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2-Hydroxy-*N*,*N*′-diisopropylpropane-1,3-diaminium dichloride

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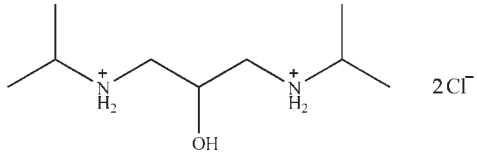
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.096; data-to-parameter ratio = 18.7.

In the crystal structure of the title amino alcohol derivative, $C_9H_{24}N_2O^{2+}\cdot 2Cl^-$, the cations and anions are linked by intermolecular O—H···Cl and N—H···Cl hydrogen bonds into a three-dimensional network.

Related literature

For the applications of amino alcohols and their derivatives in organic synthesis, see: Ellison & Gandhi (2005); Li *et al.* (2004).



Experimental

Crystal data

$C_9H_{24}N_2O^{2+}\cdot 2Cl^-$
M_r = 247.20
Triclinic, $P\overline{1}$
a = 6.240 (1) Å
b = 10.0081 (14) Å
c = 11.3519 (16) Å
α = 86.198 (1)°
β = 88.052 (2)°
γ = 83.308 (1)°
V = 702.31 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 298 K
0.50 × 0.45 × 0.44 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.810, T_{max} = 0.830
3684 measured reflections
2448 independent reflections
1999 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.096
S = 1.03
2448 reflections
131 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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	2.33	3.1445 (15)	172
N2—H2B···Cl1^{i}	0.90	2.24	3.1336 (15)	173
N2—H2A···Cl2^{ii}	0.90	2.22	3.1130 (15)	172
N1—H1B···Cl2^{iii}	0.90	2.20	3.0920 (15)	174
N1—H1A···Cl1^{iv}	0.90	2.38	3.2119 (16)	154

Symmetry codes: (i) $x, y, z-1$; (ii) $-x+1, -y+1, -z$; (iii) $x-1, y, z$; (iv) $-x, -y, -z+1$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2467).

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supporting information

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2-Hydroxy-*N*,*N*′-diisopropylpropane-1,3-diaminium dichloride

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S1. Comment

Amino alcohols are important structural elements for the asymmetric catalysis of chiral ligands (Li *et al.*, 2004) as well as of biologically active compounds (Ellison & Gandhi, 2005). In order to develop new applications for amino alcohols and their derivatives, structural modifications of these compounds have been extensively investigated. As a contribution in this filed, we report here the crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1. Bond lengths and angles in the cation are not unusual. In the crystal packing (Fig. 2), intermolecular O—H···Cl and N—H···Cl hydrogen bonds (Table 1) link molecules into a three-dimensional network.

S2. Experimental

To a solution of isopropamide (24.0 g, 0.4 mol) in acetone (200 ml), epichlorohydrin (9.2 g, 0.1 mol) and K_2CO_3 (13.8 g, 0.1 mol) were added. The mixture was stirred at room temperature for 8 h, followed by filtration and purification by crystallization from ethyl acetate, giving title compound as colourless single crystals suitable for X-ray analysis.

S3. Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms, with C—H = 0.93–0.98 Å, N—H = 0.90 Å, O—H = 0.82 Å, and with U_{iso}(H) = 1.2 U_{eq}(C, N) or 1.5 U_{eq}(C, O) for methyl and hydroxy H atoms.

