organic compounds

Polysulfonylamines. CXCI. The 'almost' polymorphs rac-trans-2-aminocyclohexan-1-aminium di(methanesulfonyl)azanide and its 0.11-hydrate[1]

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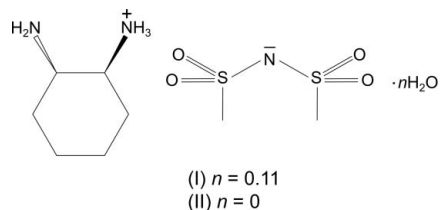
The title compound, $C_6H_{15}N_2^+ \cdot C_2H_6NO_4S_2^-$, crystallizes as a 0.11-hydrate, (I), in the space group $C2$; the asymmetric unit consists of two cations (one of each enantiomer), one anion on a general position, two half anions, each with the N atom on a twofold axis, and approximately one fifth of a water molecule. The general anion departs significantly from the usual conformation: it lacks one of the typical 'W'-shaped sequence of O—S—N—S—O atoms. The compound also crystallizes in the solvent-free form, (II), in the space group $P2_1/c$, with one formula unit in the asymmetric unit. Both compounds form ribbons of hydrogen-bonded cation dimers parallel to the b axis. In (I), there are two independent ribbons of opposite chirality, each involving one anion on a special position, and these ribbons are connected by hydrogen bonds to the anion on a general position, resulting in a layer structure parallel to (100). In (II), the chains are connected by hydrogen bonds, and again a layer structure parallel to (100) results.

Comment

We are interested in the supramolecular potential of NH-acidic di(organosulfonyl)amines, $(RSO_2)_2NH$, and have published numerous crystal structures of molecular cocrystals, solvates, metal coordination compounds and organic salts involving $(RSO_2)_2NH$ or $(RSO_2)_2N^-$ entities. In particular, proton-transfer reactions of di(methanesulfonyl)amine ('dimesylamine'), $(CH_3SO_2)_2NH$, with rationally selected nitrogen bases (amines and aza-aromatics) afforded an extended series of ionic crystals whose packing patterns are governed by a wide variety of charge-assisted hydrogen-

bonding systems in zero, one, two or three dimensions (e.g. Moers et al., 1999, 2000, 2001; Wijaya et al., 2000; Wijaya, Moers, Blaschette et al., 2004; Wijaya, Moers, Henschel et al., 2004). Our recent studies of silver complexes of rac-trans-cyclohexane-1,2-diamine (Wölper et al., 2010), in turn inspired by the work of Englert (Kalf et al., 2006) on racemic and enantiomerically pure complexes of the same amine, prompted us to use this amine to synthesize adducts with di(methanesulfonyl)amine. Here we present the structures of two crystal forms of the ionic 1:1 adduct rac-trans-2-amino-cyclohexan-1-aminium di(methanesulfonyl)azanide.

(I) n = 0.11
(II) n = 0

rac-trans-2-Aminocyclohexan-1-aminium di(methanesulfonyl)azanide 0.11-hydrate, (I), crystallizes with a partially occupied water site in the monoclinic Sohnke space group $C2$. The asymmetric unit (Fig. 1) consists of two cations (C11 and C12 are R-, and C21 and C22 S-configured, i.e. one of each enantiomer), one anion on a general position, two half anions, each with the N atom on a twofold axis, and approximately one fifth of a water molecule. Consequently, the compound is racemic despite its Sohnke space group. The anhydrous form, (II), crystallizes in the monoclinic space group $P2_1/c$ with one formula unit in the asymmetric unit (Fig. 2), which was chosen to contain the R,R-enantiomer of the 2-aminocyclohexan-1-aminium cation.

