

2-Sulfoethylammonium hexafluoroantimonate

Mathias Hopfinger, Karin Lux, Felix Schubert and Andreas Kornath*

Department of Chemistry, Ludwig-Maximilian University, Butenandtstrasse 5–13 (Haus D), D-81377 Munich, Germany
Correspondence e-mail: andreas.kornath@cup.uni-muenchen.de

Received 5 October 2011

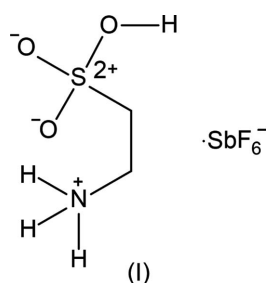
Accepted 9 November 2011

Online 30 November 2011

The title salt, $(\text{C}_2\text{H}_8\text{NO}_3\text{S})[\text{SbF}_6]$, which contains the protonated form of taurine (2-aminoethanesulfonic acid), was synthesized in anhydrous hydrofluoric acid and recrystallized as colourless block-shaped crystals from liquid SO_2 . In the solid state, a three-dimensional network is observed. This is formed by intra- and intermolecular $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{F}$ and $\text{O}-\text{H}\cdots\text{F}$ hydrogen bonds.

Comment

Taurine was first isolated from the bile of oxen by the chemists Tiedemann & Gmelin (1827). The crystal structure, with incorrect refinement of the H atoms of taurine, was first described by Sutherland & Young (1963). Three years later, Okaya (1966) showed that taurine is present as a zwitterion. Taurine plays an important role in human metabolism (Tao & Harris, 2004), but until now pure protonated taurine has not been observed in the condensed phase. We present here the first crystal structure of protonated taurine as the hexafluoroantimonate (SbF_6^-) salt, namely 2-sulfoethylammonium hexafluoroantimonate, (I). The salt crystallizes in the orthorhombic space group $Pbca$ with one molecule in the asymmetric unit. The molecular conformation of (I) is shown in Fig. 1.



The $\text{Sb}-\text{F}$ bond lengths in the SbF_6^- anion are in the range 1.8633 (16)–1.8977 (15) Å. The $\text{Sb}-\text{F}$ bonds which are involved in hydrogen bonding ($\text{Sb1}-\text{F3}$, $\text{Sb1}-\text{F4}$ and $\text{Sb1}-\text{F5}$) are slightly longer than $\text{Sb1}-\text{F1}$, $\text{Sb1}-\text{F2}$ and $\text{Sb1}-\text{F6}$.

These values are in the typical range for an SbF_6^- anion, as observed by Minkwitz *et al.* (1992) for $[(\text{C}_6\text{F}_5)_2\text{SF}]^+\cdot\text{SbF}_6^-$. The SbF_6^- anion forms a slightly distorted octahedron. The $\text{S1}-\text{O1}$ and $\text{S1}-\text{O2}$ bond lengths [1.4370 (18) and 1.4275 (19) Å, respectively] are in the typical range between an $\text{S}-\text{O}$ single and double bond, and are comparable with those in neutral taurine. The $\text{S1}-\text{O3}$ bond [1.548 (2) Å] is about 0.11 Å longer and is therefore closer in length to an $\text{S}-\text{O}$ single bond. The other bond lengths are comparable with the neutral compound and show expected values. The S atom is coordinated in a distorted tetrahedral manner [bond angles about the S atom of 104.94 (12)–119.77 (11)°]. Comparable with the neutral compound, the NH_3 group shows a *gauche* configuration [$\text{S1}-\text{C1}-\text{C2}-\text{N1} = -73.9 (3)^\circ$] with respect to the SO_3H group.