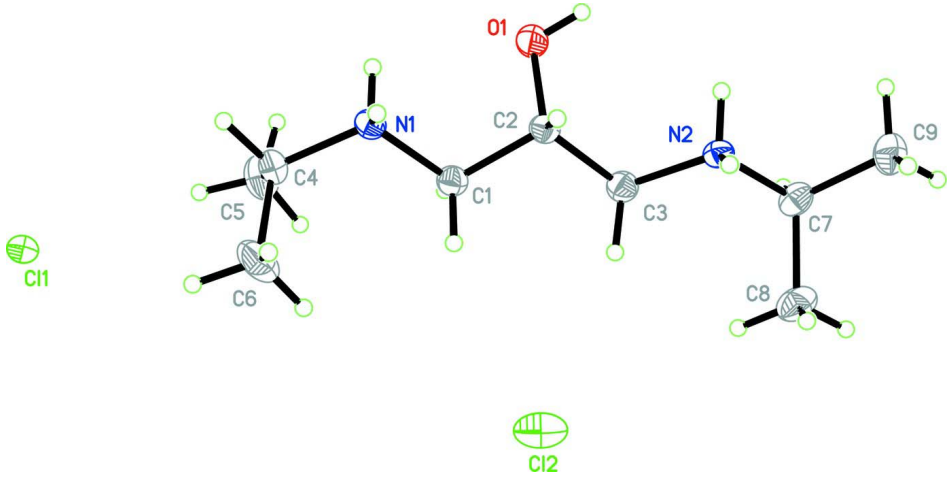

Figure 1

The molecular structure of the compound, with atom labels and 50% probability displacement ellipsoids.

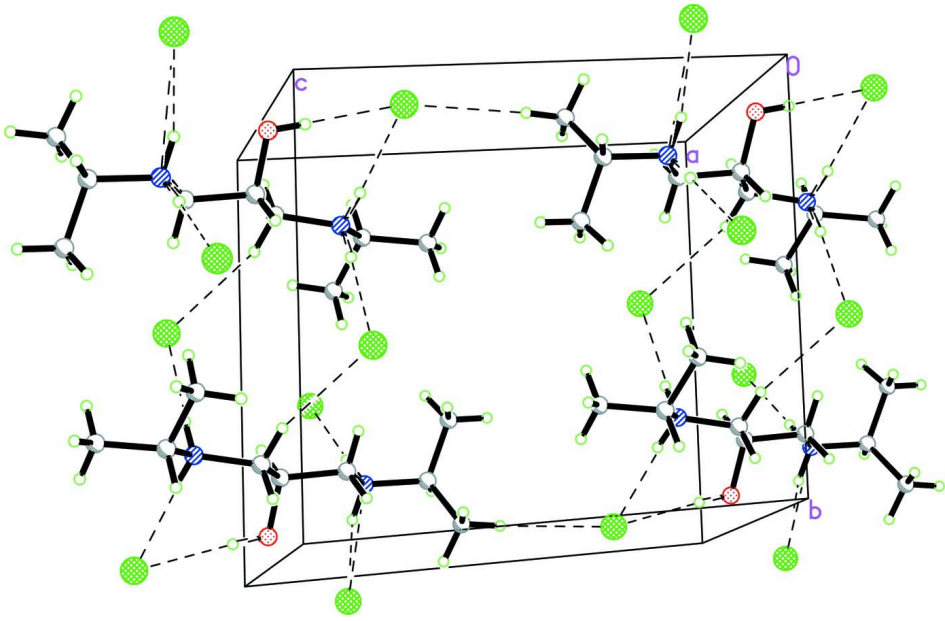

Figure 2

Crystal packing of the title compound. showing a three-dimensional structure linked by hydrogen bonds (dashed lines).

