Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Trimethylammonium perchlorate at 97 K

Kazuma Gotoh, Rie Ishikawa and Hiroyuki Ishida*

Department of Chemistry, Faculty of Science, Okayama University, Okayama 700-8530, Japan

Correspondence e-mail: ishidah@cc.okayama-u.ac.jp

Received 1 March 2006
Accepted 5 March 2006

Key indicators

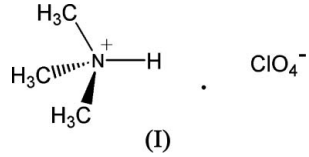
Single-crystal X-ray study
T = 93 K
Mean σ(I–O) = 0.002 Å
R factor = 0.024
wR factor = 0.062
Data-to-parameter ratio = 13.7

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

The crystal structure of the title compound, $C_3H_{10}N^+\cdot ClO_4^-$, redetermined at 97 K, is isostructural with $C_3H_{10}N^+\cdot BF_4^-$ [Gotoh, Ishikawa & Ishida (2005). *Acta Cryst.* E**61**, o4016–o4017]. Both the cation and the anion lie on a mirror plane and are connected by N—H···O and C—H···O hydrogen bonds.

Comment

Trimethylammonium perchlorate is known to have three solid phases above room temperature (Stammler *et al.*, 1966). These solid phases have been characterized in a wide temperature range of 90–530 K by ^{1}H NMR (Jurga, 1984; Ishida & Furukawa, 1996), ^{35}Cl NMR (Jurga *et al.*, 1986), Raman and IR (Mylrajan & Srinivasan, 1988), powder and single-crystal X-ray diffraction (Ishida *et al.*, 1994), as well as by electrical conductivity (Ishida & Furukawa, 1996). The high-temperature cubic phase is stable above 480 K and is an ionic plastic phase (Ikeda, 2004), in which both cation and anion perform isotropic rotation as well as self-diffusion. In the intermediate tetragonal phase, stable between 396 and 480 K, and the low-temperature phase, axial rotational motions of both ions are observed. Single-crystal X-ray diffraction data measured at 300 K showed that the displacement parameters of the O atoms of the anion were extremely large, probably due to large thermal vibration and libration or positional disorder. The space group was determined to be $P2_1$ based on Wilson's statistical analysis (Ishida *et al.*, 1994). On the other hand, Mylrajan & Srinivasan (1988) reported that the space group was $P2_1/m$, but detailed crystallographic data were not given. In the present study, we have redetermined the structure of the title compound, (I) (Fig. 1), at 93 K to clarify the state of the anion as well as the nature of the interactions between the ions at this reduced temperature.



(I)

The unit-cell parameters at 93 K are essentially the same as those at 300 K [a = 5.749 (1), b = 8.670 (2), c = 7.5585 (9) Å and β = 102.66 (1)°; Ishida *et al.*, 1994], but the space group is definitely determined to be $P2_1/m$, as pointed out by Mylrajan & Srinivasan (1988). Atoms O1, O2 and Cl1 of the anion and atoms N1 and C2 of the cation lie on a mirror plane, in the same manner as observed in $C_3H_{10}NH^+\cdot BF_4^-$ (Gotoh *et al.*, 2005). The ClO_4^- anion was refined as ordered at this

temperature, but the displacement parameters of O atoms are still larger than those of other non-H atoms. In the crystal structure, the cation and the anion are connected by a weak bifurcated N—H···O hydrogen bond (Fig. 1 and Table 1). There are also weak C—H···O interactions (Table 1), resulting in a molecular tape running parallel to the *b* axis (Fig. 2).

Experimental

Compound (I) was prepared by neutralizing trimethylamine (28% in water, 10 ml) with perchloric acid (20% in water, *ca* 20 ml). Single crystals were obtained by slow evaporation of a methanol solution of (I).

