Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(*N*,*N*-Dimethylamino)pyridinium perchlorate

Rosli Mohd Mustaqim,[a] Shamsher Ali,[a] Ibrahim Abdul Razak,[a] Hoong-Kun Fun,[a]* Shyamaprosad Goswami[b] and A. Adak[b]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Bengal Engineering and Science University, Shibpur, Howrah 711103, West Bengal, India

Correspondence e-mail: hkfun@usm.my

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.174
Data-to-parameter ratio = 22.3

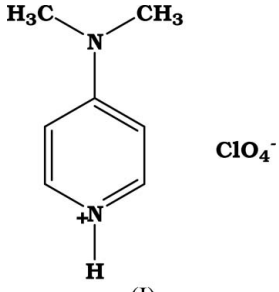
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 October 2005
Accepted 11 October 2005
Online 19 October 2005

The asymmetric unit of the title compound, $C_7H_{11}N_2^+\cdot ClO_4^-$, contains a 4-(*N*,*N*-dimethylamino)pyridinium cation and a perchlorate anion linked *via* an N—H···O hydrogen bond. Glide-related molecules are linked by C—H···O hydrogen bonds to form a chain along the *c* axis. The chains are arranged to form a layered structure parallel to the *bc* plane.

Comment

N,*N*-Dimethylaminopyridine is a basic nucleophilic catalyst in organic reactions such as acylation, whereas perchloric acid is an oxidizing agent, despite being a strong acid. We have synthesized the title compound, (I), to study its use as an oxidizing agent in more neutral conditions.



(I)

The bond lengths in (I) show normal values (Allen *et al.*, 1987) and are comparable with those reported for 2-amino-5-methyl-pyridine hydrochloride (Sherfinski & Marsh, 1975) and 4-aminopyridine hydrochloride dihydrate (Chao *et al.*, 1977). The C4—N1—C3 bond angle is wider than that in pyridine (116.94°; Sorensen *et al.*, 1974) and also that in 4-dimethylaminopyridine hydrochloride dihydrate (119.7 (2)°) (Chao *et al.*, 1977), which indicates that the pyridine ring N atom is protonated. This is consistent with theoretical studies which show that protonation on the pyridine ring N atom is

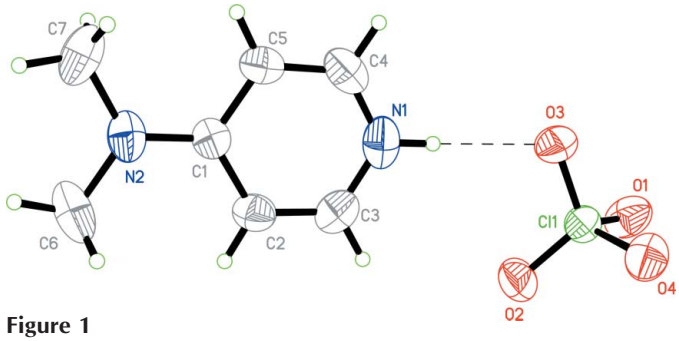


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The N—H···O hydrogen bond is shown by a dashed line.

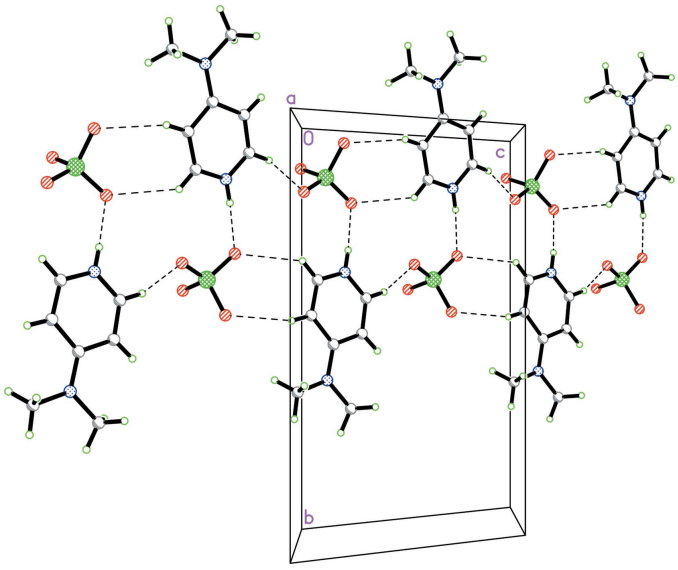


Figure 2
Part of the hydrogen-bonded (dashed lines) chain in (I), viewed down the *a* axis.

