Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,6-Diammonio-4-toluenesulfonate chloride dihydrate

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Received 25 January 2005
Accepted 3 February 2005
Online 12 February 2005

Key indicators

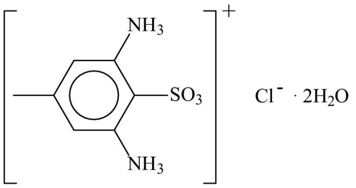
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.039
wR factor = 0.130
Data-to-parameter ratio = 11.1

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

The cation, anion and water molecules of the title compound, $C_7H_{11}N_2O_3S^+\cdot Cl^-\cdot 2H_2O$ are linked by hydrogen bonds into a three-dimensional network. The cation and anion lie on mirror planes, and both are disordered.

Comment

6-Amino-2-ammonio-4-toluenesulfonate exists as a zwitterion having an amino as well as an ammonium substituent on the aromatic ring (Huo *et al.*, 2005). The amino group should be capable of reacting with mineral acids to yield salts, and this is borne out with the hydrogen chloride adduct, which was obtained, albeit serendipitously. 2,6-Diammonio-4-toluenesulfonate crystallizes as a dihydrate, (I) (Fig. 1).



(I)

The cation, anion and water molecules in (I) are hydrogen bonded into a three-dimensional network (Table 1). Bond dimensions are generally similar to those of the parent zwitterion. The two C—N bonds in (I) are crystallographically required to be identical and are 1.459 (2) Å long. This distance exceeds the C—N$_{\text{amino}}$ bond length of 1.372 (3) Å, but is shorter than the C—N$_{\text{ammonio}}$ bond of 1.473 (3) Å in the parent zwitterion (Huo *et al.*, 2005).

Experimental

The ammonium salt was obtained unexpectedly from the reaction of barium chloride dihydrate (1.22 g, 5 mmol) and 3,5-diamino-4-methylbenzenesulfonic acid (1.01 g, 5 mmol). The reagents were dissolved in water; pale-pink crystals separated from solution after a few days. Analysis calculated for $C_7H_9ClN_2O_5S$: C 31.29, H 3.38, N 10.43%; found: C 31.33, H 3.41, N 10.47%.

Crystal data

$C_7H_{11}N_2O_3S^+\cdot Cl^-\cdot 2H_2O$
M_r = 274.72
Orthorhombic, *Pnma*
a = 9.196 (2) Å
b = 9.407 (2) Å
c = 13.322 (3) Å
V = 1152.4 (4) Å^3
Z = 4
D_x = 1.583 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9848 reflections
θ = 3.1–27.5°
μ = 0.52 mm^{-1}
T = 295 (2) K
Block, pale pink
0.36 × 0.26 × 0.20 mm

Data collection

Rigaki R-AXIS RAPID IP diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.798$, $T_{\max} = 0.903$
10743 measured reflections
1393 independent reflections
1286 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.017$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.130$
$S = 1.11$
1393 reflections
125 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0844P)^2 + 0.4374P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1n3···O1$^{\rm i}$	0.86 (1)	2.45 (3)	2.780 (2)	103 (2)
N1—H1n1···O2$^{\rm ii}$	0.85 (1)	2.37 (2)	2.827 (3)	114 (2)
N1—H1n2···O1$w^{\rm iii}$	0.85 (1)	2.25 (2)	2.952 (2)	140 (2)
N1—H1n3···O1w	0.86 (1)	2.00 (1)	2.845 (2)	169 (3)
N1—H1n1···O1$'^{\rm ii}$	0.85 (1)	2.18 (1)	2.989 (5)	160 (2)
N1—H1n2···O2$'^{\rm i}$	0.85 (1)	2.14 (2)	2.71 (1)	125 (2)
O1w—H1w1···Cl1	0.85 (1)	2.36 (1)	3.207 (4)	179 (3)
O1w—H1w2···Cl1$^{\rm iv}$	0.85 (1)	2.54 (2)	3.291 (5)	148 (2)
O1w—H1w1···Cl1$'$	0.85 (1)	2.26 (1)	3.094 (9)	168 (3)
O1w—H1w2···Cl1$'^{\rm iv}$	0.85 (1)	2.35 (2)	3.158 (5)	158 (2)

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

The structure is disordered in the chloride ion and in the sulfonate group, both of which lie on mirror planes. The C—S1 and C1—S1$'$ distances were restrained to within 0.01 Å of each other, as were the S1—O1, S1—O2, S1$'$—O1$'$ and S1$'$—O2$'$ distances. The disordered O atoms were restrained to behave in an isotropic manner. The methyl group, which also lies on a mirror plane, is rotationally disordered between two orientations. The ammonium and water H atoms were located in a difference Fourier map and refined with distance restraints of N—H = O—H = 0.85 (1) Å and H···H = 1.39 (1) Å. The carbon-bound H atoms were placed at calculated positions [C—H = 0.95 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for the aromatic H atoms and C—H = 0.96 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for the methyl H atoms] and were included in the refinement in the riding-model approximation.

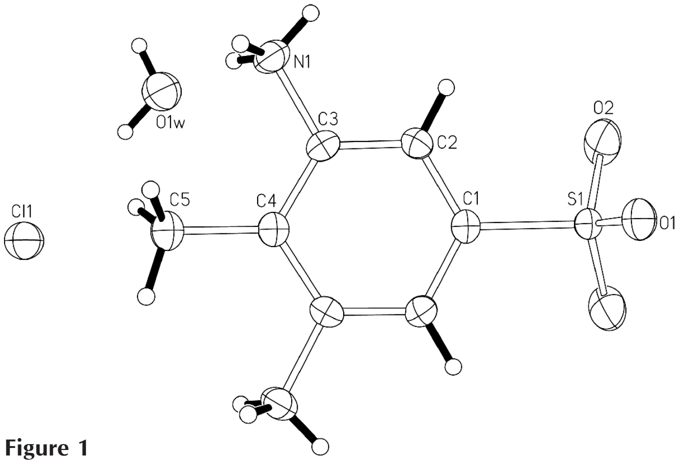


Figure 1
ORTEPII (Johnson, 1976) plot of (I), showing displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. The Cl and SO_3 groups are disordered and only the major components are shown. Unlabeled atoms are related to labeled atoms by $(x, \frac{3}{2} - y, z)$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

We thank the National Natural Science Foundation of China (No. 20101003), the Scientific Fund for Remarkable Teachers of Heilongjiang Province (No. 1054G036) and the University of Malaya for supporting this study.

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