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4-Amino-2,2,6,6-tetramethylpiperidin-1-ium chloride monohydrate

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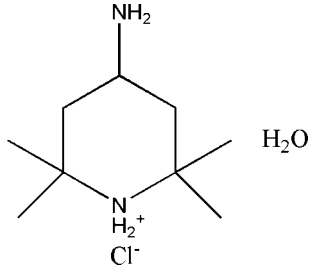
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.085; data-to-parameter ratio = 18.5.

In the title compound, $C_9H_{21}N_2^+\cdot Cl^-\cdot H_2O$, the piperidine ring adopts a chair conformation. In the crystal structure, the components are linked by N—H···N, O—H···Cl, N—H···Cl and N—H···O hydrogen bonds. There are two independent chloride ions, both on crystallographic twofold rotation axes.

Related literature

For related literature, see: Bojinov & Grabchev (2001).



Experimental

Crystal data

$C_9H_{21}N_2^+\cdot Cl^-\cdot H_2O$
M_r = 210.74
Orthorhombic, *Pbcn*
a = 14.1959 (9) Å
b = 12.7099 (7) Å
c = 12.5451 (6) Å
V = 2263.5 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 113 (2) K
0.32 × 0.26 × 0.20 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.898, T_{max} = 0.941
26456 measured reflections
2699 independent reflections
2672 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.085
S = 1.15
2699 reflections
146 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1D···Cl2	0.817 (19)	2.44 (2)	3.2509 (11)	174.7 (17)
O1—H1C···Cl1^{i}	0.854 (19)	2.244 (19)	3.0971 (11)	177.7 (17)
N2—H2C···Cl2	0.882 (18)	2.601 (18)	3.4577 (11)	164.2 (13)
N1—H1B···O1	0.894 (17)	2.098 (17)	2.9718 (14)	165.6 (14)
N1—H1A···N2^{i}	0.926 (18)	1.935 (18)	2.8602 (14)	176.1 (15)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2551).

References

Bojinov, V. B. & Grabchev, I. (2001). *Polym. Degrad. Stab.* **74**, 543–550.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
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supplementary materials

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4-Amino-2,2,6,6-tetramethylpiperidin-1-ium chloride monohydrate

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Comment

Hindered amine light stabilizers are one of the most intensively studied classes of stabilizers due to their high photo-stabilization efficiency (Bojinov & Grabchev, 2001). 2,2,6,6-Tetramethylpiperidine-4-amine is an important intermediate of hindered amine light stabilizers.

We report here the crystal structure of the title compound, (I), (Fig. 1), in which the piperidinyl ring adopts a chair conformation. In the crystal, the components are linked by intermolecular N—H···N, O—H···Cl, N—H···Cl and N—H···O hydrogen bonds (Table 1).

Experimental

The title compound was prepared by dissolving 4-ammonio-2,2,6,6-tetramethylpiperidine in aqueous hydrochloric acid solution. Colourless prisms of (I) were obtained by slow evaporation.

Refinement

The O– and N-bound H atoms were located in a difference map and their positions and U_{iso} values were freely refined. The C-bound atoms were positioned geometrically (C—H = 0.98–0.99 Å), and refined as riding with U_{iso}(H)=1.2U_{eq}(C) or 1.5$_{eq}$(methyl C).

Figures

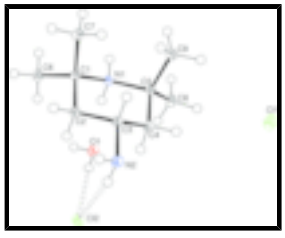

Fig. 1. A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radius. The hydrogen bonds are indicated by dashed lines.

4-Amino-2,2,6,6-tetramethylpiperidin-1-ium chloride monohydrate

Crystal data

$C_9H_{21}N_2^+\cdot Cl^-\cdot H_2O$	D_x = 1.237 Mg m^{-3}
M_r = 210.74	Melting point: >523 K
Orthorhombic, *Pbcn*	Mo *K*α radiation λ = 0.71070 Å

Hall symbol: -P 2n 2ab	Cell parameters from 6541 reflections
a = 14.1959 (9) Å	θ = 1.4–27.9º
b = 12.7099 (7) Å	μ = 0.31 mm^{-1}
c = 12.5451 (6) Å	T = 113 (2) K
V = 2263.5 (2) $Å^3$	Prism, colorless
Z = 8	0.32 × 0.26 × 0.20 mm
F_{000} = 928	