In the great majority of compounds containing the di(methanesulfonyl)azanide anion, a 'W'-shaped sequence of O—S—N—S—O atoms is observed, with two O—S—N—S torsion angles of ca $\pm 180°$. However, in one of the anions in
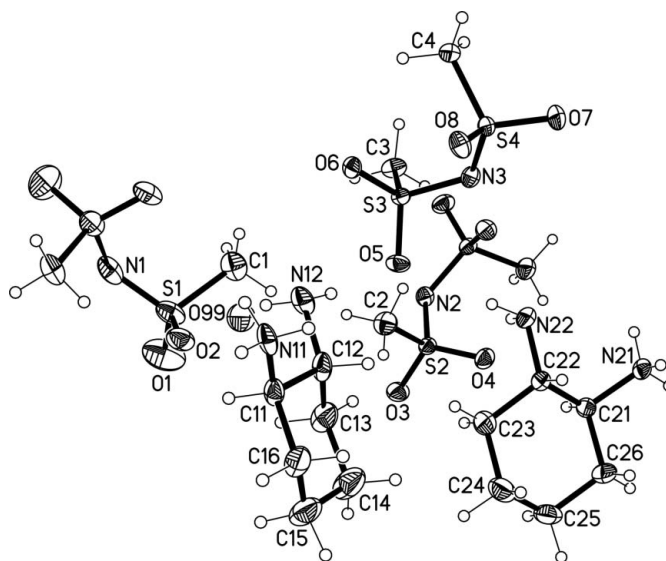

Figure 1
The unique components of (I). Displacement ellipsoids represent 50% probability levels.
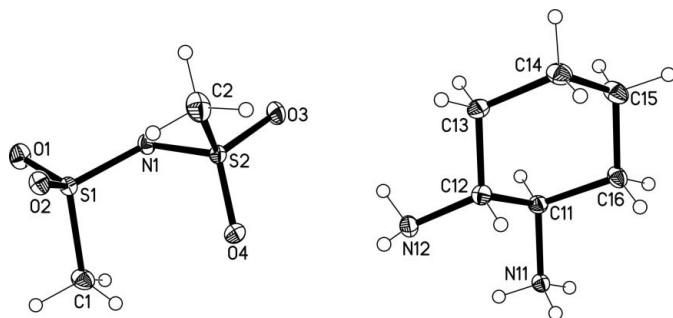
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[1] For part CXC, see Zerbe et al. (2011).

organic compounds

Figure 2
The asymmetric unit of (II). Displacement ellipsoids represent 50% probability levels.

Figure 4
The contacts within the *R,R*-ribbons of (I). Classical hydrogen bonds are shown with thick and nonclassical hydrogen bonds are shown with thin dashed lines. (Symmetry codes are as in Table 1.)
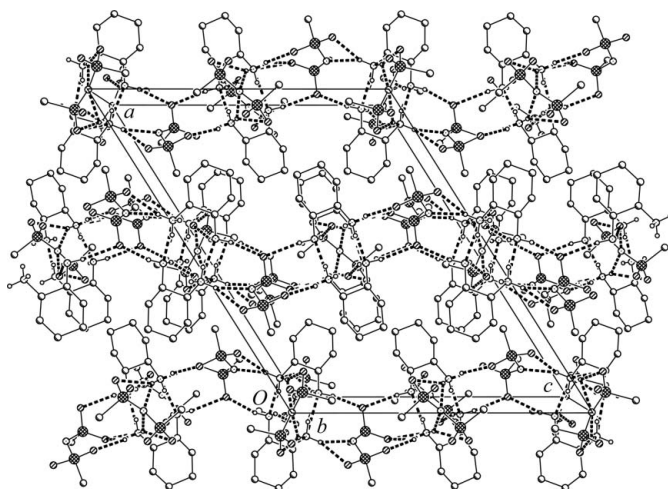

Figure 3
The corrugated layers of (I) parallel to (100) seen from the side. The view is parallel to the *b* axis along the *R,R*- and *S,S*-ribbons. Classical hydrogen bonds are shown with thick dashed lines.

Figure 5
The contacts within the *S,S*-ribbons of (I). Classical hydrogen bonds are shown with thick and nonclassical hydrogen bonds are shown with thin dashed lines. (Symmetry codes are as in Table 1.)

(I) the typical 'W' sequence is distorted; only O7—S4—N3—S3 fits the pattern [−176.18 (8)°], whereas around S3—N3, the largest (absolute) torsion angle is O5—S3—N3—S4 [−132.00 (8)°]. The pseudo-torsion angle O5—S3···S4—O7 is 65.54 (8)°, resulting in a pseudo-*gauche* conformation in contrast to the normally observed pseudo-ecliptic arrangement. The rotation of the SO_2 group constituted by atoms S3, O5 and O6 allows the formation of additional hydrogen bonds (see below).

