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N-Amino-3-methyl-2,3-dihydro-1,3-benzothiazoliminium chloride monohydrate

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Key indicators

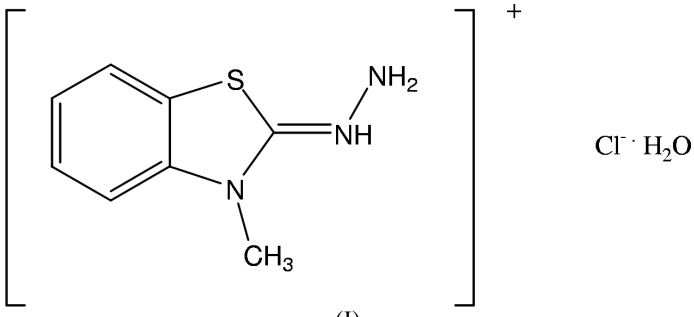
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
Disorder in solvent or counterion
R factor = 0.023
wR factor = 0.064
Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_8H_{10}N_3S^+\cdot Cl^-\cdot H_2O$, is extensively used as a spectrophotometric reagent for the determination of pharmaceutical compounds, vitamins and environmental samples.

Comment

The title compound, (I), is extensively used as a spectrophotometric reagent for the determination of pharmaceutical compounds (Sastry *et al.*, 1990), vitamins (Nagaraja *et al.*, 2002) and environmental samples (Nagaraja *et al.*, 2003). In view of the importance of this reagent, its crystal structure determination is reported.



(I)

A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.6 plus three updates; *MOGUL* Version 1.0; Allen, 2002). The ring system is planar (r.m.s. deviation = 0.010 Å).

Experimental

The title compound was purchased from the Aldrich Chemical Company and used without further purification. Recrystallization from water gave dark brown prismatic crystals after slow evaporation of the solvent.

Crystal data

$C_8H_{10}N_3S^+\cdot Cl^-\cdot H_2O$
M_r = 233.72
Monoclinic, $C2/c$
a = 21.0397 (19) Å
b = 11.6598 (12) Å
c = 8.6694 (8) Å
β = 92.935 (7)°
V = 2124.0 (4) Å^3
Z = 8
D_x = 1.462 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 18 301 reflections
θ = 3.8–26.1°
μ = 0.53 mm^{-1}
T = 173 (2) K
Prism, brown
0.37 × 0.36 × 0.21 mm

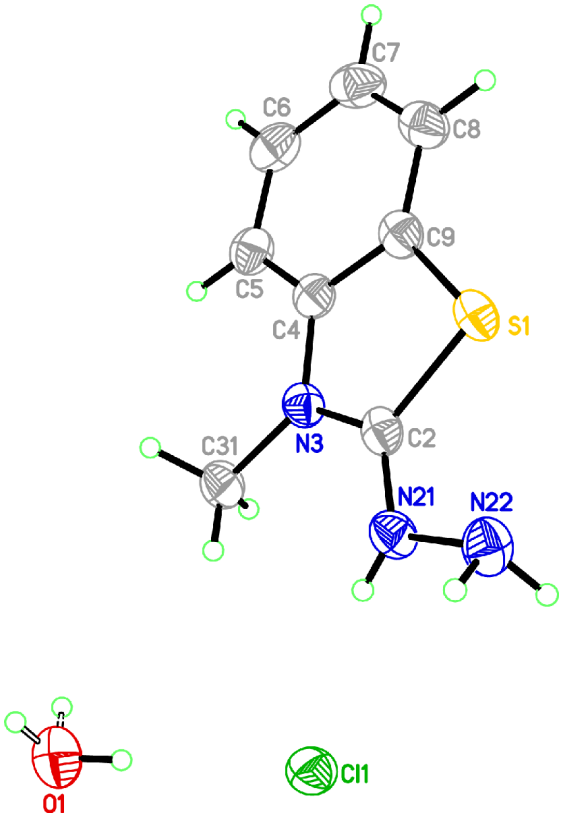


Figure 1
Perspective view of the title compound, with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. The intramolecular hydrogen bond is shown as a dashed line.