Data collection

Rigaku Saturn diffractometer	2672 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	R_{int} = 0.030
Monochromator: confocal	θ_{max} = 27.9º
T = 113(2) K	θ_{min} = 2.2º
ω scans	h = −18→17
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2005)	k = −16→16
T_{min} = 0.898, T_{max} = 0.941	l = −16→16
26456 measured reflections	Standard reflections: ?
2699 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2 + 1.133P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.085	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.15	$\Delta\rho_{max}$ = 0.34 e $Å^{-3}$
2699 reflections	$\Delta\rho_{min}$ = −0.17 e $Å^{-3}$
146 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0104 (8)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2sigma(F^2)$ is used only for calculat-

ing *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}
Cl1	0.0000	0.64751 (3)	0.2500	0.02249 (13)
Cl2	0.5000	0.56016 (3)	0.2500	0.01639 (12)
N1	0.25307 (7)	0.33787 (8)	0.40843 (8)	0.0111 (2)
H1A	0.2197 (11)	0.2761 (14)	0.4188 (13)	0.026 (4)*
H1B	0.2920 (11)	0.3283 (12)	0.3533 (13)	0.020 (4)*
N2	0.35317 (8)	0.65104 (8)	0.44971 (9)	0.0143 (2)
H2C	0.3942 (12)	0.6415 (12)	0.3978 (14)	0.020 (4)*
H2D	0.3836 (12)	0.6657 (13)	0.5061 (14)	0.025 (4)*
O1	0.37732 (7)	0.34651 (8)	0.21798 (8)	0.0200 (2)
H1C	0.4126 (13)	0.2927 (15)	0.2264 (14)	0.030*
H1D	0.4113 (13)	0.3976 (15)	0.2268 (14)	0.030*
C1	0.31784 (8)	0.35426 (9)	0.50387 (9)	0.0118 (2)
C2	0.36764 (8)	0.45990 (9)	0.48955 (9)	0.0125 (2)
H2A	0.4132	0.4538	0.4301	0.015*
H2B	0.4038	0.4758	0.5551	0.015*
C3	0.30101 (8)	0.55161 (9)	0.46649 (9)	0.0121 (2)
H3	0.2575	0.5606	0.5286	0.015*
C4	0.24252 (8)	0.52763 (9)	0.36733 (9)	0.0126 (2)
H4A	0.2852	0.5226	0.3051	0.015*
H4B	0.1988	0.5869	0.3544	0.015*
C5	0.18539 (8)	0.42568 (9)	0.37562 (9)	0.0124 (2)
C6	0.38975 (9)	0.26463 (9)	0.50102 (10)	0.0157 (3)
H6A	0.3573	0.1973	0.5111	0.024*
H6B	0.4359	0.2746	0.5582	0.024*
H6C	0.4221	0.2646	0.4320	0.024*
C7	0.26345 (9)	0.34794 (9)	0.60886 (9)	0.0154 (3)
H7A	0.2233	0.4102	0.6163	0.023*
H7B	0.3080	0.3450	0.6685	0.023*
H7C	0.2242	0.2845	0.6091	0.023*
C8	0.14726 (9)	0.39370 (10)	0.26623 (10)	0.0165 (3)
H8A	0.1999	0.3833	0.2168	0.025*
H8B	0.1059	0.4493	0.2390	0.025*
H8C	0.1115	0.3281	0.2728	0.025*
C9	0.10232 (9)	0.43610 (10)	0.45239 (10)	0.0166 (3)
H9A	0.0752	0.3664	0.4659	0.025*
H9B	0.0543	0.4819	0.4208	0.025*
H9C	0.1242	0.4666	0.5197	0.025*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0233 (2)	0.0148 (2)	0.0293 (3)	0.000	−0.00737 (18)	0.000

Cl2	0.0156 (2)	0.0194 (2)	0.0141 (2)	0.000	0.00074 (14)	0.000
N1	0.0117 (5)	0.0105 (5)	0.0110 (4)	0.0000 (4)	0.0002 (4)	−0.0006 (3)
N2	0.0153 (5)	0.0109 (5)	0.0166 (5)	−0.0014 (4)	−0.0005 (4)	0.0000 (4)
O1	0.0166 (5)	0.0188 (5)	0.0246 (5)	0.0000 (4)	−0.0012 (4)	−0.0020 (4)
C1	0.0135 (5)	0.0113 (5)	0.0106 (5)	0.0005 (4)	−0.0014 (4)	0.0003 (4)
C2	0.0129 (5)	0.0114 (5)	0.0131 (5)	−0.0007 (4)	−0.0005 (4)	0.0001 (4)
C3	0.0136 (5)	0.0104 (5)	0.0124 (5)	−0.0004 (4)	0.0007 (4)	−0.0001 (4)
C4	0.0141 (5)	0.0111 (5)	0.0128 (5)	0.0006 (4)	−0.0004 (4)	0.0014 (4)
C5	0.0116 (5)	0.0115 (5)	0.0140 (5)	0.0026 (4)	−0.0008 (4)	0.0007 (4)
C6	0.0165 (6)	0.0130 (6)	0.0176 (6)	0.0013 (4)	−0.0020 (4)	0.0009 (4)
C7	0.0188 (6)	0.0158 (6)	0.0115 (5)	−0.0008 (5)	0.0006 (5)	0.0015 (4)
C8	0.0171 (6)	0.0157 (6)	0.0167 (6)	0.0013 (5)	−0.0038 (5)	−0.0015 (5)
C9	0.0130 (5)	0.0167 (6)	0.0200 (6)	0.0009 (4)	0.0022 (5)	−0.0001 (5)

Geometric parameters (Å, °)

