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4-[(*E*)-(Hydroxyimino)methyl]-*N*,*N*-dimethylanilinium chloride

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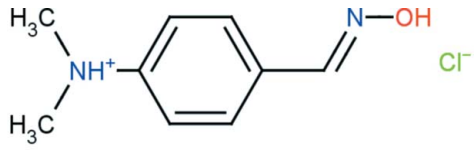
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.093; data-to-parameter ratio = 19.2.

In the title compound, $C_9H_{13}N_2O^+\cdot Cl^-$, the cation, apart from the methyl groups, is almost planar, with a maximum deviation of 0.040 (1) Å; the methyl C atoms deviate by 0.389 (2) and −1.247 (1) Å, from the mean plane. In the crystal, cations and anions associate through C—H···Cl hydrogen bonds, forming a helical arrangement. In addition, intermolecular O—H···Cl, N—H···Cl and C—H···N interactions are observed.

Related literature

For general background to hydroxylamine derivatives, see: Kataoka *et al.* (2002); Haldimann *et al.* (2011) and to benzaldehyde derivatives, see: Haraguchi *et al.* (2011); Johnston *et al.* (2011); Zhang *et al.* (2011). For a related structure, see: Bachechi & Zambonelli (1972).



Experimental

Crystal data

$C_9H_{13}N_2O^+\cdot Cl^-$
M_r = 200.66
Monoclinic, $P2_1/c$
a = 11.2696 (10) Å
b = 11.7093 (10) Å
c = 7.6961 (7) Å
β = 90.108 (2)°
V = 1015.57 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 292 K
0.24 × 0.20 × 0.19 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
11453 measured reflections
2405 independent reflections
2240 reflections with $I > 2\sigma$igma(I)
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.093
S = 1.07
2405 reflections
125 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···Cl1^{i}	0.82	2.34	3.147 (1)	167
N1—H1N···Cl1^{ii}	0.91 (1)	2.14 (1)	3.040 (1)	173 (1)
C6—H6···N2^{iii}	0.93	2.59	3.516 (2)	173
C7—H7···Cl1^{iv}	0.93	2.81	3.697 (1)	160
C9—H9B···Cl1^{v}	0.96	2.81	3.713 (2)	158

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2166).

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supplementary materials

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4-[(*E*)-(Hydroxyimino)methyl]-*N*,*N*-dimethylanilinium chloride

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Comment

Hydroxylamine derivatives possess anti-inflammatory and anti-allergic activities (Kataoka *et al.*, 2002). The novel hydroxylamine derivative NG-094 suppresses polyglutamine protein toxicity in Caenorhabditis elegans (Haldimann *et al.*, 2011). The benzaldehyde-modified starches and starch components have significantly higher water solubility than their native counterparts (Johnston *et al.*, 2011). Benzaldehyde derivatives possess antibacterial (Zhang *et al.*, 2011) and antitrypanasomal (Haraguchi *et al.*, 2011) activities. In continuation of our work, we have undertaken the crystal structure determination of the present complex, and the results are presented here.

The X-ray study confirmed the molecular structure of the title compound as illustrated in Fig. 1. Atom H1N was located from a difference Fourier map and refined freely. The protonation on the N1 site of the cation is also confirmed from the C1—N1 bond distance of 1.4777 (14) Å in comparison with the C—N bond distance of 1.380 (4) Å observed in the crystal structure of the neutral *α-p*-dimethylaminobenzaldoxime (Bachechi & Zambonelli, 1972). The bond distance N2—C7 of 1.267 (2) Å confirms the double bond character. The cation is almost planar with a maximum deviation of -0.040 (1) Å for atom C3 and the two methyl carbon atoms C8 and C9 deviate by 0.389 (2) and -1.247 (1) Å, respectively, from this plane.

