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N-(2,6-Dimethylanilino)-5,6-dihydro-4H-1,3-thiazin-3-ium chloride monohydrate

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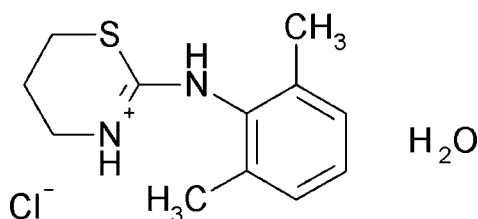
Received 22 April 2008; accepted 8 May 2008

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
 R factor = 0.033; wR factor = 0.089; data-to-parameter ratio = 16.3.

In the title compound, alternatively called xylazine hydrochloride monohydrate, $\text{C}_{12}\text{H}_{17}\text{N}_2\text{S}^+\cdot\text{Cl}^-\cdot\text{H}_2\text{O}$, the six-membered thiazine ring is in a half-chair conformation. In the crystal structure, six component centrosymmetric clusters are formed *via* intermolecular $\text{O}-\text{H}\cdots\text{Cl}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds involving xylazine cations, chloride anions and water molecules.

Related literature

For related literature see: Carpy *et al.* (1979); Kalman *et al.* (1977).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{17}\text{N}_2\text{S}^+\cdot\text{Cl}^-\cdot\text{H}_2\text{O}$
 $M_r = 274.81$
Monoclinic, $P2_1/c$
 $a = 13.4546$ (2) Å
 $b = 8.6547$ (1) Å
 $c = 12.7732$ (2) Å
 $\beta = 109.210$ (2)°

$V = 1404.56$ (4) Å³
 $Z = 4$
Cu $K\alpha$ radiation
 $\mu = 3.69$ mm⁻¹
 $T = 100$ K
 $0.44 \times 0.25 \times 0.14$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Absorption correction: numerical (de Meulenaer & Tompa, 1965)
 $T_{\min} = 0.30$, $T_{\max} = 0.61$

19046 measured reflections
2747 independent reflections
2509 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.088$
 $S = 1.01$
2509 reflections

154 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.43$ e Å⁻³
 $\Delta\rho_{\min} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N5}-\text{H5}\cdots\text{O17}$	0.87	1.97	2.815 (2)	163
$\text{O17}-\text{H171}\cdots\text{Cl16}^{\text{i}}$	0.82	2.36	3.158 (1)	164
$\text{N7}-\text{H7}\cdots\text{Cl16}^{\text{i}}$	0.87	2.37	3.204 (1)	162
$\text{O17}-\text{H172}\cdots\text{Cl16}^{\text{ii}}$	0.83	2.35	3.171 (1)	173

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CRYSTALS*.

We thank Oxford Diffraction Ltd for the low-temperature data collection and reduction. Cooperation of the University of Cincinnati Crystallography Centre and the Latvia Institute of Organic Synthesis is acknowledged. Financial aid was provided by Latvia Science Council grant 05.1737.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2620).

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supporting information

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***N*-(2,6-Dimethylanilino)-5,6-dihydro-4*H*-1,3-thiazin-3-ium chloride monohydrate**

Mikelis V. Veidis, Liana Orola and Reinis Arajs

S1. Comment

Xylazine hydrochloride monohydrate is a pharmaceutical used in veterinary medicine as an anesthetic. The substance is an alpha2-agonist with sedative, analgesic, and muscle relaxing properties.

The crystal structure of the title compound has been determined at 100 K. The structure is depicted in Fig. 1. The phenyl ring forms a dihedral angle of 83.24 (14)° with the plane defined by S1, C6 and N5 of the thiazine ring. The six-member thiazine ring assumes the half-chair conformation.

Hydrogen atoms are bonded to both nitrogen atoms forming a cation. Both hydrogen atoms participate in hydrogen bonding. The two xylazine moieties are held together through an extended H-bond network involving the nitrogen, oxygen, and chlorine anions. In the crystal structure, centrosymmetric clusters are formed by N—H···O—H···Cl···H—N hydrogen bond sequence between the two xylazine moieties.

There are H-bonds which do not join the xylazine moieties between oxygen and chlorine (Fig. 2). These may impart additional rigidity in the cluster. As a result of Cl···H—O hydrogen bonding a parallelogram is formed by the Cl—O—Cl—O atoms.