N1—C1	1.5240 (14)	C4—C5	1.5322 (16)
N1—C5	1.5290 (14)	C4—H4A	0.9900
N1—H1A	0.926 (18)	C4—H4B	0.9900
N1—H1B	0.894 (17)	C5—C9	1.5283 (16)
N2—C3	1.4798 (15)	C5—C8	1.5302 (16)
N2—H2C	0.882 (18)	C6—H6A	0.9800
N2—H2D	0.850 (18)	C6—H6B	0.9800
O1—H1C	0.854 (19)	C6—H6C	0.9800
O1—H1D	0.817 (19)	C7—H7A	0.9800
C1—C2	1.5281 (15)	C7—H7B	0.9800
C1—C7	1.5287 (16)	C7—H7C	0.9800
C1—C6	1.5301 (16)	C8—H8A	0.9800
C2—C3	1.5288 (16)	C8—H8B	0.9800
C2—H2A	0.9900	C8—H8C	0.9800
C2—H2B	0.9900	C9—H9A	0.9800
C3—C4	1.5264 (16)	C9—H9B	0.9800
C3—H3	1.0000	C9—H9C	0.9800
C1—N1—C5	119.39 (9)	C5—C4—H4B	108.8
C1—N1—H1A	108.3 (10)	H4A—C4—H4B	107.7
C5—N1—H1A	109.6 (10)	C9—C5—N1	112.24 (9)
C1—N1—H1B	104.7 (10)	C9—C5—C8	108.37 (10)
C5—N1—H1B	106.2 (10)	N1—C5—C8	105.65 (9)
H1A—N1—H1B	108.1 (14)	C9—C5—C4	112.21 (10)
C3—N2—H2C	108.6 (10)	N1—C5—C4	107.64 (9)
C3—N2—H2D	108.9 (12)	C8—C5—C4	110.55 (9)
H2C—N2—H2D	108.0 (15)	C1—C6—H6A	109.5
H1C—O1—H1D	105.9 (18)	C1—C6—H6B	109.5
N1—C1—C2	107.87 (9)	H6A—C6—H6B	109.5
N1—C1—C7	111.41 (9)	C1—C6—H6C	109.5
C2—C1—C7	112.41 (9)	H6A—C6—H6C	109.5
N1—C1—C6	106.40 (9)	H6B—C6—H6C	109.5
C2—C1—C6	110.04 (10)	C1—C7—H7A	109.5
C7—C1—C6	108.54 (9)	C1—C7—H7B	109.5

C1—C2—C3	113.95 (9)	H7A—C7—H7B	109.5
C1—C2—H2A	108.8	C1—C7—H7C	109.5
C3—C2—H2A	108.8	H7A—C7—H7C	109.5
C1—C2—H2B	108.8	H7B—C7—H7C	109.5
C3—C2—H2B	108.8	C5—C8—H8A	109.5
H2A—C2—H2B	107.7	C5—C8—H8B	109.5
N2—C3—C4	109.07 (9)	H8A—C8—H8B	109.5
N2—C3—C2	111.62 (9)	C5—C8—H8C	109.5
C4—C3—C2	109.78 (9)	H8A—C8—H8C	109.5
N2—C3—H3	108.8	H8B—C8—H8C	109.5
C4—C3—H3	108.8	C5—C9—H9A	109.5
C2—C3—H3	108.8	C5—C9—H9B	109.5
C3—C4—C5	113.67 (9)	H9A—C9—H9B	109.5
C3—C4—H4A	108.8	C5—C9—H9C	109.5
C5—C4—H4A	108.8	H9A—C9—H9C	109.5
C3—C4—H4B	108.8	H9B—C9—H9C	109.5
C5—N1—C1—C2	−50.01 (13)	N2—C3—C4—C5	179.86 (9)
C5—N1—C1—C7	73.82 (12)	C2—C3—C4—C5	57.27 (13)
C5—N1—C1—C6	−168.06 (9)	C1—N1—C5—C9	−73.64 (13)
N1—C1—C2—C3	51.43 (12)	C1—N1—C5—C8	168.44 (10)
C7—C1—C2—C3	−71.78 (13)	C1—N1—C5—C4	50.30 (13)
C6—C1—C2—C3	167.12 (9)	C3—C4—C5—C9	71.93 (12)
C1—C2—C3—N2	−178.05 (9)	C3—C4—C5—N1	−52.03 (12)
C1—C2—C3—C4	−56.98 (13)	C3—C4—C5—C8	−166.96 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1D···Cl2	0.817 (19)	2.44 (2)	3.2509 (11)	174.7 (17)
O1—H1C···Cl1^{i}	0.854 (19)	2.244 (19)	3.0971 (11)	177.7 (17)
N2—H2C···Cl2	0.882 (18)	2.601 (18)	3.4577 (11)	164.2 (13)
N1—H1B···O1	0.894 (17)	2.098 (17)	2.9718 (14)	165.6 (14)
N1—H1A···N2^{i}	0.926 (18)	1.935 (18)	2.8602 (14)	176.1 (15)

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

Fig. 1