In the crystal structure of (I), the ions are held together by a three-dimensional network of moderate $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{F}$ and $\text{O}-\text{H}\cdots\text{F}$ hydrogen bonds (Fig. 2). The bond lengths and angles of the hydrogen bonds are listed in Table 1. In the cation, one intramolecular $\text{N1}-\text{H1A}\cdots\text{O1}$ hydrogen bond [2.906 (3) Å] can be observed, with graph set $S(6)$ (Bernstein *et al.*, 1995). This bond is also present in the neutral compound. The cations form chains involving the $\text{N1}-\text{H1B}\cdots\text{O2}^i$ hydrogen bond [graph set $C(6)$] along the b axis [symmetry code: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, z$], with parallel chains of SbF_6^- octahedra. Along the a axis, the cations form zigzag chains through the $\text{N1}-\text{H1C}\cdots\text{O1}^{iii}$ hydrogen bond [graph set $C(6)$; symmetry code: (iii) $x + \frac{1}{2}, y, -z + \frac{3}{2}$]. The SbF_6^- octahedra are also connected to the cations by hydrogen bonds involving atoms F2, F3, F4 and F5. The F3 and F4 acceptors of one SbF_6^- octahedron are both linked to the N1 donor atom of the same cation [graph set $R_2^2(6)$] and build a six-membered ring. The strengths of these two hydrogen bonds [$\text{N}\cdots\text{F} = 3.060 (3)$ and $2.917 (3)$ Å, respectively] are only moderate. The weakest hydrogen bond is $\text{N1}-\text{H1C}\cdots\text{F2}$ [$\text{N}\cdots\text{F} = 3.006 (3)$ Å] which explains the shorter $\text{Sb1}-\text{F1}$ distance. The strongest hydrogen bond is, as expected, $\text{O3}-$