Cations and anions associate through intermolecular C—H···Cl hydrogen bonds. These two hydrogen bonds are run in opposite direction of the *ab* plane forming a helical shape arrangement (Fig. 2 and Table 1). Intermolecular O—H···Cl, N—H···Cl and C—H···N interactions are also observed in the crystal structure (Fig. 3). In addition, the molecules are also connected by C—H···π interactions, the H3 atom (bound to C3) is at 2.87 Å from the centroid $Cg1^{i}$ of the phenyl ring (symmetry code *i* = x, 1/2 - y, 1/2 + z), with a C3—H3···$Cg1^{i}$ angle of 135° and a C3···Cg1 distance of 3.589 (2) Å.

Experimental

Commercially availbale hydroxylamine hydrochloride with *p*-dimethyl amino benzaldehyde was taken in equimolar ratio, were dissolved in double ethanol and stirred to yield a homogeneous mixture. The solution was allowed to evaporate at room temperature which yielded a brown crystalline salt. Single crystals were grown by slow evaporation from DMF.

Refinement

Atom H1N was located from a difference Fourier map and refined with a distance restraint of 0.89 (2) Å. The remaining H atoms were positioned geometrically and were treated as riding on their parent C and O atoms, with C—H = 0.93 Å and U_{iso} = 1.2U_{eq}(C) for aromatic H atoms, with C—H = 0.96 Å and U_{iso} = 1.5U_{eq}(C) for methyl H atoms, and with O—H = 0.82 Å and U_{iso} = 1.2U_{eq}(O).

Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97*

(Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

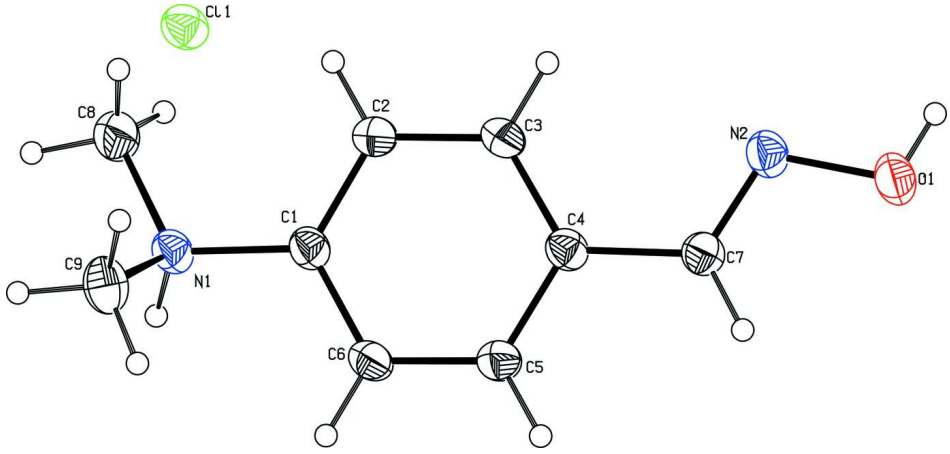

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level

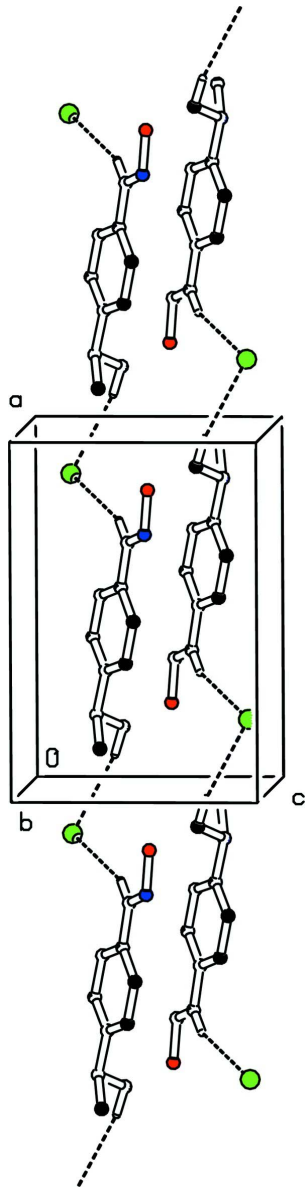

Figure 2

Molecular packing of the title compound, viewed along the *b* axis (H-bonds are shown as dashed lines). For the sake of clarity, H atoms which are not involved in hydrogen bonds have been omitted.

