

Sodium trifluoromethanesulfonate acetonitrile
solvateA. Meffre,‡ M. Barboiu and
A. van der Lee*Institut Européen des Membranes, Université de
Montpellier II, cc 047 Montpellier, France‡ Present address: Merck-Estapor, rue du
Moulin de la Canne, 45300 Pithiviers, FranceCorrespondence e-mail:
avderlee@univ-montp2.fr

Key indicators

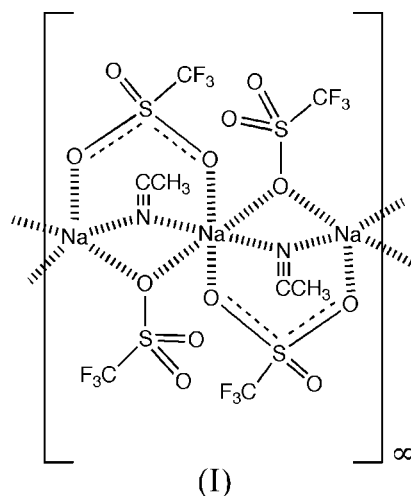
Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.035
 wR factor = 0.084
Data-to-parameter ratio = 14.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the structure of the title salt, $\text{Na}^+\cdot\text{CF}_3\text{SO}_3^-\cdot\text{C}_2\text{H}_3\text{N}$, two symmetry-related acetonitrile molecules take part in the octahedral coordination of the Na^+ cation, which is located on an inversion center. The trifluoromethanesulfonate anions and acetonitrile molecules lie on mirror planes. The Na^+ ions are arranged in layers, sandwiched by trifluoromethanesulfonate double layers with the lipophilic CF_3 groups pointing outwards and towards those of neighbouring double layers.

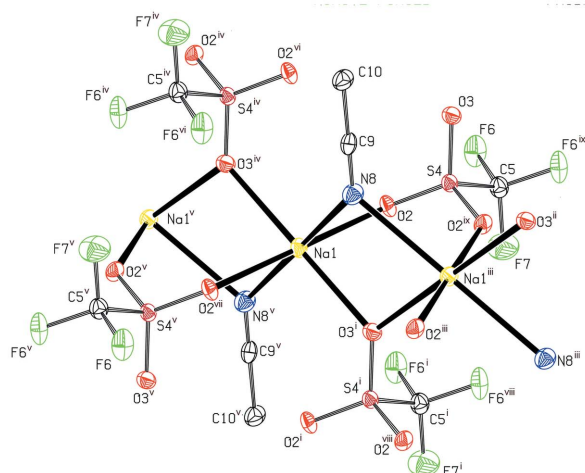
Received 5 December 2006
Accepted 12 December 2006

Comment

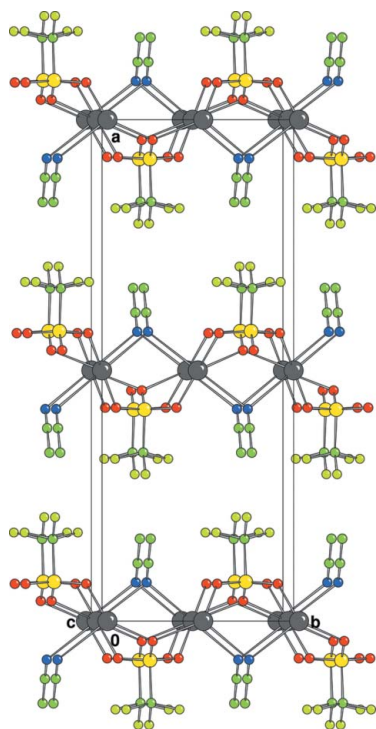
In an ongoing study on novel functional membrane materials using heteroditopic ureido crown ethers capable of complexing both cations and anions (Barboiu *et al.*, 2004, Cazacu *et al.*, 2006), we accidentally crystallized a sodium trifluoromethanesulfonate salt as an acetonitrile monosolvate. The anhydrous trifluoromethanesulfonate salts of a number of metals A have been determined recently using high-resolution powder diffraction for $A = \text{Mg}, \text{Ca}, \text{Ba}, \text{Zn}, \text{Cu}$ (Dinnebier *et al.* 2006) and using single-crystal data for $A = \text{Na}$ (Sofina *et al.*, 2003) and $A = \text{K}$ (Korus & Jansen, 2001).



