J Chem Crystallogr (2009) 39:662–668
DOI 10.1007/s10870-009-9554-8

ORIGINAL PAPER

Hydrogen Bonding in the Crystal Structures of New Imidazolium Triflimide Protic Ionic Liquids

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Received: 26 May 2008 / Accepted: 9 March 2009 / Published online: 20 March 2009

Abstract The synthesis and crystal structures of 1,3-diamino-2-methylimidazolium bis(trifluoromethylsulfonyl)imide (**1**), 1,3-dihydroxy-2-methylimidazolium bis(trifluoromethylsulfonyl)imide (**2**) and 1-(2-(diethylammonio)ethyl)-3-methylimidazolium bis(bis(trifluoromethylsulfonyl)imide) (**4**) are reported. The salts **1**, **2** and **4** have melting points below 100 °C, the intermediate 1-(2-(diethylamino)ethyl)-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (**3**) is liquid at room temperature. Compound **1** is monoclinic, space group $P2_1/n$ with $a = 8.4979(4)$ Å, $b = 12.2803(6)$ Å, $c = 13.9400(7)$ Å, $\beta = 93.086(4)^\circ$, and $Z = 4$. Compound **2** is monoclinic, space group $P2_1/c$ with $a = 7.6165(2)$ Å, $b = 20.5323(8)$ Å, $c = 9.7654(3)$ Å, $\beta = 111.046(2)^\circ$, and $Z = 4$. Compound **4** is triclinic, space group $P\bar{1}$ with $a = 8.5313(4)$ Å, $b = 9.2157(4)$ Å, $c = 20.5812(8)$ Å, $\alpha = 84.668(2)^\circ$, $\beta = 83.738(2)^\circ$, $\gamma = 63.096(2)^\circ$, and $Z = 2$. The ions in **1** build a network of N–H···O hydrogen bonds, in **2** they are linked to chains by O–H···N and bifurcated O–H···O hydrogen bonds, whereas in **4** they form pairs by N–H···O contacts. The triflimide anions adopt transoid conformations.

Keywords Crystal structure · Hydrogen bond · Ionic liquid · Triflimide

Introduction

The bis(trifluoromethylsulfonyl)imide ('triflimide') anion [1] has been introduced for the synthesis of hydrophobic ionic liquids (ILs) which are of interest in electrochemistry and separation techniques. The triflimide anion is effective at forming low-melting salts. This property has been attributed to its irregular shape and bulky nature, charge delocalization and steric shielding of the nitrogen atom [2, 3]. The triflimide anion has been considered as non-coordinating and non-hydrogen-bonding [1]. However, salts of oxophilic metals exhibit strong metal–anion interactions to the sulfonyl oxygens resulting in the formation of extended network structures [4]. Even direct contacts between metal ions, e.g., Cs [4], Cu [5], Au [6], Fe [7], Ru [7], and the triflimide nitrogen atom have been observed. Direct O–H···N interactions were also observed with solvent molecules such as methanol [4] and water [4, 8–10]. Triflimide anions are flexible, adopting either of two preferred low-energy transoid or cisoid conformations (indicated by the C–S···S–C torsion angle) as predicted by theoretical calculations [4, 11]. The cisoid orientation is often observed in the presence of chelating metal ions (Li, Na, K, Cs [4]; Ca, Sr, Ba [9, 12]). However, the structural diversity of Li complexes, depending on the presence of additional ligands, shows that the conformation is hard to predict [13].

Organic cations, typically, quaternary imidazolium [1, 14–22], pyrrolidinium [11, 23–25, 27], tetraalkylammonium [2, 26], pyridinium [23], and piperidinium [24] ions have been employed. Structures of these organic triflimide salts typically show no strong interionic contacts [17]. This

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absence of any strong interaction between the anion and cation may help explain the low melting points of these salts. In general, the triflimide anions here prefer transoid conformations. Only in rare cases, such as 1,3-dimethylimidazolium [14], 1-(1-(ethoxycarbonyl)ethyl)-3-methylimidazolium [15], 1-ethyl-3-methylimidazolium [19], and 1-hexylpyridinium [23] salts, the triflimide anion adopts a cisoid conformation in organic salts. Analysis of 143 triflimide data from the CSD (without disordered structures) revealed the presence of 83 transoid and 60 cisoid structures (more precisely 45 synperiplanar, 15 synclinal, 12 anticlinal, 71 antiperiplanar). A torsion angle of ±170 ± 5° is preferred in the majority (45 out of 143) of the structures as shown in Fig. 1. All CSD (version 5.29, Jan 2008, 436384 entries) searches were performed using Conquest 1.10. Notably, orientational disorder of triflimide ions in crystal structures is not uncommon [2, 11, 23, 27]. This disordering may contribute to the low melting points as well.

