

4-Ammonio-1-methylpiperazin-1-ium oxalate dihydrate

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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.037
 wR factor = 0.100
Data-to-parameter ratio = 15.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the crystal structure of the title salt, $\text{C}_5\text{H}_{15}\text{N}_3^{2+} \cdot \text{C}_2\text{O}_4^{2-} \cdot 2\text{H}_2\text{O}$, all four O atoms of the oxalate anion are involved in hydrogen bonding with the piperazine dication and the water molecules of crystallization.

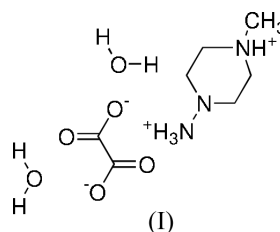
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Comment

To identify the product of the reaction of 1-amino-4-methylpiperazine with oxalic acid, the title salt, (I), was separated and its molecular structure determined.



The result showed that the product was the dihydrate of 4-ammonio-1-methylpiperazin-1-ium oxalate rather than mono-4-methylpiperazineammonium oxalate. The molecular structure of (I) is illustrated in Fig. 1. The bond distances and angles in both the cation and the anion are normal, within experimental error.

In the crystal structure, a dense hydrogen-bond network is built up (Fig. 2 and Table 1).

Experimental

Crystals of the title salt were obtained from a 1:1 aqueous solution of 1-amino-4-methylpiperazine and oxalic acid by slow concentration over a period of 3 d.

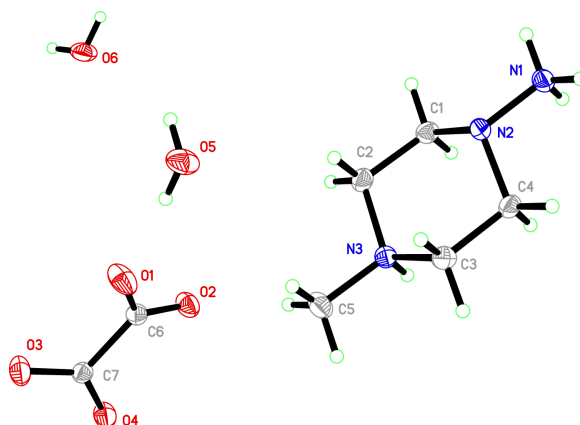


Figure 1

The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level.

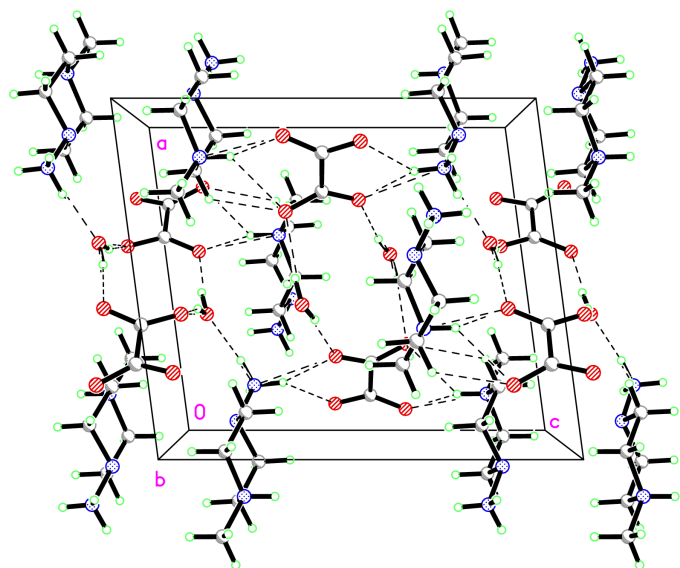


Figure 2
Packing diagram of (I), viewed down the *b* axis, showing the hydrogen-bond interactions (dashed lines).

Crystal data

$C_5H_{15}N_3^{2+} \cdot C_2O_4^{2-} \cdot 2H_2O$

$M_r = 241.25$

Monoclinic, $P2_1/n$

$a = 10.000$ (2) Å

$b = 9.9641$ (16) Å

$c = 11.661$ (4) Å

$\beta = 97.502$ (8)°

$V = 1152.0$ (5) Å³

$Z = 4$

$D_x = 1.391$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 977

reflections

$\theta = 2.7$ – 25.9°

$\mu = 0.12$ mm⁻¹

$T = 293$ (2) K

Block, colourless

$0.20 \times 0.16 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.975$, $T_{\max} = 0.985$

6250 measured reflections

2262 independent reflections

1839 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\text{max}} = 26.0^\circ$

$h = -6 \rightarrow 12$

$k = -11 \rightarrow 12$

$l = -14 \rightarrow 13$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.100$

$S = 1.04$

2262 reflections

147 parameters

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.3395P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} = 0.001$$

$$\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$$

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—H1A...O5 ⁱ	0.89	1.90	2.7598 (18)	163
N1—H1B...O6 ⁱⁱ	0.89	1.83	2.7118 (18)	172
N1—H1C...O1 ⁱⁱⁱ	0.89	1.90	2.7482 (18)	158
N3—H3...O4 ^{iv}	0.91	1.89	2.7318 (18)	154
O5—H5E...O2 ^v	0.85	2.00	2.8267 (18)	163
O5—H5D...O1	0.85	1.86	2.7026 (16)	172
O6—H6A...O3 ^{vi}	0.85	1.93	2.7617 (18)	164
O6—H6B...O4 ^{vii}	0.85	1.84	2.6787 (16)	170

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

The water H atoms were found in difference Fourier maps; however, during refinement, they were fixed at O—H distances of 0.85 Å and their $U_{\text{iso}}(\text{H})$ values were set equal to $1.2U_{\text{eq}}(\text{O})$. The H atoms of NH and CH groups were treated as riding, with N—H = 0.89 Å and C—H = 0.93–0.97 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N,C})$. For the H atoms attached to atom C5, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C5})$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 1997); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

- Bruker (1997). *SMART* (Version 5.051) and *SAINTE* (Version 5.A06). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
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