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N-Hydroxy-N-methylammonium chloride

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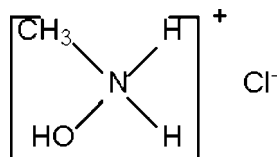
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{N}-\text{C}) = 0.003$ Å; R factor = 0.024; wR factor = 0.071; data-to-parameter ratio = 13.9.

In the crystal structure of the title compound, $\text{CH}_6\text{NO}^+\cdot\text{Cl}^-$, the cations and anions are linked by $\text{N}-\text{H}\cdots\text{Cl}$ and $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds into an undulating layer motif [Schläfli symbol: 4(8).6(8).8(2)]. All non-H atoms lie on a mirror plane.

Related literature

Only the cell dimensions of *N*-methylhydroxylammonium chloride have hitherto been reported; see: Toft & Jerslev (1967).



Experimental

Crystal data

 $\text{CH}_6\text{NO}^+\cdot\text{Cl}^-$
 $M_r = 83.52$

 Orthorhombic, *Pbcm*
 $a = 7.8084$ (3) Å

 $b = 8.7109$ (3) Å

 $c = 6.0232$ (1) Å

 $V = 409.69$ (2) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.73$ mm⁻¹
 $T = 100$ (2) K

 $0.25 \times 0.20 \times 0.15$ mm

Data collection

 Bruker SMART APEX
 diffractometer

 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)

 $T_{\min} = 0.839$, $T_{\max} = 0.899$

3330 measured reflections

558 independent reflections

 493 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.070$
 $S = 1.07$

558 reflections

40 parameters

6 restraints

All H-atom parameters refined

 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O}-\text{H1}\cdots\text{Cl}^1$	0.84 (1)	2.16 (1)	2.998 (1)	171 (2)
$\text{N}-\text{H2}\cdots\text{Cl}^1$	0.88 (1)	2.33 (1)	3.1241 (4)	149 (1)

 Symmetry code: (i) $x, -y + \frac{1}{2}, -z + 1$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); *OLEX* (Dolomanov *et al.*, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2008).

I thank the University of Malaya for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2270).

References

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supplementary materials

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***N*-Hydroxy-*N*-methylammonium chloride**

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Comment

We are interested in the crystal structures of small organic molecules, molecules whose asymmetric unit consist of four or five non-hydrogen atoms only. *N*-Methylhydroxylammonium chloride (Scheme I) provides an example of such a system. However, the crystal structure is not known with only unit-cell dimensions reported (Toft & Jerslev, 1967).

The structure (Fig. 1) consists of cations and anions that are linked by N–H···Cl and O–H···Cl hydrogen bonds into an undulating layer motif [Schläfli symbol: 4(8).6(8).8(2)], Fig. 2 & Table 1. The non-hydrogen atoms lie on a mirror plane.

Experimental

The chemical as purchased from the Aldrich Chemical Company was crystalline.

Refinement

All hydrogen atoms were located in a difference Fourier map, and were refined with distance restraints (C–H 0.99±0.01, N–H 0.88±0.01 and O–H 0.84±0.01 Å). For the methyl group, an additional H···H = 1.59±0.01 Å was imposed. The temperature factors were freely refined.

Figures

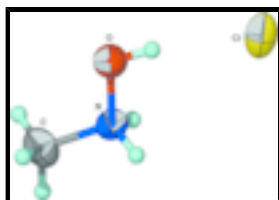


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of *N*-methylhydroxylammonium chloride at the 70% probability level.

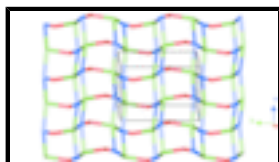


Fig. 2. OLEX (Dolomanov *et al.*, 2003) representation of the hydrogen-bonded layer structure.

***N*-Hydroxy-*N*-methylammonium chloride**

Crystal data

CH₆NO⁺·Cl⁻

M_r = 83.52

*F*₀₀₀ = 176

D_x = 1.354 Mg m⁻³

supplementary materials

Orthorhombic, *Pbcm*

Hall symbol: -P 2c 2b

$a = 7.8084$ (3) Å

$b = 8.7109$ (3) Å

$c = 6.0232$ (1) Å

$V = 409.69$ (2) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1402 reflections

$\theta = 2.6$ – 28.2°

$\mu = 0.73$ mm⁻¹

$T = 100$ (2) K

Prism, colorless

$0.25 \times 0.20 \times 0.15$ mm

Data collection

Bruker SMART APEX
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100$ (2) K

ω scans

Absorption correction: Multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.839$, $T_{\max} = 0.899$