The sodium ion, located on an inversion centre, is octahedrally coordinated by four O atoms of four different trifluoromethanesulfonate anions in a square planar environment and by two N atoms belonging to different acetonitrile molecules (Fig. 1 and Table 1). This is in contrast with the simple anhydrous metal trifluoromethanesulfonate salts, in which the alkali metal is mostly octahedrally coordinated by six O atoms belonging to trifluoromethanesulfonate anions. The mean $\text{Na}-\text{O}$ distance is $2.394(1)$ Å, which is somewhat shorter than the mean value of 2.504 Å found from a survey of

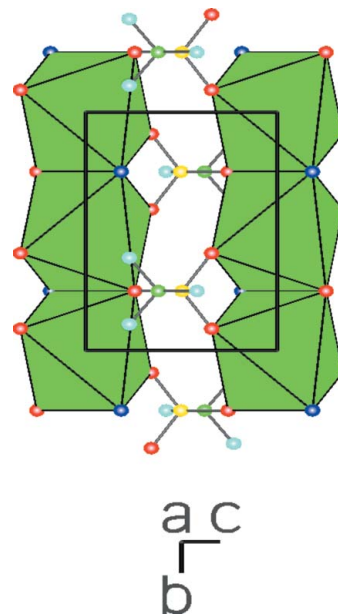

Figure 1

Part of the polymeric structure with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry codes: (i) $x, y, z + 1$; (ii) $-x, y - \frac{1}{2}, -z$; (iii) $-x, y - \frac{1}{2}, z + 1$; (iv) $-x, y + \frac{1}{2}, -z$; (v) $-x, y + \frac{1}{2}, 1 - z$; (vi) $-x, 1 - y, -z$; (vii) $-x, 1 - y, 1 - z$; (viii) $x, \frac{1}{2} - y, z + 1$; (ix) $x, \frac{1}{2} - y, z$]


Figure 2

The crystal packing of (I), showing the double layers of trifluoromethane anions.

1054 entries in the Cambridge Structural Database (CSD; Version 5.27, Allen, 2002) having Na in a square-planar environment coordinated by O. The Na–N distance, 2.627 (2) Å, is considerably longer than the mean value of 2.452 Å found from a survey of 22 entries in the CSD having close Na–acetonitrile contacts, but significantly shorter than the sum of the van der Waals radii of Na and N (1.70 and 1.55 Å, respectively; Bondi, 1964). The anions and acetonitrile molecules lie on mirror planes.


Figure 3

Arrangement of NaO_4N_2 octahedra within a double $\text{Na}(\text{CF}_3\text{SO}_3)_2$ layer. The carbons of the acetonitrile molecules are not drawn for clarity.

The crystal packing (Fig. 2) is reminiscent of that found for the simple anhydrous alkali trifluoromethanesulfonate salts, *i.e.* there are double layers of trifluoromethanesulfonate anions that sandwich the Na cations. The lipophilic CF_3 groups in adjacent single trifluoromethanesulfonate layers point towards each other and are in a staggered conformation with respect to each other. Fig. 3 shows the arrangement of the NaO_4N_2 octahedra in one double layer of $\text{Na}(\text{CF}_3\text{SO}_3)_2$; the acetonitrile carbons are not drawn for clarity. The octahedra share one edge and are arranged in rows along the b axis. The SO_3 group of each trifluoromethanesulfonate anion is linked by its O atoms to three different octahedra.

