Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,6-Dimethylanilinium chloride monohydrate

Wajda Smirani,* Olfa Amri and Mohamed Rzaigui

Laboratoire de Chimie des Matériaux, Faculté des Sciences de Bizerte, 7021 Zarzouna Bizerte, Tunisia
Correspondence e-mail: wajda_sta@yahoo.fr

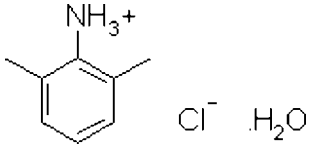
Received 20 November 2008; accepted 21 November 2008

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.080; data-to-parameter ratio = 14.4.

In the title hydrated molecular salt, $C_8H_{12}N^+\cdot Cl^-\cdot H_2O$, the component species interact by way of N—H···O, N—H···Cl and O—H···Cl hydrogen bonds, resulting in a three-dimensional network.

Related literature

For related structures, see: Abid *et al.* (2007); Mrad *et al.* (2006). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_{12}N^+\cdot Cl^-\cdot H_2O$
M_r = 175.65
Monoclinic, $P2_1/c$
a = 8.676 (3) Å
b = 14.144 (3) Å
c = 7.913 (6) Å
β = 101.88 (5)°
V = 950.2 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 293 (2) K
0.20 × 0.13 × 0.10 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Absorption correction: none
3722 measured reflections
2244 independent reflections
1827 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
2 standard reflections
frequency: 120 min
intensity decay: 5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.080
S = 1.04
2244 reflections
156 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···Cl1	0.90 (2)	2.41 (2)	3.305 (3)	173 (2)
O1—H2···Cl1^{i}	0.87 (3)	2.32 (3)	3.163 (3)	165 (2)
N1—H6···Cl1^{ii}	0.893 (18)	2.392 (18)	3.235 (3)	157.5 (15)
N1—H7···O1	0.896 (16)	1.835 (16)	2.731 (3)	177.3 (17)
N1—H8···Cl1^{iii}	0.883 (16)	2.414 (16)	3.265 (3)	162.8 (15)

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2860).

References

Abid, S., Hemissi, H. & Rzaigui, M. (2007). *Acta Cryst.* E**63**, o3117.
Bernstein, J., David, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Mrad, M. L., Ben Nasr, C. & Rzaigui, M. (2006). *Anal. Sci.* **22**, x227–x228.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2008). E**64**, o2463 [**doi:10.1107/S1600536808039159**]

2,6-Dimethylanilinium chloride monohydrate

W. Smirani, O. Amri and M. Rzaigui

Comment

As part of our ongoing studies of organic-inorganic hybrid networks containing the 2,6-xylidinium cation (Mrad *et al.*, 2006; Abid *et al.*, 2007) we now report the synthesis and structure of the title compound, (I).

As shown in Fig. 1, the asymmetric unit of (I) contains a 2,6-xylidinium cation, a chloride anion and a water molecule. A perspective view of the structure along the *a* axis is given in Fig. 2. It shows that two 2,6-xylidinium cations are interconnected through two chloride anions into dimers *via* two N—H···Cl bonds, characterized by N···Cl separations of 3.264 (3) and 3.235 (3) Å) and forming an 8-membered ring with graph-set $R_2^4(8)$ (Bernstein *et al.*, 1995).

The title compound is a crystalline hydrate including one water of crystallization, which interconnect these dimers to each other to form layers parallel to the *(b, c)* plane, through N—H···O and O—H···Cl hydrogen bonds (Table 1).

Hydrogen bonds, electrostatic and van der Waals interactions participate to the cohesion of the three-dimensional network and add stability to this compound (Fig. 2). An examination of the organic group moiety geometrical features shows that the C—C and C—N bond lengths and the C—C—C and C—C—N angles are in the range usually found for this molecule (Abid *et al.*, 2007).

Experimental

2,6-xylidinie and HCl were mixed in water in a 1: 1 molar ratio. The obtained solution was slowly evapored at room temperature to yield colourless blocks of (I).