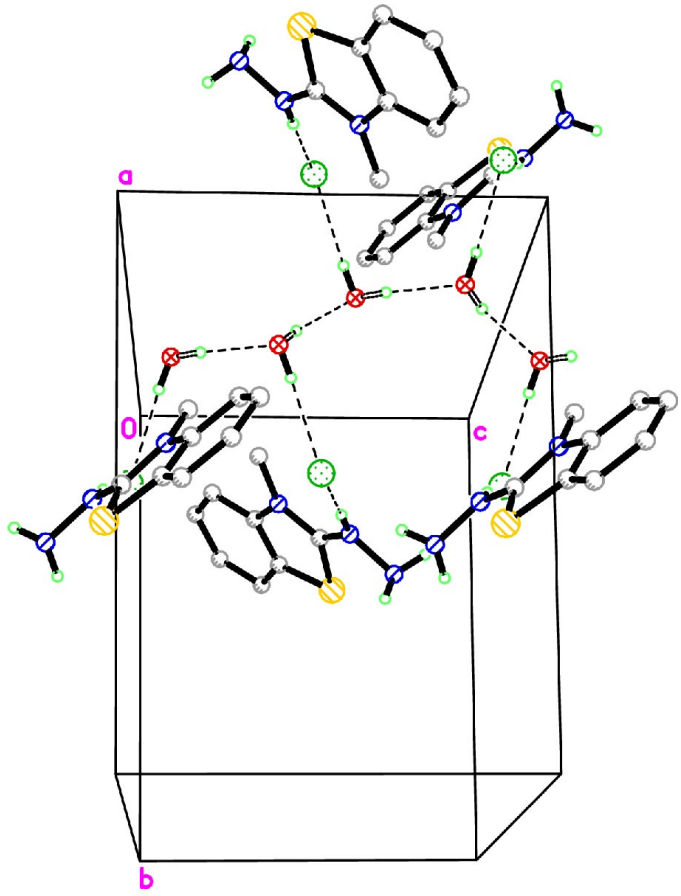


Figure 2
Packing diagram of (I), viewed on to the *bc* plane. Hydrogen bonds are drawn as dashed lines. The O—H bonds of the fully occupied H atoms are drawn with a solid line, the disordered H atoms have open or dashed open bonds to clarify the hydrogen-bond pattern. CH H atoms have been omitted for clarity and on each water molecule only one of the disordered H atoms is shown.

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.829, T_{max} = 0.897
14 856 measured reflections
2024 independent reflections
1775 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 25.7°
$h = -25 \rightarrow 25$
$k = -14 \rightarrow 14$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.064
S = 1.05
2024 reflections
152 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C2	1.7276 (13)	N3—C4	1.3987 (17)
S1—C9	1.7487 (13)	N3—C31	1.4619 (17)
C2—N21	1.3039 (17)	C4—C9	1.3876 (18)
C2—N3	1.3393 (16)	N21—N22	1.4142 (15)
C2—S1—C9	89.62 (6)	C2—N21—N22	116.25 (12)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N21—H21···Cl1	0.77 (2)	2.30 (2)	3.0586 (13)	168 (2)
N22—H22A···Cl1^{i}	0.902 (18)	2.443 (18)	3.3323 (13)	168.9 (15)
N22—H22B···Cl1^{ii}	0.90 (2)	2.43 (2)	3.2583 (14)	152.7 (14)
O1—H1A···Cl1	0.83 (3)	2.33 (3)	3.1583 (15)	177.0 (19)
O1—H1B···O1^{iii}	0.79 (5)	1.99 (5)	2.767 (3)	166 (5)
O1—H1B'···O1^{iv}	0.84 (4)	1.95 (4)	2.772 (3)	168 (4)

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

H atoms were located in a difference map. Those bonded to carbon were positioned geometrically and refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5 U_{eq}(C_{methyl})] using a riding model, with C—H = 0.98 and 0.95 Å for methyl CH and aromatic CH groups, respectively. In addition, the methyl group was allowed to rotate but not to tip. H atoms bonded to nitrogen and oxygen were refined isotropically. One H atom of the water molecule is disordered over two equally occupied positions.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine

structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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