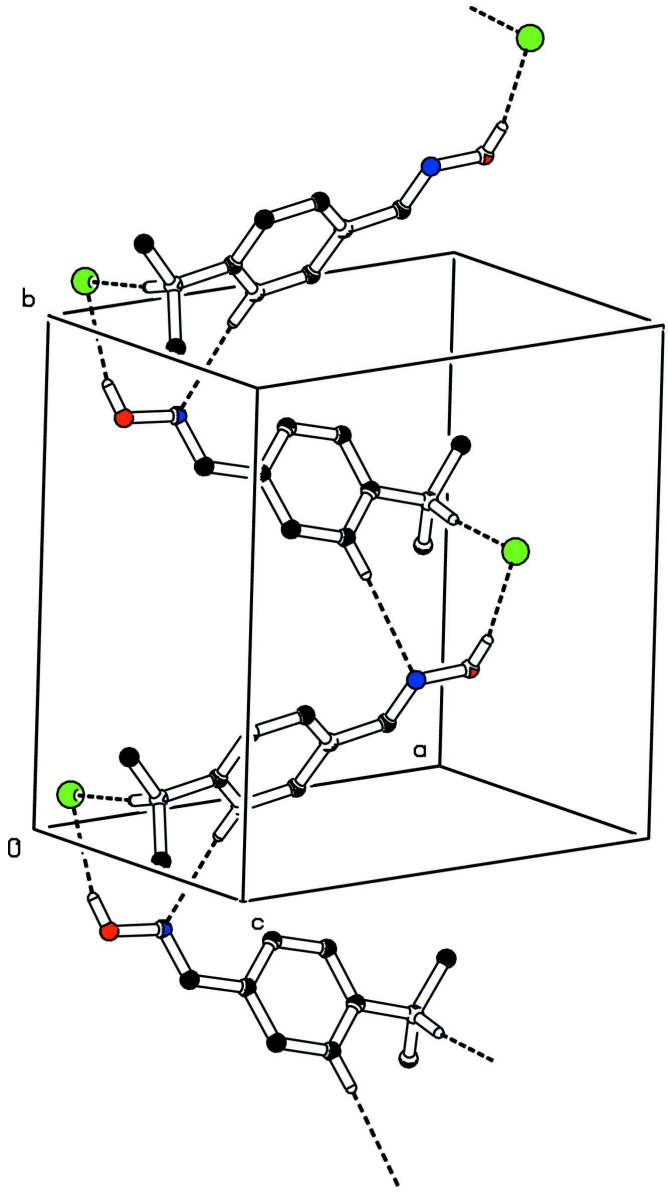

Figure 3

Molecular packing of the title compound, viewed along the *c* axis (H-bonds are shown as dashed lines). For the sake of clarity, H atoms which are not involved in hydrogen bonds have been omitted.

4-[(*E*)-(Hydroxyimino)methyl]-*N*,*N*-dimethylanilinium chloride

Crystal data

$C_9H_{13}N_2O^+\cdot Cl^-$
M_r = 200.66
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.2696 (10) Å
b = 11.7093 (10) Å
c = 7.6961 (7) Å
β = 90.108 (2)°
V = 1015.57 (16) Å^3
Z = 4
$F(000)$ = 424
D_x = 1.312 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7257 reflections
θ = 2.3–26.6°
μ = 0.34 mm^{-1}

T = 292 K
Needle, brown

0.24 × 0.20 × 0.19 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
11453 measured reflections
2405 independent reflections