In the packing of (I), an extensive system of classical hydrogen bonds combines to form corrugated layers parallel to (100) (see Fig. 3, in which the layers are seen edge-on), consisting of a grid-like arrangement of ribbons parallel to the *b* and *c* axes. All potential classical hydrogen-bond donors and acceptors are involved, with the exception of the water site (see below). Parallel to the *b* axis, two independent ribbons are associated with the twofold axes, leading to a completion of the anions with nitrogen on the special position and to the formation of cation dimers *via* N11—H3···N12[i] and N21—H8···N22[iii] interactions, respectively [all symmetry codes used in the discussion of (I) are as in Table 1]. Each of these ribbons consists of only one enantiomer of the cation, *i.e.* neighbouring ribbons are of opposite chirality (and are henceforth referred
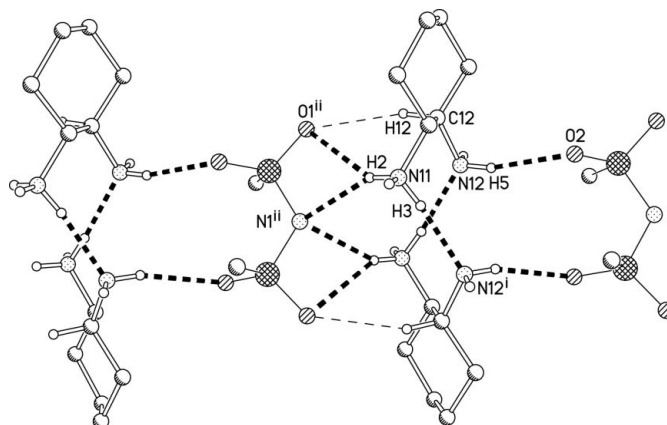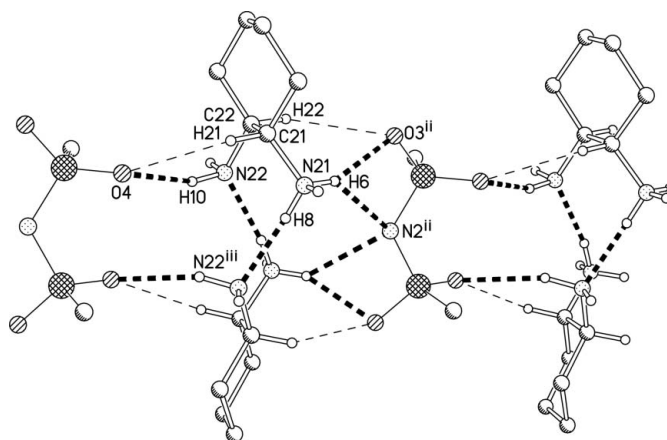
to as *R,R*- and *S,S*-ribbons according to the chirality of the respective cations). Defining the direction of the anion by the S···S vector and the direction of the cation by the vector joining the mid-points of both (N)C—C(N) bonds, the angle between these vectors is 30.0° for the anion based on S1 and 8.5° for the anion based on S2. As the anions of the *R,R*- and *S,S*-ribbons subtend different angles with the cation dimers the strengths of the hydrogen bonds forming them must differ, but the qualitative patterns are the same (Figs. 4 and 5). Within each ribbon, the ions are connected in one direction *via* the three-centred interactions with H2 (*R,R*-ribbon) or H6 (*S,S*-ribbon) as donors; in the opposite direction, the classical two-centre interactions N12—H5···O2 or N22—H10···O4 are involved, of which the former is more linear [166.1 (17)° compared to 157.6 (14)°]. The three-centred bonds are of similar strength. The different arrangement of the ions in the two ribbons has most influence on the nonclassical hydrogen bonds. In the *S,S*-ribbon, two longer interactions are observed (C21—H21···O4 and C22—H22···O3[ii]), while in the *R,R*-ribbon, only one short interaction is found (C12—H12···O1[ii]);
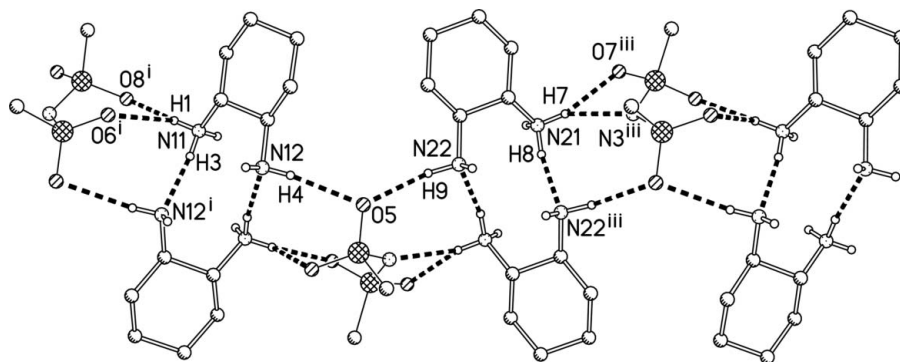
Figure 6
The contacts between the ribbons and the connecting anions in the packing of (I). Classical hydrogen bonds are shown with thick dashed lines. (Symmetry codes are as in Table 1.)