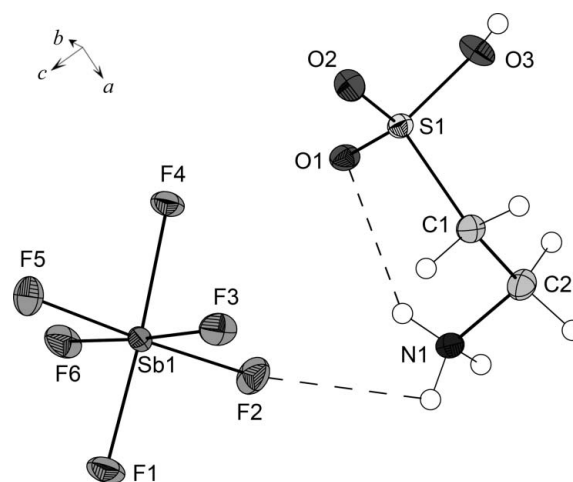


Figure 1
The independent unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

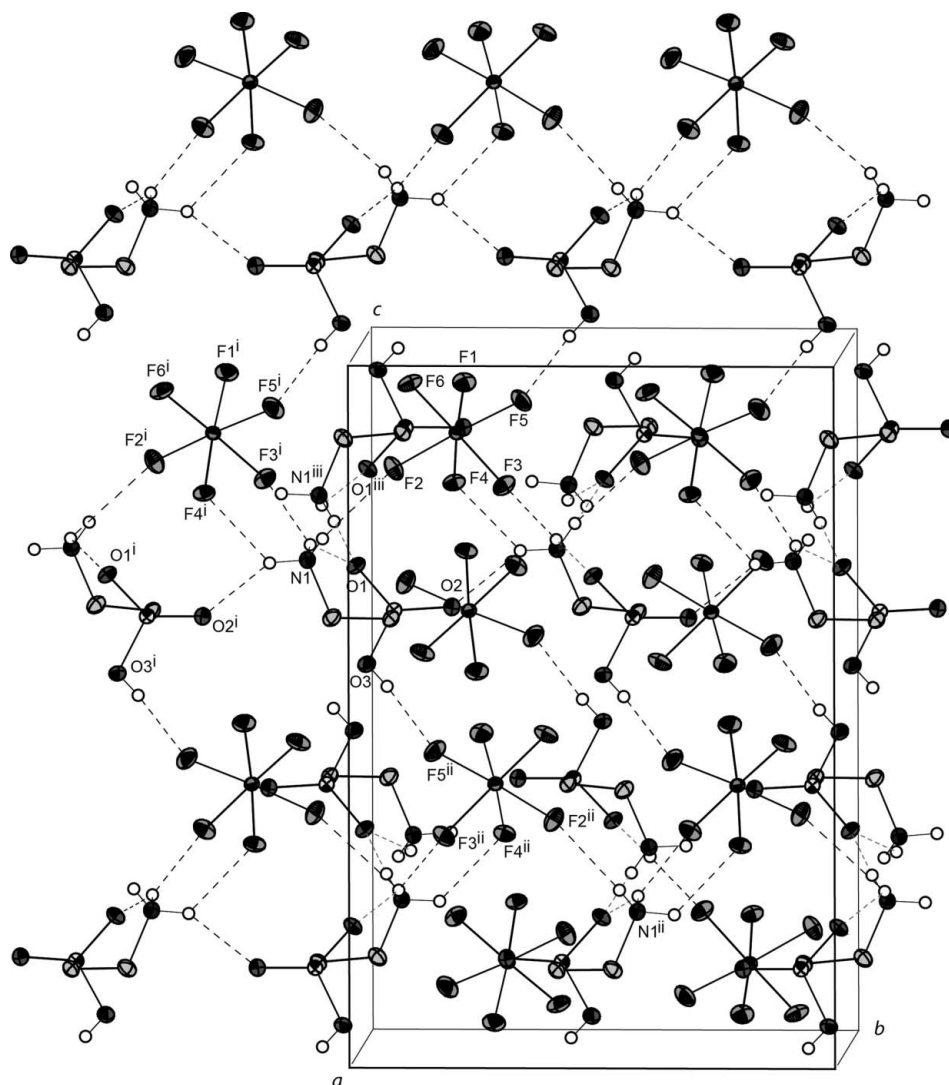


Figure 2

A view of the unit cell of (I) along the *a* axis, showing the three-dimensional network of the packing. Hydrogen bonds are shown as dashed lines. H atoms on C atoms have been omitted for clarity. [Symmetry codes: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, z$; (ii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $x + \frac{1}{2}, y, -z + \frac{3}{2}$.]

$\text{H3} \cdots \text{F5}^{\text{ii}}$ [$\text{O} \cdots \text{F} = 2.623(3) \text{ \AA}$; symmetry code: (ii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$]. In comparison, in $[\text{H}_3\text{SO}_4]^+ \cdot \text{SbF}_6^-$ (Minkwitz *et al.*, 2002), the $\text{O} \cdots \text{F}$ contacts are about 0.04–0.09 \AA shorter [$\text{O} \cdots \text{F} = 2.58(1)$ and $2.53(1) \text{ \AA}$].

Experimental

Caution! Avoid contact with the title compound and SbF_5 . Note that hydrolysis of SbF_6^- salts might form HF, which burns the skin and causes irreparable damage. Safety precautions, such as the use of special gloves and a face visor, should be taken when handling these materials.

All synthetic work and sample handling were performed by employing standard Schlenk techniques. Superacid reactions were carried out in FEP/PFA ampoules, which were closed with stainless steel valves and connected to a stainless steel vacuum line. All reaction vessels and the stainless steel line were dried with fluorine prior to use.

Into 2-aminoethanesulfonic acid (1 mmol) in an FEP reactor, an excess of hydrogen fluoride (100 mmol) was distilled at 77 K,

followed by SbF_5 (1 mmol). The mixture was warmed to 218 K for 5 min. Excess hydrogen fluoride was removed in a dynamic vacuum at 195 K. A colourless powder was obtained, which was then recrystallized from liquid SO_2 . Colourless crystals of (I) were obtained, which were suitable for X-ray diffraction studies. The crystals are stable under inert gas up to 258 K.