3330 measured reflections

558 independent reflections

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

$R_{\text{int}} = 0.029$

$\theta_{\text{max}} = 28.4^\circ$

$\theta_{\text{min}} = 2.6^\circ$

$h = -10 \rightarrow 8$

$k = -11 \rightarrow 11$

$l = -8 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.024$

$wR(F^2) = 0.070$

$S = 1.07$

558 reflections

40 parameters

6 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

$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.0529P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\text{max}} = 0.28$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Extinction correction: SHELXL,
 $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.15 (1)

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl	0.35777 (6)	0.42790 (6)	0.2500	0.0433 (2)
O	0.1297 (2)	0.1511 (2)	0.2500	0.0389 (3)
N	0.2586 (2)	0.0376 (2)	0.2500	0.0300 (3)
C	0.1739 (3)	-0.1127 (3)	0.2500	0.0457 (5)
H1	0.184 (3)	0.2349 (17)	0.2500	0.048 (6)*
H2	0.3265 (17)	0.0482 (17)	0.366 (2)	0.041 (4)*
H3	0.2644 (18)	-0.190 (2)	0.2500	0.048 (6)*

H4 0.1075 (14) -0.121 (2) 0.1170 (8) 0.064 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0455 (3)	0.0591 (4)	0.0254 (3)	-0.0196 (2)	0.000	0.000
O	0.0315 (7)	0.0324 (7)	0.0529 (8)	0.0032 (5)	0.000	0.000
N	0.0282 (6)	0.0336 (7)	0.0282 (7)	0.0016 (5)	0.000	0.000
C	0.053 (1)	0.033 (1)	0.051 (1)	-0.004 (1)	0.000	0.000

Geometric parameters (Å, °)

O—N	1.411 (2)	N—H2	0.88 (1)
N—C	1.467 (3)	C—H3	0.98 (1)
O—H1	0.84 (1)	C—H4	0.96 (1)
N—O—H1	104.5 (16)	N—C—H3	107.0 (12)
O—N—C	107.70 (14)	N—C—H4	108.0 (11)
O—N—H2	110.8 (10)	H3—C—H4	110.0 (9)
C—N—H2	111.3 (10)		

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O—H1 \cdots Cl	0.84 (1)	2.16 (1)	2.998 (1)	171 (2)
N—H2 \cdots Cl ⁱ	0.88 (1)	2.33 (1)	3.1241 (4)	149 (1)

Symmetry codes: (i) $x, -y+1/2, -z+1$.

Fig. 1

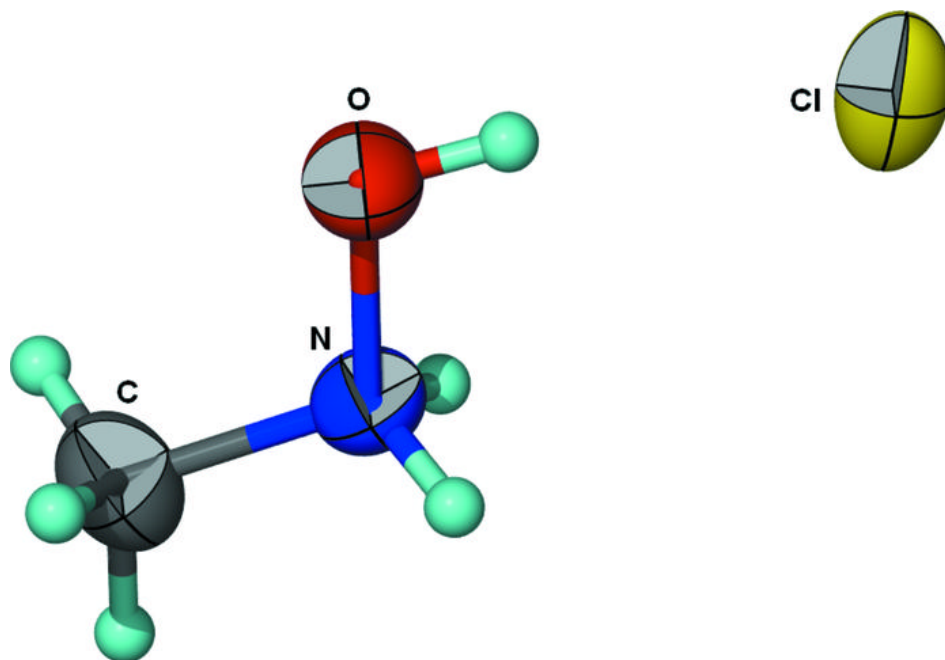


Fig. 2

