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4-Hydroxy-2,2,6,6-tetramethylpiperidinium perchlorate

Ying Cui,* Yun-Hui Zhang and Peng-Wei Zhang

School of Pharmaceutical Science and Technology, Tianjin University, Tianjin 300072, People's Republic of China
Correspondence e-mail: flyingyting@yahoo.com.cn

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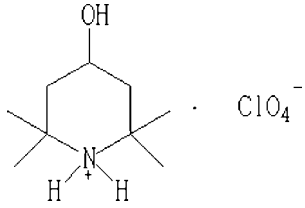
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.127; data-to-parameter ratio = 13.9.

In the title salt, $C_9H_{20}NO^+\cdot ClO_4^-$, intermolecular hydrogen bonds are observed, which determine the crystal packing.

Related literature

For general background, see Borzatta & Carrozza (1991).



Experimental

Crystal data

$C_9H_{20}NO^+\cdot ClO_4^-$
M_r = 257.71
Monoclinic, $P2_1/n$
a = 7.5712 (15) Å
b = 13.927 (3) Å
c = 12.007 (2) Å
β = 100.71 (3)°
V = 1244.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 113 (2) K
0.12 × 0.04 × 0.04 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.963, T_{max} = 0.988
7480 measured reflections
2183 independent reflections
1797 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.127
S = 1.10
2183 reflections
157 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4^{i}	0.92 (3)	2.05 (3)	2.914 (3)	157 (2)
N1—H1B···O1^{ii}	0.88 (3)	1.97 (3)	2.847 (3)	173 (2)
O1—H1···O2^{iii}	0.82	2.09	2.896 (2)	167
O1—H1···Cl1^{iii}	0.82	2.93	3.6985 (16)	158

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2373).

References

Borzatta, V. & Carrozza, P. (1991). Eur. Patent No. EP 0 462 069.
Rigaku/MSC (2005). *CrystalClear* and *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2008). E**64**, o654 [doi:10.1107/S1600536808002997]

4-Hydroxy-2,2,6,6-tetramethylpiperidinium perchlorate

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S1. Comment

2,2,6,6-Tetramethyl-4-hydroxy-piperidin-4-ol is a very important intermediate in the synthesis of hindered light stabilizers (Borzatta & Carrozza, 1991). We report here the crystal structure (2,2,6,6-tetramethyl-4-hydroxypiperidinium perchlorate) (Fig. 1).

Intermolecular N—H···O, O—H···O, O—H···Cl hydrogen bonds are observed which help to establish the crystal packing. The piperidine ring adopts chair conformation.

S2. Experimental

2,2,6,6-tetramethylpiperidin-4-ol (3.2 mmol,0.5 g) was dissolved in perchloric acid solution(2.5 mol/l, 3 ml). Block shaped colorless crystals grew with slow evaporation of solvent.

S3. Refinement

All H atoms were constrained; positioned geometrically (C—H = 0.99–1.00 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5$_{eq}$(methyl groups).