2-Hydroxy-N,N′-diisopropylpropane-1,3-diaminium dichloride

Crystal data

$C_9H_{24}N_2O^{2+}\cdot 2Cl^-$
$M_r = 247.20$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.240\ (1)$ Å
$b = 10.0081\ (14)$ Å
$c = 11.3519\ (16)$ Å
$\alpha = 86.198\ (1)°$
$\beta = 88.052\ (2)°$
$\gamma = 83.308\ (1)°$
$V = 702.31\ (18)$ Å^3
$Z = 2$
$F(000) = 268$
$D_x = 1.169$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2066 reflections
$\theta = 2.6–27.6°$
$\mu = 0.44$ mm^{-1}
$T = 298$ K
Block, colourless
0.50 × 0.45 × 0.44 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.810$, $T_{max} = 0.830$
3684 measured reflections
2448 independent reflections
1999 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -7\rightarrow5$
$k = -11\rightarrow11$
$l = -13\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.096$
$S = 1.03$
2448 reflections
131 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.1942P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} < 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.25$ e Å^{-3}
	$\Delta\rho_{min} = -0.23$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.19859 (8)	0.04318 (5)	0.75891 (4)	0.04461 (17)
Cl2	0.73770 (9)	0.43851 (5)	0.17643 (6)	0.0585 (2)
N1	0.0878 (2)	0.20330 (14)	0.24284 (12)	0.0307 (3)
H1A	0.0376	0.1276	0.2223	0.037*
H1B	−0.0074	0.2733	0.2183	0.037*
N2	0.4576 (2)	0.26100 (14)	−0.14466 (12)	0.0302 (3)
H2A	0.3877	0.3447	−0.1542	0.036*
H2B	0.3742	0.2041	−0.1738	0.036*
O1	0.1777 (3)	0.09416 (15)	0.02964 (12)	0.0524 (4)
H1	0.1697	0.0850	−0.0413	0.079*
C1	0.2960 (3)	0.21572 (19)	0.17837 (16)	0.0345 (4)
H1C	0.4012	0.1409	0.2033	0.041*
H1D	0.3503	0.2987	0.1968	0.041*
C2	0.2656 (3)	0.21601 (17)	0.04656 (15)	0.0316 (4)
H2	0.1629	0.2932	0.0209	0.038*
C3	0.4813 (3)	0.22602 (19)	−0.01638 (16)	0.0355 (4)
H3A	0.5534	0.2942	0.0182	0.043*
H3B	0.5710	0.1405	−0.0051	0.043*
C4	0.0935 (3)	0.1991 (2)	0.37596 (16)	0.0394 (5)
H4	−0.0526	0.1880	0.4065	0.047*
C5	0.2409 (4)	0.0782 (2)	0.4228 (2)	0.0577 (6)
H5A	0.2060	−0.0012	0.3880	0.087*
H5B	0.2223	0.0675	0.5071	0.087*
H5C	0.3881	0.0913	0.4031	0.087*
C6	0.1505 (5)	0.3315 (2)	0.4160 (2)	0.0646 (7)
H6A	0.2959	0.3434	0.3908	0.097*
H6B	0.1385	0.3309	0.5006	0.097*
H6C	0.0534	0.4043	0.3821	0.097*
C7	0.6661 (3)	0.25586 (19)	−0.21644 (18)	0.0385 (4)
H7	0.7421	0.1646	−0.2069	0.046*

C8	0.8085 (3)	0.3540 (2)	−0.1736 (2)	0.0548 (6)
H8A	0.7330	0.4433	−0.1790	0.082*
H8B	0.9383	0.3518	−0.2216	0.082*
H8C	0.8445	0.3291	−0.0929	0.082*
C9	0.6110 (4)	0.2857 (3)	−0.34475 (19)	0.0578 (6)
H9A	0.5186	0.2223	−0.3678	0.087*
H9B	0.7412	0.2783	−0.3926	0.087*
H9C	0.5383	0.3754	−0.3554	0.087*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0475 (3)	0.0492 (3)	0.0417 (3)	−0.0200 (2)	0.0034 (2)	−0.0139 (2)
Cl2	0.0460 (3)	0.0349 (3)	0.0946 (5)	0.0039 (2)	−0.0206 (3)	−0.0090 (3)
N1	0.0315 (8)	0.0292 (8)	0.0316 (8)	−0.0018 (6)	−0.0026 (6)	−0.0046 (6)
N2	0.0298 (8)	0.0272 (7)	0.0345 (8)	−0.0060 (6)	−0.0002 (6)	−0.0043 (6)
O1	0.0714 (10)	0.0555 (9)	0.0375 (8)	−0.0341 (8)	0.0079 (7)	−0.0129 (7)
C1	0.0317 (10)	0.0387 (10)	0.0336 (10)	−0.0049 (8)	−0.0013 (8)	−0.0051 (8)
C2	0.0328 (10)	0.0309 (9)	0.0319 (10)	−0.0050 (7)	−0.0029 (8)	−0.0031 (7)
C3	0.0337 (10)	0.0371 (10)	0.0358 (10)	−0.0044 (8)	−0.0030 (8)	−0.0010 (8)
C4	0.0432 (11)	0.0474 (11)	0.0284 (10)	−0.0070 (9)	0.0023 (8)	−0.0065 (8)
C5	0.0689 (16)	0.0640 (15)	0.0370 (12)	0.0020 (12)	−0.0064 (11)	0.0072 (10)
C6	0.0913 (19)	0.0615 (15)	0.0450 (13)	−0.0129 (13)	−0.0062 (13)	−0.0226 (11)
C7	0.0317 (10)	0.0351 (10)	0.0479 (12)	−0.0020 (8)	0.0083 (8)	−0.0046 (9)
C8	0.0344 (11)	0.0639 (15)	0.0677 (15)	−0.0160 (10)	0.0017 (10)	0.0003 (12)
C9	0.0636 (15)	0.0685 (15)	0.0427 (13)	−0.0159 (12)	0.0136 (11)	−0.0068 (11)