2240 reflections with $I > 2\sigma$igma(I)
R_{int} = 0.025
θ_{max} = 28.0°, θ_{min} = 1.8°
h = −14→14
k = −15→15
l = −9→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.093
S = 1.07
2405 reflections
125 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 0.1829P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.16123 (3)	0.02780 (2)	0.86101 (4)	0.04216 (12)
O1	0.81494 (8)	0.28038 (8)	0.48301 (14)	0.0490 (2)
H1	0.8321	0.3407	0.5309	0.073*
N1	0.17646 (8)	0.07318 (8)	0.24932 (13)	0.0370 (2)
H1N	0.1744 (14)	0.0531 (13)	0.1354 (18)	0.050 (4)*
N2	0.69124 (9)	0.27159 (9)	0.46814 (14)	0.0405 (2)
C1	0.30082 (10)	0.10295 (10)	0.29034 (14)	0.0354 (2)
C2	0.32816 (11)	0.20083 (11)	0.38181 (18)	0.0472 (3)
H2	0.2683	0.2494	0.4197	0.057*
C3	0.44580 (11)	0.22569 (11)	0.41636 (18)	0.0471 (3)
H3	0.4647	0.2913	0.4787	0.057*
C4	0.53633 (10)	0.15418 (9)	0.35930 (14)	0.0351 (2)
C5	0.50650 (11)	0.05628 (10)	0.26681 (16)	0.0395 (3)
H5	0.5661	0.0076	0.2283	0.047*
C6	0.38908 (11)	0.03048 (9)	0.23151 (16)	0.0400 (3)

H6	0.3697	−0.0349	0.1689	0.048*
C7	0.66167 (10)	0.18062 (10)	0.38936 (15)	0.0370 (2)
H7	0.7199	0.1305	0.3506	0.044*
C8	0.08882 (12)	0.16749 (13)	0.2724 (2)	0.0568 (4)
H8A	0.0145	0.1459	0.2209	0.085*
H8B	0.1179	0.2354	0.2172	0.085*
H8C	0.0776	0.1818	0.3941	0.085*
C9	0.13654 (13)	−0.03099 (11)	0.34631 (19)	0.0494 (3)
H9A	0.1896	−0.0931	0.3223	0.074*
H9B	0.0577	−0.0512	0.3100	0.074*
H9C	0.1367	−0.0154	0.4688	0.074*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.04579 (19)	0.03872 (18)	0.04194 (19)	0.00255 (10)	−0.00722 (12)	−0.00218 (10)
O1	0.0383 (5)	0.0437 (5)	0.0649 (6)	−0.0055 (3)	−0.0106 (4)	−0.0029 (4)
N1	0.0367 (5)	0.0376 (5)	0.0367 (5)	−0.0020 (4)	−0.0068 (4)	−0.0022 (4)
N2	0.0368 (5)	0.0381 (5)	0.0467 (5)	−0.0010 (4)	−0.0075 (4)	0.0001 (4)
C1	0.0351 (5)	0.0366 (5)	0.0344 (5)	−0.0020 (4)	−0.0050 (4)	−0.0021 (4)
C2	0.0380 (6)	0.0472 (7)	0.0564 (7)	0.0046 (5)	−0.0034 (5)	−0.0200 (6)
C3	0.0412 (6)	0.0445 (6)	0.0555 (7)	0.0002 (5)	−0.0067 (5)	−0.0213 (6)
C4	0.0376 (5)	0.0343 (5)	0.0334 (5)	0.0002 (4)	−0.0050 (4)	−0.0008 (4)
C5	0.0393 (6)	0.0339 (5)	0.0453 (6)	0.0036 (4)	−0.0021 (5)	−0.0059 (5)
C6	0.0425 (6)	0.0324 (5)	0.0450 (6)	−0.0012 (4)	−0.0047 (5)	−0.0089 (4)
C7	0.0371 (6)	0.0359 (5)	0.0379 (5)	0.0022 (4)	−0.0046 (4)	−0.0004 (4)
C8	0.0409 (7)	0.0507 (7)	0.0786 (10)	0.0058 (6)	−0.0136 (6)	−0.0133 (7)
C9	0.0463 (7)	0.0514 (8)	0.0506 (7)	−0.0096 (5)	−0.0054 (6)	0.0100 (5)