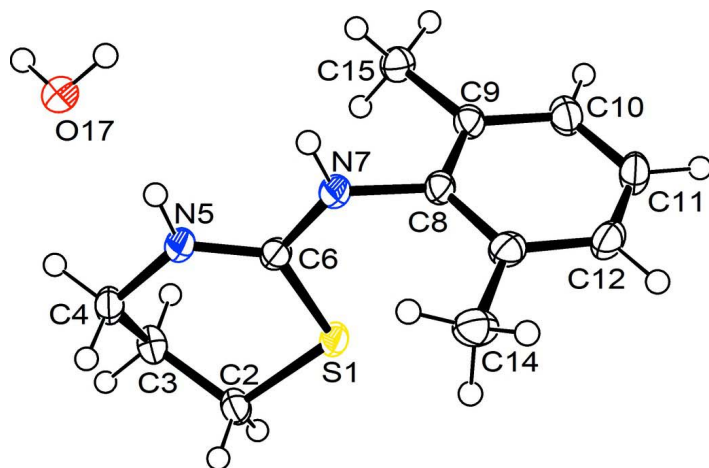
The hydrogen bond lengths are given in the Table 1.

S2. Experimental

The title compound was supplied by Grindeks Company. For crystal structure determination suitable crystals were grown by slow evaporation of an ethanol (96%) solution at room temperature.

S3. Refinement

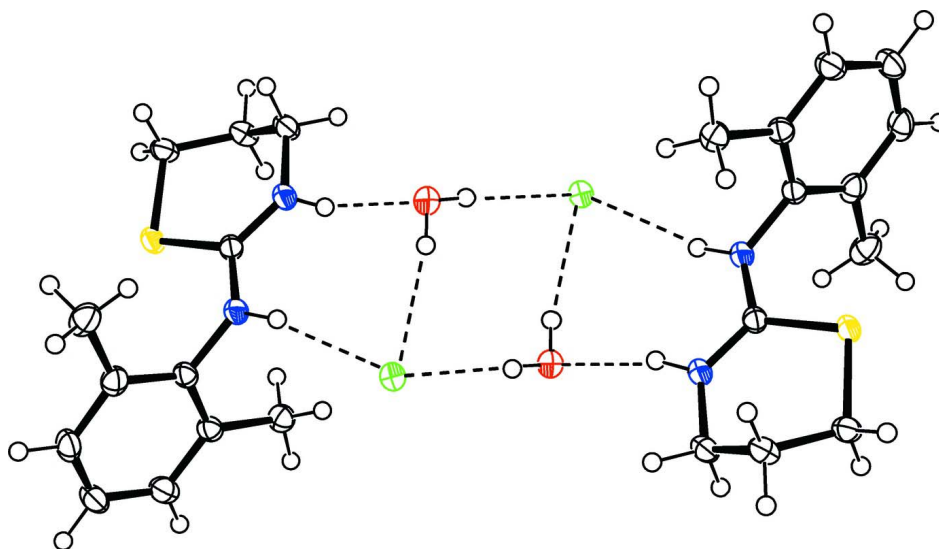
The hydrogen atoms were located by difference Fourier method. During refinement hydrogen atoms were constrained to the riding mode. $U_{\text{iso}}(\text{H})=xU_{\text{eq}}(\text{C},\text{N},\text{O})$, where the average values of x are 1.15 for H atoms bonded to the thiazine ring, 1.48 for methyl H atoms, 1.16 for benzene ring H atoms, 1.17 for the H atoms bonded to the nitrogen atoms and 1.44 for the H atoms of the water molecule.



Cl16

Figure 1

The molecular structure of the title compound with thermal ellipsoids drawn at the 50% probability level.

**Figure 2**

Intermolecular hydrogen bond formation (dashed lines) in the title compound.

N-(2,6-Dimethylanilino)-5,6-dihydro-4*H*-1,3-thiazin-3-ium chloride monohydrate

Crystal data

$C_{12}H_{17}N_2S^+ \cdot Cl^- \cdot H_2O$

$M_r = 274.81$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2_1/c$

$a = 13.4546 (2) \text{ \AA}$

$b = 8.6547 (1) \text{ \AA}$

$c = 12.7732 (2) \text{ \AA}$

$\beta = 109.210 (2)^\circ$

$V = 1404.56 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 584$

$D_x = 1.300 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.5418 \text{ \AA}$

Cell parameters from 19046 reflections

$\theta = 3.5\text{--}74.6^\circ$

$\mu = 3.69 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Prism, white

$0.44 \times 0.25 \times 0.14 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur diffractometer	19046 measured reflections 2747 independent reflections
Radiation source: Enhance (Cu) X-ray Source	2509 reflections with $I > 2.0\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.029$
φ and ω scans	$\theta_{\text{max}} = 74.6^\circ$, $\theta_{\text{min}} = 3.5^\circ$
Absorption correction: numerical (de Meulenaer & Tompa, 1965)	$h = -16 \rightarrow 16$
$T_{\text{min}} = 0.30$, $T_{\text{max}} = 0.61$	$k = -10 \rightarrow 10$
	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	0 restraints
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.033$	$W = [\text{weight}][1 - (\delta F/6\sigma F)^2]^2$
$wR(F^2) = 0.088$	$(\Delta/\sigma)_{\text{max}} = 0.000294$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.43 \text{ e } \text{\AA}^{-3}$
2509 reflections	$\Delta\rho_{\text{min}} = -0.33 \text{ e } \text{\AA}^{-3}$
154 parameters	