much easier than on the N atom of the amino group attached to pyridine (Konishi *et al.*, 1970). The *N*,*N*-dimethylaminopyridinium cation is essentially planar, with a maximum deviation of 0.031 (2) Å for atom C5.

In the asymmetric unit, the dimethylaminopyridinium and perchlorate ions are linked *via* N1—H1···O3 hydrogen bonds to form a pair. The glide-related pairs are linked by C—H···O hydrogen bonds (Table 2) to form a chain along the *c* axis (Fig. 2). The chains are arranged in such a way as to form a layered structure parallel to the *bc* plane. A short O1···O4($-x$, $-y$, $-z$) contact of 2.617 (3) Å is observed between inversion-related perchlorate ions in adjacent layers.

Experimental

N,*N*-Dimethylaminopyridine (30 mg) was dissolved in 70% perchloric acid (0.5 ml) with gentle warming and the reaction mixture was kept at room temperature. After several days, colourless needle-shaped crystals of compound (I) separated, which were collected and dried.

Crystal data

$C_7H_{11}N_2^+\cdot ClO_4^-$
M_r = 222.63
Monoclinic, $P2_1/c$
a = 8.1429 (1) Å
b = 14.8202 (2) Å
c = 10.4493 (1) Å
β = 128.771 (1)°
V = 983.16 (2) Å^3
Z = 4
D_x = 1.504 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5229 reflections
θ = 2.8–30.1°
μ = 0.38 mm^{-1}
T = 293 (2) K
Needle, colourless
0.70 × 0.33 × 0.16 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.747, T_{max} = 0.942
15960 measured reflections
2881 independent reflections
2368 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 30.1°
$h = -11 \rightarrow 11$
$k = -20 \rightarrow 20$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.174
S = 1.07
2881 reflections
129 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0974P)^2 + 0.3658P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—O2	1.4277 (16)	N1—C3	1.341 (3)
Cl1—O3	1.4405 (16)	N2—C1	1.339 (3)
Cl1—O1	1.4786 (17)	N2—C6	1.461 (3)
Cl1—O4	1.5412 (17)	N2—C7	1.462 (3)
N1—C4	1.324 (3)		
O2—Cl1—O3	112.92 (10)	C1—N2—C6	121.6 (2)
O2—Cl1—O1	112.25 (12)	C6—N2—C7	117.6 (2)
O3—Cl1—O1	110.47 (10)	N2—C1—C2	122.60 (19)
O2—Cl1—O4	108.63 (11)	C5—C1—C2	115.98 (18)
O3—Cl1—O4	105.90 (10)	N1—C3—C2	121.1 (2)
O1—Cl1—O4	106.24 (10)	N1—C4—C5	121.6 (2)
C4—N1—C3	120.9 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	2.00	2.851 (3)	168
C2—H2···O1^{ii}	0.93	2.55	3.428 (3)	157
C3—H3···O3^{ii}	0.93	2.53	3.271 (3)	137
C4—H4···O2^{iii}	0.93	2.31	3.207 (4)	163

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

H atoms were placed in calculated positions, with N—H = 0.86 Å and C—H = 0.93 or 0.96 Å. U_{iso}(H) values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms, and $1.2U_{eq}$ for the remaining H atoms.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

SA thanks Universiti Sains Malaysia for a Scientific Advancement Grant Allocation (SAGA) (grant No. 304/PFIZIK/635003/A118) and a USM short-term grant (No. 304/PFIZIK/635028).

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