the corresponding hydrogen H11 shows a long contact to O6^i which is strongly bent and presumably has a limited structure-determining influence.

The ribbons are connected by hydrogen bonds to the anion on a general position (Fig. 6). Both cases involve one three-centred (N11—H1$\cdots$O6^i, N11—H1$\cdots$O8^i and N21—H7$\cdots$N3^{iii} and N21—H7$\cdots$O7^{iii}) and one bifurcated system in which H4 and H9 share the same acceptor (O5). These interactions could explain the deviation from the typical conformation of this anion. Without the distortion of the 'W' sequence, only one of the SO$_2$ groups could take part in interactions. Apparently the energy gained by the hydrogen bonds at least compensates the energy necessary to change the conformation of the anion. It should be noted that a similarly distorted anion occurs in the structure of inversion-symmetric

[*trans*-(CH$_3$)$_2$Sn(urea)$_4$]·[(CH$_3$SO$_2$)$_2$N]$_2$·6(urea), where the (SO$_2$)$_2$N group accepts a total of 12 hydrogen bonds with urea NH$_2$ as donors (Wirth *et al.*, 1998). The bond patterns between the different ribbons and the anion on the general position necessarily differ, since the *S,S*- and *R,R*-ribbons approach the (distorted) 'W' sequence of the anion from opposite sides, and consequently the acceptors of the three-centred hydrogen bonds are not alike. The anions on general positions connect the *R,R*- and *S,S*-ribbons to constitute a grid-like layer in whose meshes the partially occupied water molecule is included (Fig. 7). The layers are corrugated because the cation dimers are mutually rotated about 38.7° [angle between vectors joining the mid-points of the (N)C—C(N) bonds]. Atom O99 is a potential acceptor for several contacts, of which only C2—H2B$\cdots$O99 can be described unequivocally as a hydrogen bond. As the water H atoms could not be identified, no certain statement about the donor properties of O99 can be given, but O1 [3.225 (6) Å] and O5^{iv}, O8^{iv} and N3^{iv} [3.435 (6) to 3.512 (6) Å] lie within the range of potential acceptors. It is
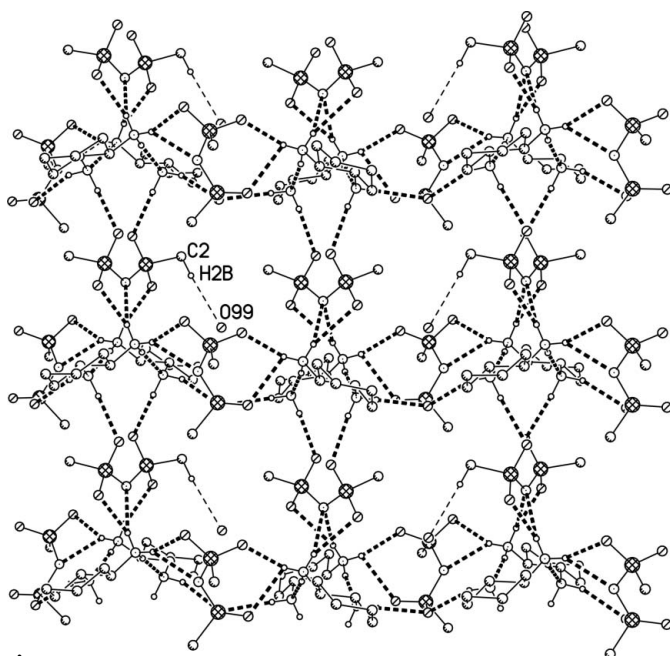
Figure 7
The grid-like arrangement of the ribbons in (I) (running vertically with an *R,R*-ribbon in the middle and *S,S*-ribbons at the sides) within the layer [seen from above, view perpendicular to (100), with *b* pointing up and *c* pointing to the right]. Classical hydrogen bonds are shown with thick and nonclassical hydrogen bonds with thin dashed lines.

