of Aurangzeb *et al.* (1993), who treated this H atom as disordered between two atoms, N1 and N2. In the present case, the residual peaks near atoms N1 and N2 were 0.68 and 0.78 e Å^{-3}, respectively. However, after refinement, which gave $wR(F^2) = 0.180$, we found a relatively high residual peak of 0.64 e Å^{-3} at 0.85 Å from the atom O4. Therefore, we applied a model where the lone H atom is disordered between three atoms, N1, N2 and O4, with equal occupancies fixed at 0.333 each.

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

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