Crystal data

$(\text{C}_2\text{H}_8\text{NO}_3\text{S})[\text{SbF}_6]$
 $M_r = 361.90$
 Orthorhombic, *Pbca*
 $a = 9.9037(4) \text{ \AA}$
 $b = 11.5575(4) \text{ \AA}$
 $c = 16.7270(6) \text{ \AA}$

$V = 1914.61(12) \text{ \AA}^3$
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 3.18 \text{ mm}^{-1}$
 $T = 100 \text{ K}$
 $0.20 \times 0.18 \times 0.12 \text{ mm}$

Data collection

Agilent Xcalibur Sapphire3
 diffractometer
 Absorption correction: multi-scan
 (*CrysAlis PRO*; Agilent, 2011)
 $T_{\text{min}} = 0.569, T_{\text{max}} = 0.702$

9108 measured reflections
 1870 independent reflections
 1543 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1A...O1	0.81 (4)	2.29 (4)	2.906 (3)	134 (3)
N1—H1A...F3 ⁱ	0.81 (4)	2.38 (4)	3.060 (3)	143 (3)
N1—H1B...O2 ⁱ	0.91 (3)	2.12 (3)	2.860 (3)	138 (3)
N1—H1B...F4 ⁱ	0.91 (3)	2.27 (3)	2.917 (3)	127 (2)
O3—H3...F5 ⁱⁱ	0.71 (3)	1.92 (3)	2.623 (3)	173 (3)
N1—H1C...O1 ⁱⁱⁱ	0.89 (3)	2.00 (4)	2.868 (3)	163 (3)
N1—H1C...F2	0.89 (3)	2.57 (3)	3.006 (3)	111 (2)

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

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.018$	159 parameters
$wR(F^2) = 0.043$	All H-atom parameters refined
$S = 0.91$	$\Delta\rho_{\max} = 0.59 \text{ e } \text{Å}^{-3}$
1870 reflections	$\Delta\rho_{\min} = -0.64 \text{ e } \text{Å}^{-3}$

All H atoms were located in a difference Fourier map, included in their observed positions and refined freely, including their isotropic displacement parameters. The range of refined C—H distances is 0.92 (3)–0.98 (3) Å.

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008), with atomic masses from Coplen (1996); molecular graphics: *DIAMOND* (Brandenburg, 2009); software used to prepare material for publication: *PLATON*

(Spek, 2009), *publCIF* (Westrip, 2010) and *WinGX* (Farrugia, 1999), and with atomic masses from Coplen (1996).

This work was supported by the University of Munich. The X-ray data were collected at the Department of Chemistry.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OV3009). Services for accessing these data are described at the back of the journal.

References

- Agilent (2011). *CrysAlis PRO*. Version 1.171.35.11 (release 16-05-2011 CrysAlis 171.NET). Agilent Technologies, Yarnton, Oxfordshire, England.
- Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Brandenburg, K. (2009). *DIAMOND*. Version 2.1c. Crystal Impact GbR, Bonn, Germany.
- Coplen, T. B. (1996). *Pure Appl. Chem.* **68**, 2339–2359.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Minkwitz, R., Nowicki, G. & Preut, H. (1992). *Z. Anorg. Allg. Chem.* **611**, 23–27.
- Minkwitz, R., Seelbinder, R. & Schöbel, R. (2002). *Angew. Chem. Int. Ed.* **41**, 111–114.
- Okaya, Y. (1966). *Acta Cryst.* **21**, 726–735.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Sutherland, H. H. & Young, D. W. (1963). *Acta Cryst.* **16**, 897–901.
- Tao, L. & Harris, A. L. (2004). *J. Biol. Chem.* **279**, 38544–38554.
- Tiedemann, F. & Gmelin, L. (1827). *Ann. Phys.* **85**, 326–337.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.