Figure 8
Layers of (II) parallel to (100), seen from the side. Classical hydrogen bonds are shown with thick and nonclassical hydrogen bonds are shown with thin dashed lines.

Figure 9
The repeat unit of the ribbons in (II). Classical hydrogen bonds are shown with thick dashed lines. (Symmetry codes are as in Table 2.)

Figure 10
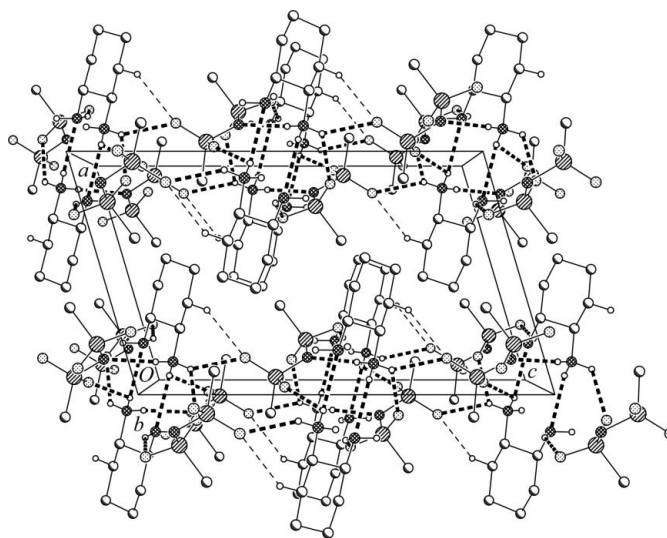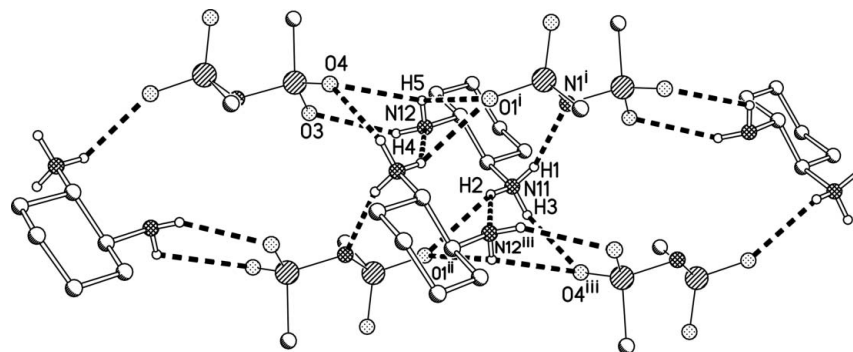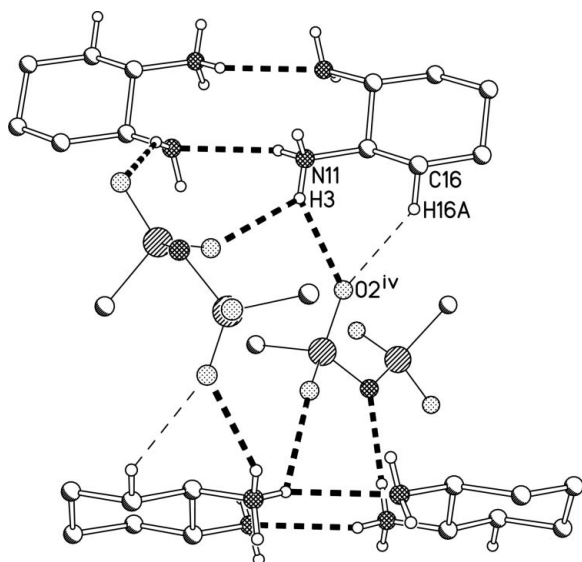The contacts connecting the ribbons in (II). Classical hydrogen bonds are shown with thick and nonclassical hydrogen bonds are shown with thin dashed lines. (Symmetry codes are as in Table 2.)

not clear to what extent the partially occupied water site is essential for the formation of the partial hydrate, (I), nor over what range of water occupation factors the same structure is maintained; we have conducted no experiments to investigate this. The difference in composition between structures (I) and (II) means that, strictly speaking, they are not polymorphs, but could perhaps be classified as pseudopolymorphs (Nangia, 2006, and references therein). A similar case of two crystal forms not being polymorphs because of traces of water in one of the structures was reported recently (Minkov, 2011).