Geometric parameters (Å, º)

O1—N2	1.4023 (13)	C4—C5	1.3903 (16)
O1—H1	0.8200	C4—C7	1.4640 (15)
N1—C1	1.4777 (14)	C5—C6	1.3838 (17)
N1—C8	1.4924 (17)	C5—H5	0.9300
N1—C9	1.4995 (16)	C6—H6	0.9300
N1—H1N	0.908 (13)	C7—H7	0.9300
N2—C7	1.2698 (15)	C8—H8A	0.9600
C1—C2	1.3796 (16)	C8—H8B	0.9600
C1—C6	1.3842 (16)	C8—H8C	0.9600
C2—C3	1.3827 (18)	C9—H9A	0.9600
C2—H2	0.9300	C9—H9B	0.9600
C3—C4	1.3916 (17)	C9—H9C	0.9600
C3—H3	0.9300		
N2—O1—H1	109.5	C6—C5—H5	119.6
C1—N1—C8	115.32 (9)	C4—C5—H5	119.6
C1—N1—C9	111.77 (9)	C5—C6—C1	119.30 (10)
C8—N1—C9	110.08 (11)	C5—C6—H6	120.4
C1—N1—H1N	106.8 (10)	C1—C6—H6	120.4

C8—N1—H1N	106.9 (10)	N2—C7—C4	120.34 (11)
C9—N1—H1N	105.3 (10)	N2—C7—H7	119.8
C7—N2—O1	111.13 (10)	C4—C7—H7	119.8
C2—C1—C6	121.09 (11)	N1—C8—H8A	109.5
C2—C1—N1	121.05 (10)	N1—C8—H8B	109.5
C6—C1—N1	117.86 (10)	H8A—C8—H8B	109.5
C1—C2—C3	119.09 (11)	N1—C8—H8C	109.5
C1—C2—H2	120.5	H8A—C8—H8C	109.5
C3—C2—H2	120.5	H8B—C8—H8C	109.5
C2—C3—C4	121.06 (11)	N1—C9—H9A	109.5
C2—C3—H3	119.5	N1—C9—H9B	109.5
C4—C3—H3	119.5	H9A—C9—H9B	109.5
C5—C4—C3	118.75 (11)	N1—C9—H9C	109.5
C5—C4—C7	119.18 (10)	H9A—C9—H9C	109.5
C3—C4—C7	122.05 (10)	H9B—C9—H9C	109.5
C6—C5—C4	120.72 (11)		

C8—N1—C1—C2	14.77 (17)	C3—C4—C5—C6	0.22 (18)
C9—N1—C1—C2	−111.95 (13)	C7—C4—C5—C6	−178.05 (11)
C8—N1—C1—C6	−164.17 (12)	C4—C5—C6—C1	−0.43 (19)
C9—N1—C1—C6	69.11 (14)	C2—C1—C6—C5	0.67 (19)
C6—C1—C2—C3	−0.7 (2)	N1—C1—C6—C5	179.61 (11)
N1—C1—C2—C3	−179.60 (12)	O1—N2—C7—C4	−179.96 (10)
C1—C2—C3—C4	0.5 (2)	C5—C4—C7—N2	177.52 (11)
C2—C3—C4—C5	−0.2 (2)	C3—C4—C7—N2	−0.69 (18)
C2—C3—C4—C7	177.96 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···Cl1^{i}	0.82	2.34	3.147 (1)	167
N1—H1*N*···Cl1^{ii}	0.91 (1)	2.14 (1)	3.040 (1)	173 (1)
C6—H6···N2^{iii}	0.93	2.59	3.516 (2)	173
C7—H7···Cl1^{iv}	0.93	2.81	3.697 (1)	160
C9—H9*B*···Cl1^{v}	0.96	2.81	3.713 (2)	158

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