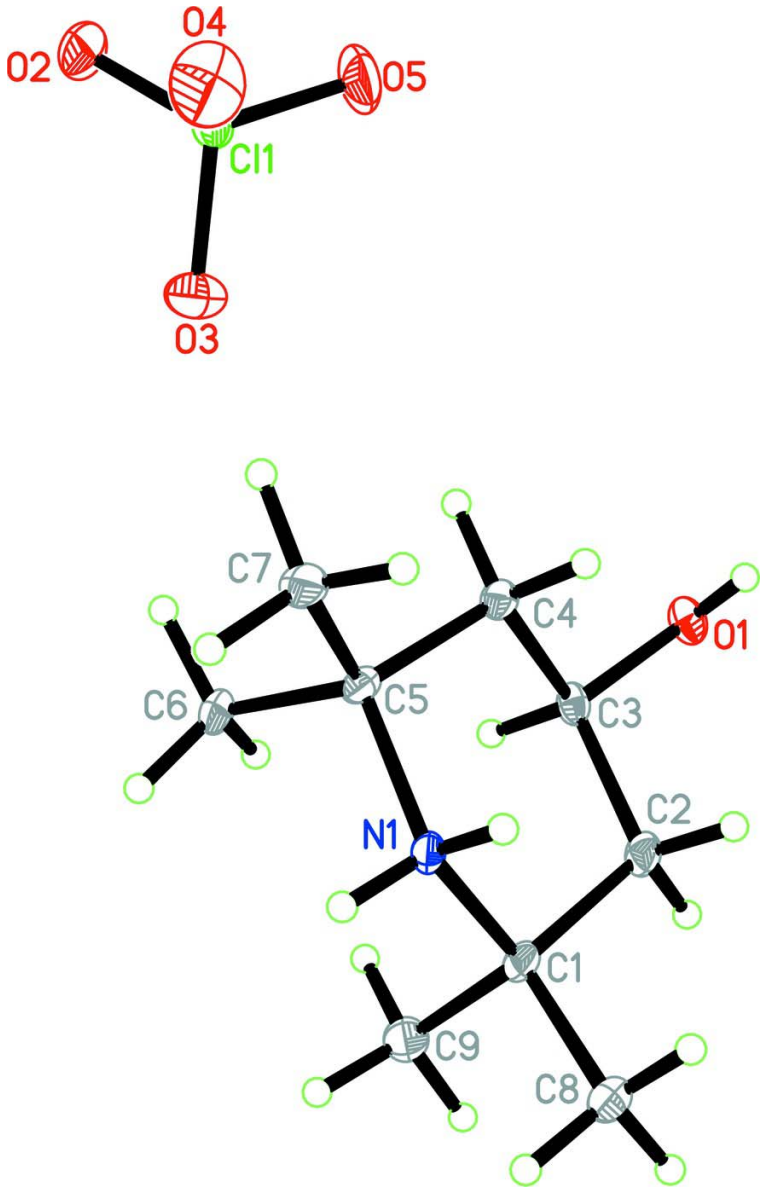

Figure 1

A view of the molecule (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

4-Hydroxy-2,2,6,6-tetramethylpiperidinium perchlorate

Crystal data

$C_9H_{20}NO^+\cdot ClO_4^-$
M_r = 257.71
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 7.5712 (15) Å
b = 13.927 (3) Å
c = 12.007 (2) Å
β = 100.71 (3)°
V = 1244.0 (4) Å^3
Z = 4
F(000) = 552
D_x = 1.376 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2824 reflections
θ = 2.3–28.1°
μ = 0.31 mm^{-1}

T = 113 K
Block, colorless

0.12 × 0.04 × 0.04 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.963, T_{max} = 0.988

7480 measured reflections
2183 independent reflections
1797 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −9→7
k = −11→16
l = −13→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.127
S = 1.10
2183 reflections
157 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0763P)^2 + 0.0376P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.8859 (2)	0.66280 (10)	0.28774 (12)	0.0224 (4)
H1	0.9165	0.6118	0.2631	0.034*
N1	0.6419 (2)	0.74364 (13)	−0.04347 (15)	0.0172 (4)
H1A	0.694 (4)	0.6951 (17)	−0.078 (2)	0.027 (6)*
H1B	0.570 (4)	0.7733 (17)	−0.099 (2)	0.029 (7)*
C1	0.7957 (3)	0.80834 (14)	0.01410 (18)	0.0200 (5)
C2	0.8982 (3)	0.75325 (14)	0.11605 (18)	0.0216 (5)
H2A	0.9652	0.7001	0.0882	0.026*
H2B	0.9874	0.7969	0.1609	0.026*
C3	0.7796 (3)	0.71199 (14)	0.19322 (16)	0.0184 (5)
H3	0.7150	0.7661	0.2230	0.022*
C4	0.6403 (3)	0.64456 (14)	0.12660 (17)	0.0187 (5)
H4A	0.5646	0.6181	0.1782	0.022*