Recently, protic ionic liquids (PILs) [28] have been recognized as an interesting class of ILs. A convenient method of preparation of PILs by protonation of 1-alkylimidazoles using Tf_2NH has been proposed [29]. In protic salts more interactions between the ions are to be expected. However, in the few crystalline examples known, no unexpected networks or direct contacts to the triflimide nitrogen atom have been detected. Thus, in trimethylammonium [26] and ammonium triflimide [13], the cations again participate in weak hydrogen bonding with two of the sulfonyl oxygen atoms. Surprisingly, in one of the structures of *N*,*N*,*N*-trimethylglycine triflimide [11], solely cationic interactions were seen. For the sake of completeness, it should be mentioned that the corresponding acid, bis(trifluoromethylsulfonyl)amide, exhibits bifurcated N–H···O hydrogen bonds [10, 30].

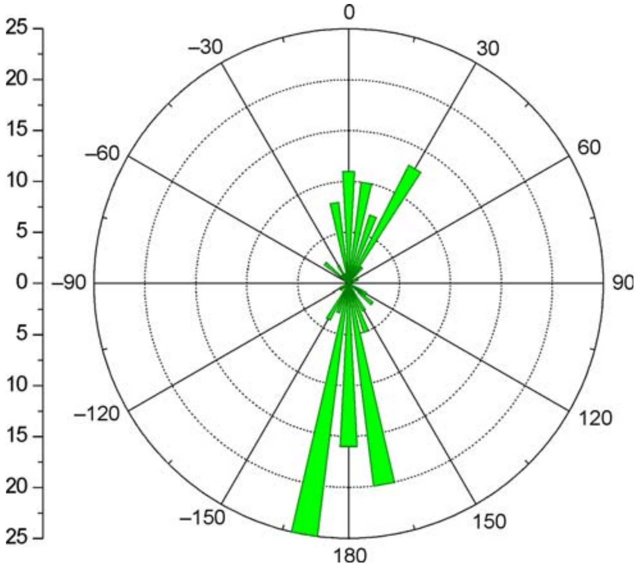


Fig. 1 Polar histogram showing the distribution of C–S···S–C torsion angles in known crystalline triflimides

Here, we report three crystal structures of new, low melting, protic organic triflimide salts with different hydrogen bonding motifs.

Experimental Section

NMR spectra were recorded with a Bruker AC 300 spectrometer. IR spectra were obtained with a Nicolet 5700 FT instrument. The crystal structures were determined using Nonius KappaCCD and STOE IPDS 2 diffractometers, respectively. The experimental conditions and crystallographic data are listed in Table 1. X-Ray diffraction data were collected with graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). The structures were solved using direct methods and refined by full-matrix least-squares techniques. All non-hydrogen atoms were assigned anisotropic displacement parameters in the refinement. The structures were refined on F^2 using SHELXTL-97 [31]. The crystallographic data have been deposited with the Cambridge Crystallographic Data Center, under reference CCDC 686015–686017.

1,3-Diamino-2-Methylimidazolium Bis(trifluoromethylsulfonyl)imide (**1**)

A mixture of 1,3-diamino-2-methylimidazolium chloride [32] (1.20 g, 8.2 mmol) and $LiNTf_2$ (2.36 g, 8.2 mmol) in H_2O (20 mL) was ultrasonicated for 5 min. The precipitate was filtered and dried over P_2O_5. Yield: 1.2 g (26%). M.p. 58–59 °C. IR (neat): 3,384, 3,308, 3,144, 1,644, 1,565, 1,353, 1,323, 1,182, 1,131, 1,051, 919, 797, 736, 729, 644, 608, 569, 515 cm^{-1}. ^{1}H NMR (DMSO-d_6): 2.49 (s, 3H), 6.56 (br), 7.46 (s, 2H) ppm. ^{13}C NMR (DMSO-d_6): 8.1, 119.5 (q, 2C, $J = 325$ Hz), 120.9 (2C), 144.4 ppm.