Crystal data

$C_3H_{10}N^+\cdot ClO_4^-$
M_r = 159.57
Monoclinic, $P2_1/m$
a = 5.504 (4) Å
b = 8.605 (6) Å
c = 7.580 (8) Å
β = 103.67 (4)°
V = 348.8 (5) Å^3
Z = 2
D_x = 1.519 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3847 reflections
θ = 3.6–29.0°
μ = 0.50 mm^{-1}
T = 93 K
Prism, colorless
0.45 × 0.35 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID II diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.615, T_{max} = 0.905
3864 measured reflections
985 independent reflections
941 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 29.0°
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 11$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.062
S = 1.13
985 reflections
72 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 0.1361P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.89 (2)	2.40 (2)	3.059 (4)	131.1 (19)
N1—H1···O2	0.89 (2)	2.07 (2)	2.940 (3)	166 (2)
C1—H4···O3^{i}	0.934 (16)	2.610 (17)	3.451 (4)	150.2 (13)
C2—H5···O3^{i}	0.995 (17)	2.477 (17)	3.406 (4)	155.2 (13)

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

H atoms were located in a Fourier map and refined isotropically, giving N—H = 0.89 (2) Å and C—H = 0.919 (17)–1.02 (2) Å.

Data collection: *PROCESS-AUTO* (Rigaku/MSC and Rigaku Corporation, 2004); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku Corporation, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

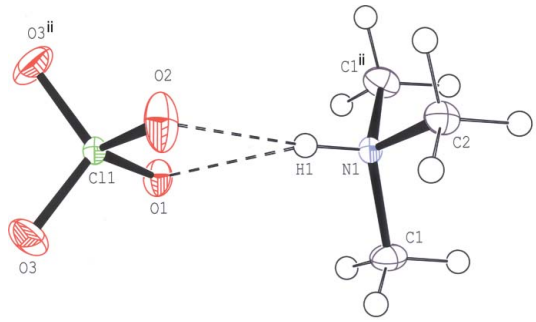


Figure 1
View of (I), showing the atom numbering. Displacement ellipsoids are drawn at the 50% probability level. N—H···O hydrogen bonds are indicated by dashed lines [symmetry code: (ii) $x, -y+\frac{1}{2}, z$].

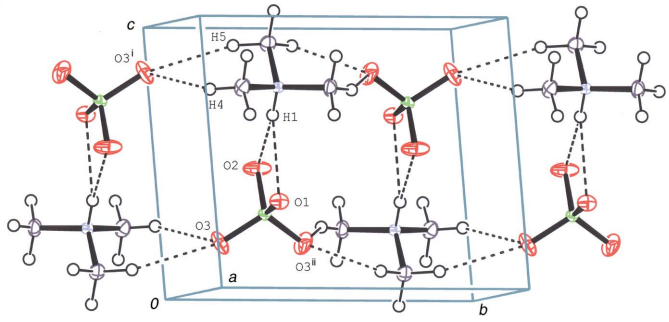


Figure 2
Packing diagram of (I), showing the molecular tape formed by N—H···O and C—H···O hydrogen bonds (dashed lines) [symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x, -y+\frac{1}{2}, z$].

This work was partly supported by a Grant-in-Aid for Scientific Research (C) (No. 16550014) from the Ministry of Education, Science, Sports and Culture of Japan.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Gotoh, K., Ishikawa, R & Ishida, H. (2005). *Acta Cryst.* E**61**, o4016–o4017.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Ikeda, R. (2004). *Recent Res. Dev. Chem. Phys.* **5**, 257–301.
Ishida, H. & Furukawa, Y. (1996). *Z. Naturforsch. Teil A*, **51**, 83–86.
Ishida, H., Kubozono, Y., Kashino, S. & Ikeda, R. (1994). *Z. Naturforsch. Teil A*, **55**, 723–726.
Jurga, S. (1984). *Phys. Stat. Sol. A*, **81**, 77–85.
Jurga, S., Harbison, G. S., Bluemich, B., Spiess, H. W., Fujara, F. & Olinger, A. (1986). *Ber. Bunsen-Geselshaft*, **90**, 1153–1159.
Mylrajan, M. & Srinivasan, T. K. K. (1988). *Spectrochim. Acta Sect. A*, **44**, 485–495.
Rigaku/MSC and Rigaku Corporation (2004). *RAPID-AUTO* and *CrystalStructure* (Version 3.7.0). Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Stammler, M., Bruenner, R. S., Schmit, W. G. & Orcutt, D. (1966). *Advan. X-Ray Anal.* **9**, 170–189.