H4B	0.7030	0.5902	0.0977	0.022*
C5	0.5198 (3)	0.69386 (14)	0.02718 (17)	0.0188 (5)
C6	0.3909 (3)	0.76627 (15)	0.06528 (19)	0.0240 (5)
H6A	0.3364	0.8060	0.0006	0.036*
H6B	0.4574	0.8073	0.1250	0.036*
H6C	0.2963	0.7318	0.0946	0.036*
C7	0.4117 (3)	0.62072 (15)	−0.05253 (19)	0.0248 (5)
H7A	0.4940	0.5759	−0.0797	0.037*
H7B	0.3397	0.6542	−0.1172	0.037*
H7C	0.3319	0.5851	−0.0118	0.037*
C8	0.9141 (3)	0.82429 (17)	−0.0744 (2)	0.0292 (6)
H8A	1.0164	0.8651	−0.0422	0.044*
H8B	0.8437	0.8557	−0.1412	0.044*
H8C	0.9582	0.7623	−0.0965	0.044*
C9	0.7249 (3)	0.90544 (15)	0.0458 (2)	0.0277 (5)
H9A	0.6607	0.8970	0.1088	0.042*
H9B	0.6427	0.9320	−0.0197	0.042*
H9C	0.8259	0.9496	0.0685	0.042*
Cl1	0.15810 (8)	0.46849 (3)	0.19458 (5)	0.0256 (2)
O2	−0.0091 (3)	0.47048 (12)	0.23547 (17)	0.0431 (5)
O3	0.2067 (3)	0.56462 (12)	0.17449 (16)	0.0423 (5)
O4	0.1359 (3)	0.41692 (15)	0.08989 (19)	0.0627 (7)
O5	0.2955 (3)	0.42562 (16)	0.2770 (2)	0.0603 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0226 (9)	0.0236 (8)	0.0180 (7)	0.0044 (6)	−0.0039 (7)	−0.0023 (6)
N1	0.0133 (10)	0.0196 (9)	0.0184 (9)	0.0023 (7)	0.0026 (8)	0.0006 (7)
C1	0.0124 (11)	0.0205 (11)	0.0266 (12)	−0.0036 (8)	0.0023 (10)	−0.0006 (8)
C2	0.0135 (11)	0.0233 (11)	0.0262 (11)	−0.0014 (8)	−0.0012 (10)	−0.0017 (8)
C3	0.0153 (12)	0.0205 (10)	0.0173 (10)	0.0024 (8)	−0.0021 (9)	−0.0012 (8)
C4	0.0159 (11)	0.0203 (10)	0.0191 (11)	−0.0012 (8)	0.0009 (9)	0.0001 (8)
C5	0.0146 (12)	0.0202 (10)	0.0218 (11)	−0.0040 (8)	0.0040 (10)	0.0013 (8)
C6	0.0141 (12)	0.0292 (12)	0.0300 (12)	0.0026 (9)	0.0075 (10)	0.0034 (9)
C7	0.0222 (13)	0.0248 (11)	0.0240 (12)	−0.0065 (9)	−0.0043 (10)	0.0025 (9)
C8	0.0194 (13)	0.0350 (13)	0.0344 (13)	0.0002 (10)	0.0081 (11)	0.0099 (10)
C9	0.0241 (13)	0.0215 (12)	0.0365 (13)	−0.0024 (9)	0.0030 (11)	−0.0001 (9)
Cl1	0.0215 (4)	0.0218 (3)	0.0361 (4)	0.0047 (2)	0.0119 (3)	0.0035 (2)
O2	0.0254 (11)	0.0522 (12)	0.0575 (13)	0.0051 (8)	0.0223 (10)	0.0141 (8)
O3	0.0465 (13)	0.0299 (10)	0.0501 (11)	−0.0063 (8)	0.0079 (10)	0.0074 (8)
O4	0.0644 (16)	0.0566 (13)	0.0694 (15)	0.0081 (11)	0.0189 (13)	−0.0359 (11)
O5	0.0340 (12)	0.0705 (15)	0.0760 (15)	0.0229 (10)	0.0091 (12)	0.0437 (12)

Geometric parameters (Å, º)

O1—C3	1.437 (2)	C5—C6	1.532 (3)
O1—H1	0.8199	C6—H6A	0.9800

N1—C1	1.532 (3)	C6—H6B	0.9800
N1—C5	1.532 (3)	C6—H6C	0.9800
N1—H1A	0.92 (3)	C7—H7A	0.9800
N1—H1B	0.88 (3)	C7—H7B	0.9800
C1—C9	1.528 (3)	C7—H7C	0.9800
C1—C2	1.529 (3)	C8—H8A	0.9800
C1—C8	1.529 (3)	C8—H8B	0.9800
C2—C3	1.518 (3)	C8—H8C	0.9800
C2—H2A	0.9900	C9—H9A	0.9800
C2—H2B	0.9900	C9—H9B	0.9800
C3—C4	1.523 (3)	C9—H9C	0.9800
C3—H3	1.0000	Cl1—O3	1.4210 (18)
C4—C5	1.524 (3)	Cl1—O5	1.427 (2)
C4—H4A	0.9900	Cl1—O4	1.430 (2)
C4—H4B	0.9900	Cl1—O2	1.4408 (18)
C5—C7	1.527 (3)		