1,3-Dihydroxy-2-Methylimidazolium Bis(trifluoromethylsulfonyl)imide (**2**)

A mixture of Tf_2NH (35.9 g, 128 mmol) and of 1-hydroxy-2-methylimidazole 3-oxide [33] (14.6 g, 128 mmol) was heated to 80 °C, causing the mixture to liquefy. After stirring for 1 h, the reaction mixture was allowed to cool to room temperature, giving 50.5 g (100%) of crystalline product. M.p. 72 °C. IR (neat): 3,323, 3,152, 2,797, 1,364, 1,354, 1,184, 1,132, 1,052, 793, 748, 714, 605 cm^{-1}. ^{1}H NMR (DMSO-d_6): 2.48 (s, 3H), 7.86 (s, 2H), 13.5 (br, 2H) ppm. ^{13}C NMR (DMSO-d_6): 7.1, 117.4 (2C), 119.5 (q, 2C, $J = 321$ Hz), 136.6 ppm.

Table 1 Crystallographic data and structure refinement of **1**, **2** and **4**

Compound	**1**	**2**	**4**
CCDC no.	686015	686016	686017
Empirical formula	$C_6H_9F_6N_5O_4S_2$	$C_6H_7F_6N_3O_6S_2$	$C_{14}H_{21}F_{12}N_5O_8S_4$
Formula weight	393.29	395.27	743.60
Temperature (K)	173(2)	233(2)	233(2)
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	$P2_1/n$	$P2_1/c$	$P\overline{1}$
Unit cell dimensions			
a (Å)	8.4979(4)	7.6165(2)	8.5313(4)
b (Å)	12.2803(6)	20.5323(8)	9.2157(4)
c (Å)	13.9400(7)	9.7654(3)	20.5812(8)
α (°)	90	90	84.668(2)
β (°)	93.086(4)	111.046(2)	83.738(2)
γ (°)	90	90	63.096(2)
Volume ($Å^3$)	1452.61(13)	1425.28(8)	1432.65(11)
Z	4	4	2
D_{calc} (g/cm^3)	1.798	1.842	1.724
Absorption coefficient (mm^{-1})	0.46	0.477	0.458
F(000)	792	792	752
Crystal size (mm^3)	0.4 × 0.2 × 0.2	0.3 × 0.15 × 0.15	0.2 × 0.15 × 0.08
θ range for data collection	1.46–25.07	1.98–25.00°	1.99–24.98°
Index ranges	$-10 \le h \le 9$	$-7 \le h \le 9$	$-10 \le h \le 10$
	$-14 \le k \le 14$	$-24 \le k \le 24$	$-10 \le k \le 10$
	$-15 \le l \le 16$	$-11 \le l \le 11$	$-24 \le l \le 23$
Reflections collected	9,188	7,637	7,983
Independent reflections	2,498 ($R_{int} = 0.0288$)	2,485 ($R_{int} = 0.0232$)	4,932 ($R_{int} = 0.0211$)
Reflections [$I > 2\sigma(I)$]	2,112	2,154	3,743
Absorption correction	None	None	None
Diffractometer	STOE IPDS 2	Nonius KappaCCD	Nonius KappaCCD
Data/restraints/parameters	2,498/0/225	2,485/2/272	4,932/0/493
Goodness-of-fit on F^2	1.051	1.060	1.071
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0311$, $wR_2 = 0.0692$	$R_1 = 0.0387$, $wR_2 = 0.0984$	$R_1 = 0.0459$, $wR_2 = 0.1087$
R indices (all data)	$R_1 = 0.0409$, $wR_2 = 0.0727$	$R_1 = 0.0448$, $wR_2 = 0.1016$	$R_1 = 0.0670$, $wR_2 = 0.1178$
Largest diff. peak and hole (e Å^{-3})	0.265, −0.264	0.358, −0.249	0.294, −0.352

1-(2-(Diethylamino)ethyl)-3-Methylimidazolium Bis(trifluoromethylsulfonyl)imide (**3**)