Refinement

The H atoms were located in a difference map and their positions and U_{iso} values were freely refined.

Figures

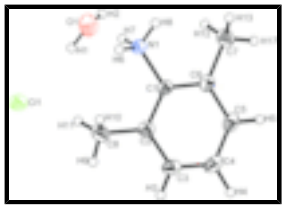


Fig. 1. View of (I) with displacement ellipsoids for non-H atoms drawn at the 30% probability level (arbitrary spheres for the H atoms).

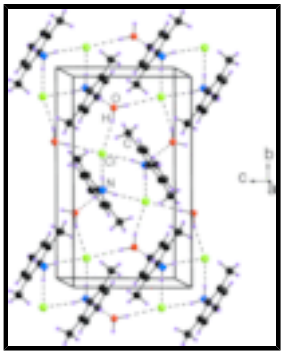


Fig. 2. A perspective view of the packing in (I).

2,6-Dimethylanilinium chloride monohydrate

Crystal data

$C_8H_{12}N^+\cdot Cl^-\cdot H_2O$	F_{000} = 376
M_r = 175.65	D_x = 1.228 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 8.676 (3) Å	θ = 9.2–10.8º
b = 14.144 (3) Å	μ = 0.35 mm^{-1}
c = 7.913 (6) Å	T = 293 (2) K
β = 101.88 (5)º	Block, colourless
V = 950.2 (8) Å^3	0.20 × 0.13 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius TurboCAD-4 diffractometer	θ_{max} = 28.0º
Monochromator: graphite	θ_{min} = 2.8º
T = 293 K	h = −5→11
Non–profiled ω scans	k = −18→0
Absorption correction: none	l = −10→10
3722 measured reflections	2 standard reflections
2244 independent reflections	every 120 min
1827 reflections with $I > 2\sigma(I)$	intensity decay: 5%
R_{int} = 0.033	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.031	H-atom parameters not refined
$wR(F^2)$ = 0.080	$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 0.1063P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
2244 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
156 parameters	$\Delta\rho_{min} = -0.24$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
H8	0.3326 (19)	0.0879 (11)	0.050 (2)	0.048 (4)*
H7	0.3721 (19)	0.1013 (11)	0.233 (2)	0.045 (4)*
H5	−0.159 (2)	0.2065 (13)	0.033 (2)	0.058 (4)*
H6	0.3494 (19)	0.0085 (13)	0.161 (2)	0.053 (4)*
H3	−0.121 (2)	−0.0291 (12)	0.318 (2)	0.055 (4)*
H13	0.171 (2)	0.2037 (14)	−0.109 (2)	0.066 (5)*
H11	0.265 (2)	−0.0493 (14)	0.427 (3)	0.070 (6)*
H17	0.056 (2)	0.2698 (15)	−0.066 (2)	0.073 (5)*
H2	0.488 (3)	0.2297 (19)	0.405 (3)	0.083 (7)*
H4	−0.278 (2)	0.0943 (13)	0.191 (2)	0.067 (5)*
H1	0.472 (3)	0.1562 (17)	0.518 (3)	0.090 (7)*
H10	0.215 (2)	−0.1099 (13)	0.268 (2)	0.062 (5)*
H9	0.124 (2)	−0.1045 (14)	0.406 (3)	0.071 (6)*
H12	0.215 (3)	0.2560 (15)	0.065 (3)	0.087 (7)*
N1	0.31170 (12)	0.06739 (8)	0.14878 (14)	0.0353 (2)
C7	0.1308 (2)	0.22609 (11)	−0.0155 (2)	0.0510 (3)
C8	0.1800 (2)	−0.07172 (12)	0.3454 (2)	0.0512 (3)
C1	0.14475 (13)	0.07516 (8)	0.15925 (14)	0.0322 (2)
C6	0.05793 (14)	0.15126 (9)	0.07822 (14)	0.0359 (3)
C5	−0.09996 (16)	0.15637 (10)	0.08975 (17)	0.0435 (3)
C2	0.08245 (14)	0.00745 (9)	0.25368 (15)	0.0367 (3)
C3	−0.07586 (16)	0.01607 (10)	0.26089 (17)	0.0445 (3)
C4	−0.16594 (15)	0.08936 (11)	0.18003 (18)	0.0466 (3)
O1	0.48995 (14)	0.16863 (9)	0.41224 (15)	0.0580 (3)
Cl1	0.45854 (4)	0.11499 (2)	0.81025 (4)	0.04716 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0319 (5)	0.0398 (5)	0.0364 (5)	0.0052 (4)	0.0125 (4)	0.0025 (4)
C7	0.0553 (9)	0.0467 (7)	0.0546 (8)	0.0130 (7)	0.0200 (7)	0.0119 (6)
C8	0.0580 (9)	0.0523 (8)	0.0493 (8)	0.0100 (7)	0.0253 (7)	0.0123 (7)
C1	0.0282 (5)	0.0398 (6)	0.0296 (5)	0.0024 (4)	0.0083 (4)	−0.0058 (4)
C6	0.0370 (6)	0.0389 (6)	0.0318 (5)	0.0050 (5)	0.0074 (4)	−0.0049 (4)
C5	0.0356 (6)	0.0504 (7)	0.0428 (6)	0.0102 (6)	0.0039 (5)	−0.0091 (6)
C2	0.0378 (6)	0.0416 (6)	0.0328 (5)	0.0007 (5)	0.0121 (4)	−0.0047 (5)
C3	0.0402 (7)	0.0524 (8)	0.0452 (6)	−0.0068 (6)	0.0186 (5)	−0.0077 (6)
C4	0.0298 (6)	0.0602 (8)	0.0508 (7)	−0.0003 (5)	0.0107 (5)	−0.0145 (6)
O1	0.0654 (7)	0.0543 (7)	0.0509 (6)	0.0072 (5)	0.0039 (5)	−0.0041 (5)
Cl1	0.04621 (19)	0.04719 (19)	0.0534 (2)	0.01013 (14)	0.02267 (14)	0.00643 (13)