C3—O1—H1	106.4	C4—C5—N1	107.61 (17)
C1—N1—C5	120.17 (16)	C7—C5—N1	105.26 (16)
C1—N1—H1A	106.5 (16)	C6—C5—N1	110.57 (16)
C5—N1—H1A	105.7 (15)	C5—C6—H6A	109.5
C1—N1—H1B	112.1 (16)	C5—C6—H6B	109.5
C5—N1—H1B	106.2 (16)	H6A—C6—H6B	109.5
H1A—N1—H1B	105 (2)	C5—C6—H6C	109.5
C9—C1—C2	113.22 (18)	H6A—C6—H6C	109.5
C9—C1—C8	108.82 (17)	H6B—C6—H6C	109.5
C2—C1—C8	110.68 (18)	C5—C7—H7A	109.5
C9—C1—N1	111.14 (18)	C5—C7—H7B	109.5
C2—C1—N1	107.24 (16)	H7A—C7—H7B	109.5
C8—C1—N1	105.46 (17)	C5—C7—H7C	109.5
C3—C2—C1	114.12 (18)	H7A—C7—H7C	109.5
C3—C2—H2A	108.7	H7B—C7—H7C	109.5
C1—C2—H2A	108.7	C1—C8—H8A	109.5
C3—C2—H2B	108.7	C1—C8—H8B	109.5
C1—C2—H2B	108.7	H8A—C8—H8B	109.5
H2A—C2—H2B	107.6	C1—C8—H8C	109.5
O1—C3—C2	110.75 (17)	H8A—C8—H8C	109.5
O1—C3—C4	110.62 (16)	H8B—C8—H8C	109.5
C2—C3—C4	110.06 (16)	C1—C9—H9A	109.5
O1—C3—H3	108.4	C1—C9—H9B	109.5
C2—C3—H3	108.4	H9A—C9—H9B	109.5
C4—C3—H3	108.4	C1—C9—H9C	109.5
C3—C4—C5	112.87 (16)	H9A—C9—H9C	109.5
C3—C4—H4A	109.0	H9B—C9—H9C	109.5
C5—C4—H4A	109.0	O3—Cl1—O5	109.47 (13)
C3—C4—H4B	109.0	O3—Cl1—O4	108.40 (13)
C5—C4—H4B	109.0	O5—Cl1—O4	110.53 (14)
H4A—C4—H4B	107.8	O3—Cl1—O2	108.16 (11)

C4—C5—C7	111.31 (16)	O5—Cl1—O2	110.23 (11)
C4—C5—C6	112.63 (16)	O4—Cl1—O2	110.00 (13)
C7—C5—C6	109.21 (18)		
C5—N1—C1—C9	−76.1 (2)	O1—C3—C4—C5	178.28 (15)
C5—N1—C1—C2	48.2 (2)	C2—C3—C4—C5	−59.0 (2)
C5—N1—C1—C8	166.19 (18)	C3—C4—C5—C7	167.26 (17)
C9—C1—C2—C3	72.5 (2)	C3—C4—C5—C6	−69.7 (2)
C8—C1—C2—C3	−165.05 (17)	C3—C4—C5—N1	52.4 (2)
N1—C1—C2—C3	−50.5 (2)	C1—N1—C5—C4	−49.6 (2)
C1—C2—C3—O1	−179.04 (15)	C1—N1—C5—C7	−168.37 (17)
C1—C2—C3—C4	58.3 (2)	C1—N1—C5—C6	73.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4^{i}	0.92 (3)	2.05 (3)	2.914 (3)	157 (2)
N1—H1*B*···O1^{ii}	0.88 (3)	1.97 (3)	2.847 (3)	173 (2)
O1—H1···O2^{iii}	0.82	2.09	2.896 (2)	167
O1—H1···Cl1^{iii}	0.82	2.93	3.6985 (16)	158

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*−1/2, −*y*+3/2, *z*−1/2; (iii) *x*+1, *y*, *z*.