A mixture of 1-methylimidazole (8.0 g, 97 mmol) and *N*,*N*-diethyl-2-amino-1-bromoethane hydrobromide [CARN 1069-72-3] (25.4 g, 97 mmol) in acetonitrile (100 mL) was refluxed for 24 h. The resulting 1-(2-(diethylammonio)ethyl)-3-methylimidazolium dibromide (29.0 g, 82%) was filtered, washed with Et_2O and dried. M.p. 164 °C. IR (neat): 3,080, 2,924, 2,662, 1,574, 1,459, 1,165, 1,020, 768 cm^{-1}. ^{1}H NMR (DMSO-d$_6$): 1.20 (t, 6H, $J = 7.2$ Hz), 3.19 (q, 4H, $J = 7.2$ Hz), 3.61 (t, 2H, $J = 6.4$ Hz), 3.85 (s, 3H), 4.63 (t, 2H, $J = 6.4$ Hz), 7.73 (s, 1H), 7.89 (s, 1H), 9.26 (s, 1H) ppm.

A solution of this dibromide (12.0 g, 35 mmol) in H_2O (50 mL) was stirred with NaOH (1.4 g, 35 mmol) and $LiNTf_2$ (10.0 g, 35 mmol) for 4 h. The mixture was extracted with CH_2Cl_2 (50 mL). The organic phase was washed with H_2O (3 × 50 mL) and concentrated to give the liquid product **3** (14.25 g, 88%). $n_D^{20} = 1.4360$. IR (neat): 3,158, 2,973, 2,818, 1,571, 1,459, 1,347, 1,177, 1,133, 1,051, 739, 569 cm^{-1}. ^{1}H NMR (DMSO-d$_6$): 0.85

(t, 6H, $J = 7.1$ Hz), 2.46 (q, 4H, $J = 7.1$ Hz), 2.73 (t, 2H, $J = 5.8$ Hz), 3.86 (s, 3H), 4.18 (t, 2H, $J = 5.8$ Hz), 7.61 (s, 1H), 7.70 (s, 1H), 9.02 (s, 1H) ppm.

1-(2-(Diethylammonio)ethyl)-3-Methylimidazolium Bis(bis(trifluoromethylsulfonyl)imide) (**4**)

To a solution of **3** (2.68 g, 5.8 mmol) in CH_2Cl_2 (20 mL) Tf_2NH (1.63 g, 5.8 mmol) was added, causing a second, heavier layer to form. While stirring this mixture at room temperature, the product solidified. Filtration of the precipitate, washing with CH_2Cl_2 (10 mL) and drying in vacuum yielded **4** as a white powder (3.34 g, 78%).

Alternatively, to a solution of the dibromide (3.09 g, 9 mmol) in H_2O (10 mL) an aqueous solution (14.8 g, 70%) of $LiNTf_2$ (corresponding to 10.4 g of $LiNTf_2$, 36 mmol) was added. Immediately, a second, heavier layer formed. The reaction mixture was stirred at room temperature for 10 min, then CH_2Cl_2 (10 mL) was added, resulting in the formation of a third layer. After stirring for additional 10 min, the heaviest layer was separated and freed from residual solvents by means of a rotary evaporator. The resulting colorless solid **4** was dried in a vacuum desiccator (5.64 g, 84%). M.p. 69–71 °C. IR (neat): 3,155, 3,123, 3,082, 1,343, 1,175, 1,135, 1,051, 973, 872, 791, 752, 742, 607, 569, 511 cm^{-1}. ^1H NMR (DMSO-d_6): 1.19 (t, 6H, $J = 7.2$ Hz), 3.19 (q, 4H, $J = 7.2$ Hz), 3.57 (t, 2H, $J = 6.6$ Hz), 3.87 (s, 3H), 4.55 (t, 2H, $J = 6.6$ Hz), 7.73 (s, 1H), 7.80 (s, 1H), 9.12 (s, 1H), 9.24 (br s, 1H) ppm. ^{13}C NMR (DMSO-d_6): 8.4 (2C), 35.9, 43.1, 47.0 (2C), 49.4, 119.5 (q, 2C, $J = 322$ Hz), 122.5, 125.9, 137.5 ppm.

Results and Discussion

The essential feature of the new concept for protic imidazolium salts consists in attaching polar functional groups to the ring nitrogen atoms. Thus, 1,3-diaminoimidazolium and 1,3-dihydroxyimidazolium triflimides have been recently discovered to be novel protic ionic liquids (PILs) at room temperature [34]. As noticed before, 2-methyl derivatives have a higher tendency to crystallize than unsubstituted imidazolium salts [1, 18, 35]. Hence, we obtained suitable single crystals only of the related 1,3-diamino-2-methylimidazolium and 1,3-dihydroxy-2-methylimidazolium triflimides. The crystallographic data and refinement details are summarized in Table 1.