The methyl H atoms of the anion are activated because of the electron-withdrawing effects of the hetero atoms, and this leads to several nonclassical hydrogen bonds that reinforce the classical hydrogen bonds. Special attention should be paid to C3—H3C···O7^v and C4—H4C···O7^v since they are inter-layer interactions, but the latter is weak. Further C—H donors of the cation form hydrogen bonds, of which one (C25—H25A···O8^{viii}) also connects the layers. It is noteworthy that all hydrogen bonds within the layer are formed by lattice translation and twofold rotation, while interactions beyond

the layer always include lattice centring, *i.e.* lattice centring itself and the 2_1-screw axes resulting from its combination with twofold axes.

The packing of (II) is broadly similar to that of (I) in that a system of classical hydrogen bonds combines to form layers parallel to (100); again, all potential classical hydrogen-bond donors and acceptors are involved (Fig. 8). No directed interaction can be found between these layers. The layers can best be described as parallel ribbons of alternating cation dimers and anions. The dimers are formed *via* inversion, with N11—H2···N12^{iii} connecting the cations, hence leading, unlike in (I), to a racemic composition of the ribbons [all symmetry codes used in the discussion of (II) are as in Table 2]. N11—H3···O4^{iii}, N12—H4···O3 and N12—H5···O4 attach the anion to the dimer, thus establishing the repeat unit of the chain (Fig. 9) while N11—H1···N1^i, N11—H2···O1^{ii} and N12—H5···O1^i connect to the next unit, thus completing the chain formation parallel to *b*. The remaining acceptor O2 enables the connection of the ribbons by the N11—H3···O2^{iv} hydrogen bond, which is accompanied by the nonclassical C16—H16A···O2^{iv} hydrogen bond (Fig. 10). Since most of the hydrogen-bond donors interact with more than one acceptor, most angles at the H atoms are narrow [113.1 (16)–139.6 (14)°]; the bonds involving one acceptor only are far more linear [150.5 (16) and 161.6 (15)°]. Except for those from H5, which are both 2.62 (2) Å long, all hydrogen bonds are significantly shorter than the sum of the van der Waals radii. As N12—H5···O1^i and N12—H5···O4 are not only long but also deviate the most from linearity [113.1 (16) and 116.5 (16)°, respectively], they must be regarded as weak.

In both structures, well defined layers, with a separation of the hydrophilic regions from the hydrophobic alkyl residues of the cations, can be observed. Ribbons of alternating anions and cation dimers can be found in both cases. However, the most obvious difference between the two structures is that in (I) the layers are corrugated while in (II) they are flat. In (I), each cation dimer consists of only one enantiomer, in contrast to (II) which is composed of racemic dimers. Because of the partially occupied water molecule, the overall density of (I) is lower [1.359 Mg m^{-3} in (I) and 1.478 Mg m^{-3} in (II)]. Apart from the formation of the cation dimers, the hydrogen-bond patterns show little similarity. For this reason it seems sensible to assume that the enantiopure or racemic dimers are

responsible during nucleation for the constitution of the different structures. Alternatively, one might speculate that the water molecule strongly influences the formation of the packing at an early stage of crystal growth.

Experimental

The first attempts to synthesize adducts of *rac-trans*-cyclohexane-1,2-diamine and di(methanesulfonyl)amine used a 2:1 molar ratio in dichloromethane. Despite this, only the 1:1 adduct was obtained. Liquid–liquid diffusion of diethyl ether into such solutions led to crystals of form (I), whereas the use of petroleum ether led to form (II). We did not investigate whether both crystal forms were formed from the alternative solvent mixtures. Elemental analysis of form (II) was satisfactory (found: C 33.42, H 7.30, N 14.32, S 22.18%; calculated C 33.43, H 7.36, N 14.62, S 22.31%).