Experimental

1,1 Equivalents of 2-isocynoethyl methacrylate (240 mg; 1.55 mmol) was added to a solution of 4'-aminobenzo-15-crown-5 (400 mg; 1.41 mmol) in acetonitrile (20 ml) at room temperature. The mixture was refluxed for one night. The solvent was removed in vacuum and the residue was washed with 10 ml hexane to give a white solid, 4-methacrylate ethylurea-benzo-15-crown-5 (1). A solution of (1) (15 mg, 0.034 mmol) in 2 ml acetonitrile and 2 equivalents of solid sodium trifluoromethanesulfonate (11.75 mg, 0.068 mmol) was ultrasonicated at room temperature, then filtered by gravity. Crystallization was achieved by adding isopropyl ether, which resulted in the formation of colourless single crystals of (I).

Crystal data

$\text{Na}^+ \cdot \text{CF}_3\text{SO}_3^- \cdot \text{C}_2\text{H}_3\text{N}$
 $M_r = 213.12$
 Orthorhombic, $Pnma$
 $a = 20.0447$ (7) Å
 $b = 7.6833$ (2) Å
 $c = 4.91966$ (17) Å
 $V = 757.67$ (4) Å³

$Z = 4$
 $D_x = 1.868$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.51$ mm⁻¹
 $T = 173$ K
 Needle, colourless
 $0.45 \times 0.08 \times 0.04$ mm

Data collection

Oxford Diffraction GEMINI diffractometer
 φ and ω scans
 Absorption correction: analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1995).

(*CrysAlis RED*; Oxford Diffraction, 2006)
 $T_{\min} = 0.841$, $T_{\max} = 0.977$
 29572 measured reflections
 1016 independent reflections
 523 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.090$
 $\theta_{\max} = 28.8^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.084$
 $S = 0.71$
 1016 reflections
 68 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F^2) + (0.05P)^2]$,
 where $P = [\max(F_o^2, 0) + 2F_c^2]/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.85 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.75 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Na1—O2 ⁱ	2.3558 (14)	Na1—N8 ⁱ	2.627 (2)
Na1—O3 ⁱⁱ	2.4315 (14)	Na1—O2	2.3558 (14)
Na1—O3 ⁱⁱⁱ	2.4315 (14)	Na1—N8	2.627 (2)
O2 ⁱ —Na1—O3 ⁱⁱ	94.37 (6)	O3 ⁱⁱⁱ —Na1—O2	94.37 (6)
O2 ⁱ —Na1—O3 ⁱⁱⁱ	85.63 (6)	N8 ⁱ —Na1—O2	95.36 (7)
O3 ⁱⁱ —Na1—O3 ⁱⁱⁱ	179.995	O2 ⁱ —Na1—N8	95.36 (7)
O2 ⁱ —Na1—N8 ⁱ	84.64 (7)	O3 ⁱⁱⁱ —Na1—N8	78.16 (7)
O3 ⁱⁱ —Na1—N8 ⁱ	101.84 (7)	O3 ⁱⁱⁱ —Na1—N8	101.84 (7)
O3 ⁱⁱⁱ —Na1—N8 ⁱ	78.16 (7)	N8 ⁱ —Na1—N8	179.996
O2 ⁱ —Na1—O2	179.995	O2—Na1—N8	84.64 (7)
O3 ⁱⁱ —Na1—O2	85.63 (6)	Na1—O2—S4	131.82 (9)

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

The two independent H atoms were located in a difference map and allowed to ride on their parent atom with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{parent atom})$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2006); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *PLATON* (Spek, 2003), *CAMERON* (Watkin *et al.*, 1996) and *DRAWxtl* (Kroeker & Finger, 2005); software used to prepare material for publication: *CRYSTALS*.

This research was supported in part by the European Science Foundation (ESF), and the European Heads of Research Councils (EUROHORCs) EURYI Award 2004 (MB).

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