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.72343 (3)	0.06408 (5)	0.19253 (3)	0.0234
C2	0.79732 (14)	-0.11421 (19)	0.19970 (14)	0.0244
C3	0.90751 (14)	-0.0827 (2)	0.19854 (14)	0.0252
C4	0.90283 (13)	-0.0099 (2)	0.08935 (14)	0.0244
N5	0.84522 (11)	0.13793 (17)	0.06888 (12)	0.0225
C6	0.76970 (12)	0.17936 (19)	0.10687 (13)	0.0199
N7	0.72435 (11)	0.31807 (16)	0.08222 (11)	0.0217
C8	0.65460 (13)	0.37922 (18)	0.13689 (14)	0.0210
C9	0.69975 (13)	0.4718 (2)	0.23077 (14)	0.0224
C10	0.63438 (14)	0.5346 (2)	0.28445 (15)	0.0278
C11	0.52719 (15)	0.5046 (2)	0.24466 (17)	0.0319
C12	0.48404 (14)	0.4128 (2)	0.15214 (17)	0.0300
C13	0.54694 (13)	0.3482 (2)	0.09525 (15)	0.0255
C14	0.49879 (15)	0.2481 (2)	-0.00495 (16)	0.0319
C15	0.81647 (13)	0.4980 (2)	0.27462 (14)	0.0249
Cl16	0.18401 (3)	0.10294 (5)	0.51765 (3)	0.0238
O17	0.94136 (9)	0.31268 (14)	-0.05720 (10)	0.0272
H21	0.8012	-0.1644	0.2678	0.0280*
H31	0.9450	-0.1803	0.2067	0.0276*
H32	0.9432	-0.0145	0.2573	0.0277*
H41	0.9748	0.0101	0.0908	0.0289*
H42	0.8694	-0.0804	0.0300	0.0289*
H141	0.4312	0.2885	-0.0488	0.0475*
H142	0.5426	0.2414	-0.0510	0.0467*
H143	0.4889	0.1450	0.0189	0.0475*
H151	0.8332	0.5724	0.3339	0.0357*
H152	0.8415	0.5357	0.2174	0.0356*
H153	0.8515	0.4017	0.3030	0.0359*

H171	0.9124	0.3966	-0.0570	0.0391*
H172	1.0056	0.3286	-0.0335	0.0395*
H22	0.7604	-0.1794	0.1379	0.0278*
H5	0.8667	0.2066	0.0318	0.0267*
H7	0.7466	0.3812	0.0417	0.0250*
H10	0.6635	0.5960	0.3474	0.0320*
H11	0.4838	0.5483	0.2825	0.0362*
H12	0.4111	0.3933	0.1255	0.0341*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0236 (2)	0.0216 (2)	0.0306 (2)	0.00450 (15)	0.01642 (17)	0.00271 (14)
C2	0.0292 (9)	0.0208 (8)	0.0269 (8)	0.0023 (6)	0.0143 (7)	0.0055 (6)
C3	0.0257 (8)	0.0265 (8)	0.0251 (8)	-0.0001 (7)	0.0109 (7)	0.0059 (7)
C4	0.0227 (8)	0.0262 (9)	0.0274 (8)	-0.0012 (7)	0.0127 (7)	0.0037 (6)
N5	0.0227 (7)	0.0222 (7)	0.0268 (7)	0.0015 (5)	0.0137 (5)	0.0004 (5)
C6	0.0186 (7)	0.0218 (8)	0.0203 (7)	-0.0012 (6)	0.0076 (6)	-0.0019 (6)
N7	0.0237 (7)	0.0206 (7)	0.0245 (7)	0.0018 (5)	0.0130 (6)	0.0002 (5)
C8	0.0203 (7)	0.0193 (8)	0.0258 (8)	0.0046 (6)	0.0107 (6)	0.0032 (6)
C9	0.0225 (8)	0.0204 (8)	0.0258 (8)	0.0035 (6)	0.0103 (7)	0.0035 (6)
C10	0.0297 (9)	0.0274 (8)	0.0296 (9)	-0.0003 (7)	0.0140 (7)	0.0043 (7)
C11	0.0274 (9)	0.0319 (10)	0.0432 (10)	0.0024 (8)	0.0209 (8)	0.0065 (7)
C12	0.0181 (8)	0.0290 (9)	0.0442 (11)	0.0072 (8)	0.0121 (7)	0.0020 (7)
C13	0.0221 (8)	0.0218 (8)	0.0319 (9)	0.0052 (7)	0.0078 (7)	0.0008 (6)
C14	0.0264 (8)	0.0263 (9)	0.0382 (10)	0.0001 (8)	0.0042 (7)	-0.0029 (7)
C15	0.0224 (8)	0.0254 (9)	0.0259 (8)	0.0012 (7)	0.0065 (7)	0.0007 (6)
Cl16	0.0232 (2)	0.0239 (2)	0.0266 (2)	0.00033 (14)	0.01150 (16)	0.00127 (14)
O17	0.0244 (6)	0.0248 (6)	0.0351 (7)	-0.0025 (5)	0.0135 (5)	-0.0028 (5)