Compound (I)

Crystal data

$C_6H_{15}N_2^+ \cdot C_2H_6NO_4S_2^- \cdot 0.11H_2O$	$V = 2828.2\ (2)\ \text{Å}^3$
$M_r = 289.38$	$Z = 8$
Monoclinic, $C2$	Mo $K\alpha$ radiation
$a = 21.9116\ (6)\ \text{Å}$	$\mu = 0.39\ \text{mm}^{-1}$
$b = 8.84942\ (10)\ \text{Å}$	$T = 100\ \text{K}$
$c = 17.2357\ (4)\ \text{Å}$	$0.25 \times 0.20 \times 0.10\ \text{mm}$
$\beta = 122.196\ (6)°$	

Data collection

Oxford Diffraction Xcalibur E diffractometer	7844 independent reflections
59469 measured reflections	6813 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.054$	
$S = 0.95$	
7844 reflections	$\Delta\rho_{max} = 0.29\ \text{e Å}^{-3}$
347 parameters	$\Delta\rho_{min} = -0.27\ \text{e Å}^{-3}$
9 restraints	Absolute structure: Flack (1983), 3637 Friedel pairs
	Flack parameter: 0.04 (3)

Compound (II)

Crystal data

$C_6H_{15}N_2^+ \cdot C_2H_6NO_4S_2^-$	$V = 1291.77\ (6)\ \text{Å}^3$
$M_r = 287.40$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 9.7958\ (3)\ \text{Å}$	$\mu = 0.42\ \text{mm}^{-1}$
$b = 8.5522\ (2)\ \text{Å}$	$T = 90\ \text{K}$
$c = 16.0779\ (4)\ \text{Å}$	$0.25 \times 0.20 \times 0.18\ \text{mm}$
$\beta = 106.455\ (3)°$	

Data collection

Oxford Diffraction Xcalibur E diffractometer	3620 independent reflections
39527 measured reflections	3110 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.075$	
$S = 1.09$	
3620 reflections	$\Delta\rho_{max} = 0.55\ \text{e Å}^{-3}$
176 parameters	$\Delta\rho_{min} = -0.32\ \text{e Å}^{-3}$

Table 1
Geometry of hydrogen bonds and interspecies contacts (Å, °) for (I).

$D—\text{H}\cdots A$	$D—\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D—\text{H}\cdots A$
N11—H1$\cdots$O6[i]	0.85 (1)	2.52 (2)	2.9567 (16)	112.6 (13)
N11—H1$\cdots$O8[i]	0.85 (1)	2.04 (1)	2.8721 (16)	164.1 (16)
N11—H2$\cdots$O1[ii]	0.86 (1)	2.54 (1)	3.304 (2)	149.9 (15)
N11—H2$\cdots$N1[ii]	0.86 (1)	2.30 (1)	3.076 (2)	150.6 (15)
N11—H1$\cdots$O6[i]	0.85 (1)	2.52 (2)	2.9567 (16)	112.6 (13)
N11—H3$\cdots$N12[i]	0.89 (1)	2.01 (1)	2.900 (2)	171.4 (16)
N12—H4$\cdots$O5	0.86 (1)	2.30 (1)	3.1585 (15)	173.2 (16)
N12—H5$\cdots$O2	0.85 (1)	2.24 (1)	3.0656 (18)	166.1 (17)
N21—H6$\cdots$O3[ii]	0.90 (1)	2.11 (1)	2.9433 (17)	154.7 (15)
N21—H6$\cdots$N2[ii]	0.90 (1)	2.49 (2)	3.2499 (19)	141.9 (14)
N21—H7$\cdots$O7[iii]	0.89 (1)	2.22 (1)	3.0613 (16)	157.3 (14)
N21—H7$\cdots$N3[iii]	0.89 (1)	2.32 (1)	3.0453 (16)	138.3 (14)
N21—H8$\cdots$N22[iii]	0.92 (1)	1.99 (1)	2.8889 (19)	166.9 (14)
N22—H9$\cdots$O5	0.94 (1)	2.24 (2)	3.1416 (16)	159.7 (15)
N22—H10$\cdots$O4	0.97 (1)	2.34 (2)	3.2593 (18)	157.6 (14)
C1—H1$C\cdots$O8[iv]	0.98	2.63	3.5771 (19)	162
C2—H2$B\cdots$O99	0.98	2.62	3.578 (6)	166
C3—H3$C\cdots$O7[v]	0.98	2.61	3.5373 (19)	159
C4—H4$A\cdots$O4[vi]	0.98	2.68	3.4874 (19)	140
C4—H4$C\cdots$O7[v]	0.98	2.83	3.6972 (19)	149
C12—H12$\cdots$O1[ii]	1.00	2.48	3.353 (2)	146
C14—H14$B\cdots$O1[vii]	0.99	2.81	3.706 (3)	151
C16—H16$A\cdots$O1[ii]	0.99	2.74	3.533 (2)	138
C21—H21$\cdots$O4	1.00	2.62	3.4812 (18)	144
C22—H22$\cdots$O3[ii]	1.00	2.74	3.5025 (18)	134
C25—H25$A\cdots$O8[viii]	0.99	2.69	3.549 (2)	145
C26—H26$A\cdots$O3[ii]	0.99	2.58	3.377 (2)	137
Interspecies contacts				
N22—H9$\cdots$O99[ii]	0.94 (1)	2.83 (2)	3.312 (6)	113.2 (12)
C1—H1$C\cdots$O99	0.98	2.87	3.452 (7)	119
C11—H11$\cdots$O6[i]	1.00	2.71	3.2789 (17)	116
C13—H13$A\cdots$O2	0.99	2.77	3.519 (2)	133
C22—H22$\cdots$O99[ii]	1.00	2.70	3.328 (6)	121