Geometric parameters (Å, °)

N1—C1	1.4718 (16)	C1—C2	1.3907 (17)
N1—H8	0.883 (18)	C1—C6	1.3921 (17)
N1—H7	0.896 (17)	C6—C5	1.3934 (18)
N1—H6	0.893 (18)	C5—C4	1.380 (2)
C7—C6	1.504 (2)	C5—H5	0.935 (18)
C7—H13	0.93 (2)	C2—C3	1.3917 (18)
C7—H17	0.92 (2)	C3—C4	1.374 (2)
C7—H12	0.96 (2)	C3—H3	0.918 (18)
C8—C2	1.498 (2)	C4—H4	1.00 (2)
C8—H11	0.93 (2)	O1—H2	0.87 (3)
C8—H10	0.92 (2)	O1—H1	0.90 (3)
C8—H9	0.88 (2)		
C1—N1—H8	114.1 (10)	C2—C1—N1	118.34 (11)
C1—N1—H7	110.5 (10)	C6—C1—N1	118.50 (11)
H8—N1—H7	106.5 (15)	C1—C6—C5	117.13 (12)
C1—N1—H6	114.0 (11)	C1—C6—C7	121.91 (11)
H8—N1—H6	105.3 (15)	C5—C6—C7	120.95 (12)
H7—N1—H6	105.9 (14)	C4—C5—C6	121.09 (13)
C6—C7—H13	114.5 (12)	C4—C5—H5	121.5 (11)
C6—C7—H17	110.9 (13)	C6—C5—H5	117.4 (11)
H13—C7—H17	103.3 (16)	C1—C2—C3	117.24 (12)
C6—C7—H12	108.9 (13)	C1—C2—C8	122.14 (12)
H13—C7—H12	108.2 (18)	C3—C2—C8	120.62 (12)
H17—C7—H12	111.0 (18)	C4—C3—C2	121.22 (13)
C2—C8—H11	111.7 (12)	C4—C3—H3	119.6 (11)
C2—C8—H10	110.5 (11)	C2—C3—H3	119.2 (11)
H11—C8—H10	109.9 (17)	C3—C4—C5	120.15 (12)
C2—C8—H9	109.7 (13)	C3—C4—H4	118.9 (11)
H11—C8—H9	104.3 (18)	C5—C4—H4	120.9 (11)
H10—C8—H9	110.5 (17)	H2—O1—H1	105 (2)