Geometric parameters (Å, °)

S1—C2	1.8215 (17)	C9—C10	1.391 (2)
S1—C6	1.7403 (16)	C9—C15	1.501 (2)
C2—C3	1.512 (2)	C10—C11	1.387 (3)
C2—H21	0.959	C10—H10	0.936
C2—H22	0.964	C11—C12	1.383 (3)
C3—C4	1.513 (2)	C11—H11	0.950
C3—H31	0.971	C12—C13	1.401 (3)
C3—H32	0.952	C12—H12	0.942
C4—N5	1.474 (2)	C13—C14	1.504 (3)
C4—H41	0.977	C14—H141	0.964
C4—H42	0.961	C14—H142	0.961
N5—C6	1.312 (2)	C14—H143	0.966
N5—H5	0.866	C15—H151	0.963
C6—N7	1.336 (2)	C15—H152	0.957
N7—C8	1.442 (2)	C15—H153	0.967
N7—H7	0.870	O17—H171	0.825

C8—C9	1.404 (2)	O17—H172	0.829
C8—C13	1.395 (2)		
C2—S1—C6	102.42 (8)	C9—C8—C13	122.61 (15)
S1—C2—C3	111.57 (12)	C8—C9—C10	118.55 (16)
S1—C2—H21	107.2	C8—C9—C15	120.71 (15)
C3—C2—H21	109.3	C10—C9—C15	120.71 (16)
S1—C2—H22	109.2	C9—C10—C11	119.76 (17)
C3—C2—H22	109.9	C9—C10—H10	119.4
H21—C2—H22	109.7	C11—C10—H10	120.8
C2—C3—C4	109.91 (14)	C10—C11—C12	120.93 (16)
C2—C3—H31	108.6	C10—C11—H11	118.6
C4—C3—H31	108.9	C12—C11—H11	120.5
C2—C3—H32	110.3	C11—C12—C13	121.14 (16)
C4—C3—H32	109.0	C11—C12—H12	120.4
H31—C3—H32	110.2	C13—C12—H12	118.5
C3—C4—N5	112.65 (13)	C12—C13—C8	117.01 (16)
C3—C4—H41	108.4	C12—C13—C14	120.45 (16)
N5—C4—H41	108.0	C8—C13—C14	122.53 (16)
C3—C4—H42	109.2	C13—C14—H141	110.2
N5—C4—H42	109.1	C13—C14—H142	112.1
H41—C4—H42	109.3	H141—C14—H142	108.5
C4—N5—C6	126.70 (14)	C13—C14—H143	109.2
C4—N5—H5	116.2	H141—C14—H143	108.5
C6—N5—H5	116.9	H142—C14—H143	108.3
S1—C6—N5	123.83 (13)	C9—C15—H151	109.9
S1—C6—N7	115.66 (12)	C9—C15—H152	110.8
N5—C6—N7	120.50 (15)	H151—C15—H152	108.8
C6—N7—C8	122.35 (13)	C9—C15—H153	109.3
C6—N7—H7	118.9	H151—C15—H153	108.8
C8—N7—H7	117.6	H152—C15—H153	109.3
N7—C8—C9	117.10 (14)	H171—O17—H172	106.9
N7—C8—C13	120.28 (15)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N5—H5 \cdots O17	0.87	1.97	2.815 (2)	163
O17—H171 \cdots C116 ⁱ	0.82	2.36	3.158 (1)	164
N7—H7 \cdots C116 ⁱ	0.87	2.37	3.204 (1)	162
O17—H172 \cdots C116 ⁱⁱ	0.83	2.35	3.171 (1)	173

Symmetry codes: (i) $-x+1, y+1/2, -z+1/2$; (ii) $x+1, -y+1/2, z-1/2$.