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

Table 2
Hydrogen-bond geometry (Å, °) for (II).

$D—\text{H}\cdots A$	$D—\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D—\text{H}\cdots A$
N11—H1$\cdots$N1[i]	0.862 (17)	2.129 (18)	2.9593 (14)	161.6 (15)
N11—H2$\cdots$O1[ii]	0.848 (18)	2.403 (17)	2.9627 (13)	124.0 (14)
N11—H2$\cdots$N12[iii]	0.848 (18)	2.356 (17)	3.0408 (14)	138.1 (14)
N11—H3$\cdots$O2[iv]	0.892 (18)	2.237 (17)	2.9735 (13)	139.6 (14)
N11—H3$\cdots$O4[iii]	0.892 (18)	2.393 (17)	3.0377 (13)	129.3 (14)
N12—H4$\cdots$O3	0.87 (2)	2.40 (2)	3.1864 (14)	150.5 (16)
N12—H5$\cdots$O1[i]	0.87 (2)	2.62 (2)	3.0667 (14)	113.1 (16)
N12—H5$\cdots$O4	0.87 (2)	2.62 (2)	3.1029 (13)	116.5 (16)
C16—H16$A\cdots$O2[iv]	0.99	2.45	3.1891 (14)	132

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

For (I), H atoms attached to N atoms were refined freely, but with the N—H distances at each N atom restrained to be similar and with $U_{iso}(\text{H})$ values constrained to $1.2U_{eq}(\text{N})$. Methyl H atoms were identified in difference syntheses; the geometry was idealized (C—H = 0.98 Å and H—C—H = 109.5°) and the methyl groups were refined as rigid groups that were allowed to rotate but not to tip. Other H atoms were included at calculated positions (methine C—H = 1.00 Å and methylene C—H = 0.99 Å) and refined using a riding model. $U_{iso}(\text{H})$ values were set at $mU_{eq}(\text{C})$, with $m = 1.5$ for methyl and 1.2 for riding H atoms.

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The compound is a racemate but crystallizes by chance in a Sohnke space group. The refinement of the highest remaining Fourier peak as oxygen yielded a site-occupancy factor of 22.1 (5)%. The H atoms of the water molecule could not be identified, but the short distances (3.225–3.512 Å) to potential hydrogen-bond acceptors and to H atoms of (nonclassical) donor groups (2.62–2.87 Å) strongly suggest that treating this peak as partially occupied water is justified. Ignoring the peak results in a solvent-accessible void of 36 Å^3 at $x = 0.944$, $y = 0.726$, $z = 0.713$ (coordinates of the peak = 0.9622, 0.7419, 0.7383).

For (II), H atoms attached to N atoms were refined freely; other H atoms were refined as described for compound (I).

For both compounds, data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: EG3071). Services for accessing these data are described at the back of the journal.

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