C2—C1—C6	123.14 (11)		
C2—C1—C6—C5	−2.12 (17)	N1—C1—C2—C3	−179.68 (10)
N1—C1—C6—C5	179.69 (10)	C6—C1—C2—C8	−177.60 (12)
C2—C1—C6—C7	176.85 (12)	N1—C1—C2—C8	0.60 (18)
N1—C1—C6—C7	−1.34 (17)	C1—C2—C3—C4	−0.96 (18)
C1—C6—C5—C4	0.94 (17)	C8—C2—C3—C4	178.78 (14)
C7—C6—C5—C4	−178.04 (13)	C2—C3—C4—C5	−0.1 (2)
C6—C1—C2—C3	2.13 (17)	C6—C5—C4—C3	0.1 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···Cl1	0.90 (2)	2.41 (2)	3.305 (3)	173 (2)
O1—H2···Cl1^{i}	0.87 (3)	2.32 (3)	3.163 (3)	165 (2)
N1—H6···Cl1^{ii}	0.893 (18)	2.392 (18)	3.235 (3)	157.5 (15)
N1—H7···O1	0.896 (16)	1.835 (16)	2.731 (3)	177.3 (17)
N1—H8···Cl1^{iii}	0.883 (16)	2.414 (16)	3.265 (3)	162.8 (15)

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

Fig. 1

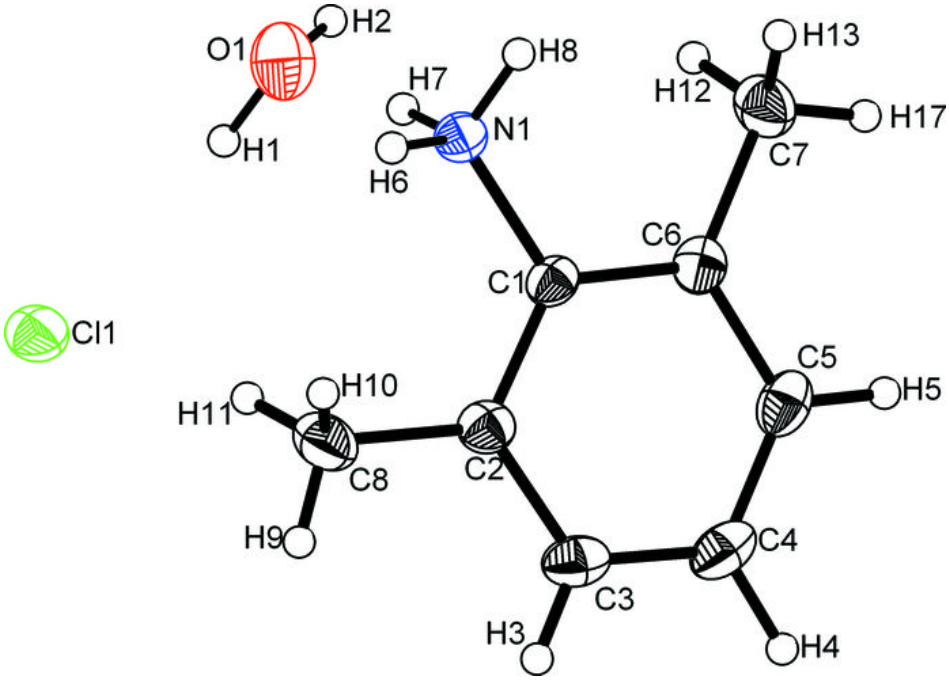

O1
H2
H1
H7
H8
N1
H6
H12
H13
H17
C7
C6
C1
Cl1
C5
H5
H10
H11
C8
C2
H9
C4
C3
H3
H4

Fig. 2