In the crystal structure of 1,3-diamino-2-methylimidazolium triflimide **1**, all of the ions are ordered (Fig. 2). All hydrogen atoms of the amino groups are involved in contacts to sulfonyl oxygen atoms of three neighboring anions building a three-dimensional network of hydrogen bonds (Fig. 3). Details of the hydrogen bond geometries are given in Table 2. The C–S···S–C torsion angle in the triflimide anion is 171.6°.

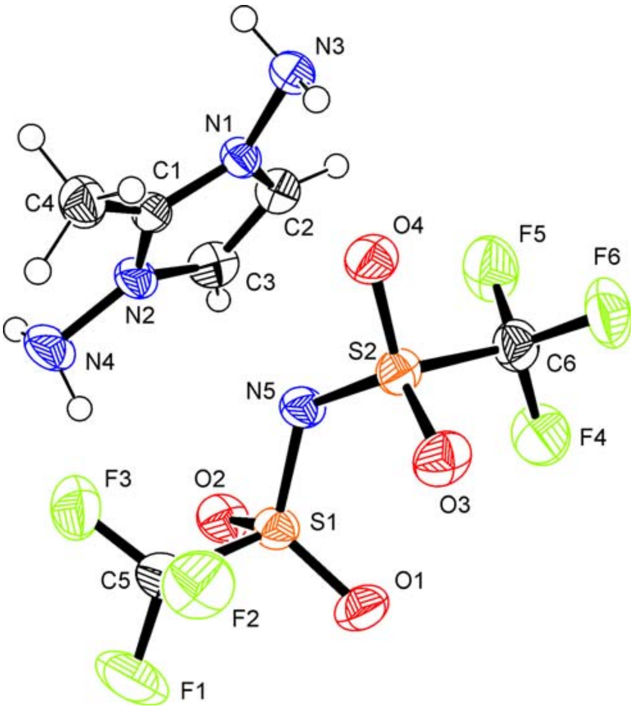


Fig. 2 ORTEP plot of 1,3-diamino-2-methylimidazolium bis(trifluoromethylsulfonyl)imide (**1**) showing 50% probability ellipsoids

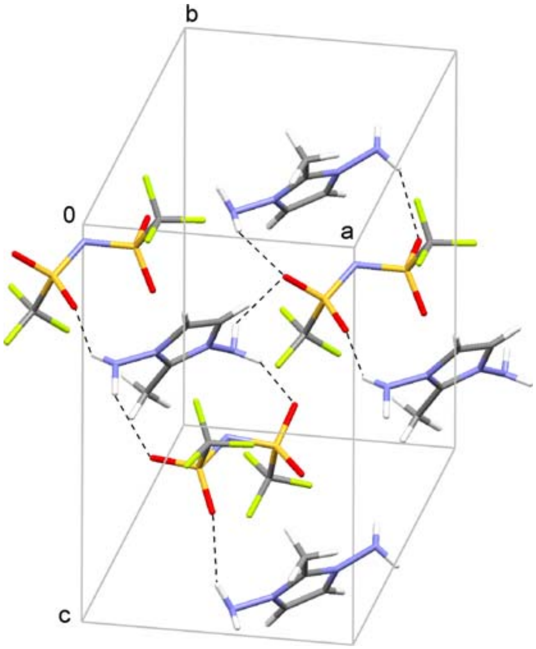


Fig. 3 Network of hydrogen bonds in 1,3-diamino-2-methylimidazolium bis(trifluoromethylsulfonyl)imide (**1**)

Table 2 Hydrogen-bonding geometries in **1**, **2** and **4**

	Interaction	H···*A* (Å)	*D*···*A* (Å)	*D*–H···*A* (°)
1	N3–H···O4	2.30	3.085	149
	N4–H···O2	2.32	3.080	145
	N4–H···O4[a]	2.42	3.105	144
	N3–H···O3[b]	2.27	3.030	145
2	O1–H···N3	1.95	2.751	174
	O2–H···O4[c]	2.24	2.844	133
	O2–H···O5[c]	2.18	2.889	146
4	N3–H···O4	2.04	2.826	163