Geometric parameters (Å, º)

N1—C1	1.483 (2)	C4—C6	1.513 (3)
N1—C4	1.510 (2)	C4—H4	0.9800
N1—H1A	0.9000	C5—H5A	0.9600
N1—H1B	0.9000	C5—H5B	0.9600
N2—C3	1.483 (2)	C5—H5C	0.9600
N2—C7	1.509 (2)	C6—H6A	0.9600
N2—H2A	0.9000	C6—H6B	0.9600
N2—H2B	0.9000	C6—H6C	0.9600
O1—C2	1.421 (2)	C7—C9	1.510 (3)
O1—H1	0.8200	C7—C8	1.512 (3)
C1—C2	1.514 (2)	C7—H7	0.9800
C1—H1C	0.9700	C8—H8A	0.9600
C1—H1D	0.9700	C8—H8B	0.9600
C2—C3	1.513 (2)	C8—H8C	0.9600
C2—H2	0.9800	C9—H9A	0.9600
C3—H3A	0.9700	C9—H9B	0.9600
C3—H3B	0.9700	C9—H9C	0.9600
C4—C5	1.511 (3)		

C1—N1—C4	116.24 (14)	N1—C4—H4	107.3
C1—N1—H1A	108.2	C5—C4—H4	107.3
C4—N1—H1A	108.2	C6—C4—H4	107.3
C1—N1—H1B	108.2	C4—C5—H5A	109.5
C4—N1—H1B	108.2	C4—C5—H5B	109.5
H1A—N1—H1B	107.4	H5A—C5—H5B	109.5
C3—N2—C7	115.27 (14)	C4—C5—H5C	109.5
C3—N2—H2A	108.5	H5A—C5—H5C	109.5
C7—N2—H2A	108.5	H5B—C5—H5C	109.5
C3—N2—H2B	108.5	C4—C6—H6A	109.5
C7—N2—H2B	108.5	C4—C6—H6B	109.5
H2A—N2—H2B	107.5	H6A—C6—H6B	109.5
C2—O1—H1	109.5	C4—C6—H6C	109.5
N1—C1—C2	110.12 (14)	H6A—C6—H6C	109.5
N1—C1—H1C	109.6	H6B—C6—H6C	109.5
C2—C1—H1C	109.6	N2—C7—C9	108.02 (16)
N1—C1—H1D	109.6	N2—C7—C8	110.37 (16)
C2—C1—H1D	109.6	C9—C7—C8	112.24 (18)
H1C—C1—H1D	108.1	N2—C7—H7	108.7
O1—C2—C3	113.58 (15)	C9—C7—H7	108.7
O1—C2—C1	105.22 (14)	C8—C7—H7	108.7
C3—C2—C1	108.81 (14)	C7—C8—H8A	109.5
O1—C2—H2	109.7	C7—C8—H8B	109.5
C3—C2—H2	109.7	H8A—C8—H8B	109.5
C1—C2—H2	109.7	C7—C8—H8C	109.5
N2—C3—C2	112.00 (14)	H8A—C8—H8C	109.5
N2—C3—H3A	109.2	H8B—C8—H8C	109.5
C2—C3—H3A	109.2	C7—C9—H9A	109.5
N2—C3—H3B	109.2	C7—C9—H9B	109.5
C2—C3—H3B	109.2	H9A—C9—H9B	109.5
H3A—C3—H3B	107.9	C7—C9—H9C	109.5
N1—C4—C5	110.60 (16)	H9A—C9—H9C	109.5
N1—C4—C6	110.44 (16)	H9B—C9—H9C	109.5
C5—C4—C6	113.51 (19)		

C4—N1—C1—C2	178.96 (14)	C1—C2—C3—N2	−165.75 (14)
N1—C1—C2—O1	−56.87 (18)	C1—N1—C4—C5	−61.5 (2)
N1—C1—C2—C3	−178.91 (14)	C1—N1—C4—C6	65.0 (2)
C7—N2—C3—C2	−172.65 (14)	C3—N2—C7—C9	176.59 (16)
O1—C2—C3—N2	77.43 (19)	C3—N2—C7—C8	−60.4 (2)

Hydrogen-bond geometry (Å, º)

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