Symmetry code: [a] 1/2 + *x*, 1/2 − *y*, −1/2 + *z*; [b] −1/2 + *x*, 1/2 − *y*, −1/2 + *z*; [c] 1 + *x*, *y*, 1 + *z*

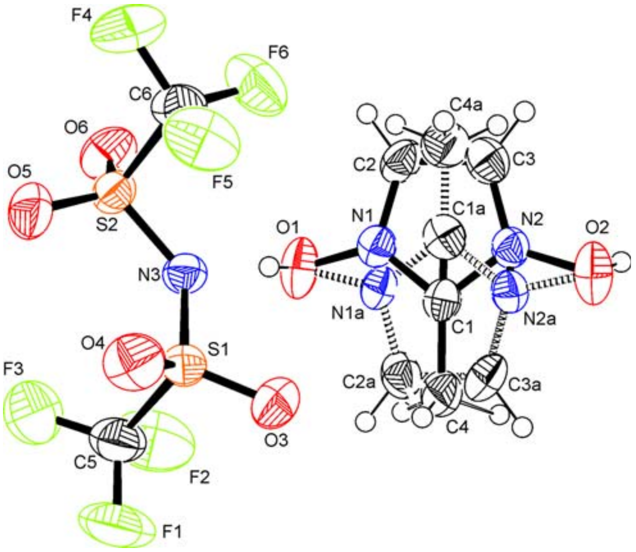


Fig. 4 ORTEP plot of 1,3-dihydroxy-2-methylimidazolium bis(trifluoromethylsulfonyl)imide (**2**) showing 50% probability ellipsoids. The position of the minor component of the disordered imidazolium cation is indicated by the *dashed lines*

The cation in 1,3-dihydroxy-2-methylimidazolium triflimide **2** is disordered, with refined occupancies of 0.55 and 0.45 (Fig. 4). The two components have common oxygen atoms.

Hydrogen atoms at O1 and O2 were found and refined with bond restraints ($d = 0.82$ Å). The anion is almost perfectly antiperiplanar with a torsion angle of 179.4°. The ions are alternately linked into chains by bifurcated hydrogen bonds from a hydroxy group of the cation to two oxygen atoms of adjacent sulfonyl groups and short O–H···N contacts (Fig. 5). This is the first case of a strong interaction of a triflimide nitrogen atom with an organic cation. The hydrogen bond parameters are given in Table 2.

A different kind of protic IL was designed by introduction of an alkyl side chain containing an amino group resulting in RTIL **3**, and further commencing by protonation to the PIL **4**. A similar cation was recently reported in a task-specific IL for the hydrogenation of carbon dioxide [36]. In the structure of 1-(2-(diethylammonio)ethyl)-3-methylimidazolium bis(bis(trifluoromethylsulfonyl)imide) **4** (Fig. 6), two half triflimide anions are located near a symmetry center and display 1:1 disorder of two differently orientated components with shared carbon and sulfur atoms

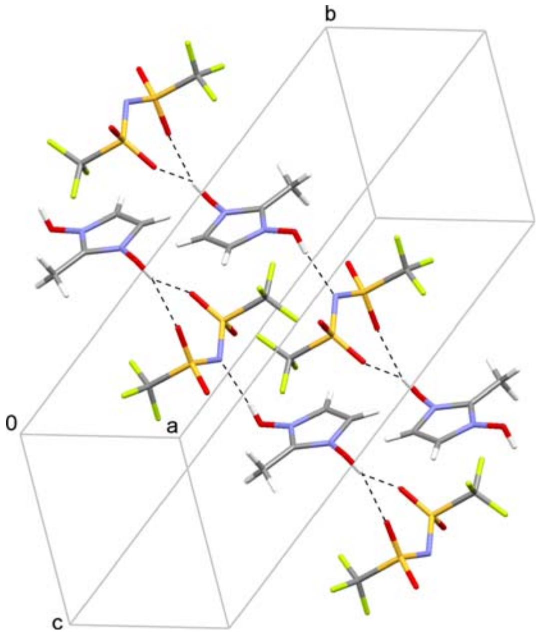


Fig. 5 Hydrogen bonds in 1,3-dihydroxy-2-methylimidazolium bis(trifluoromethylsulfonyl)imide (**2**)

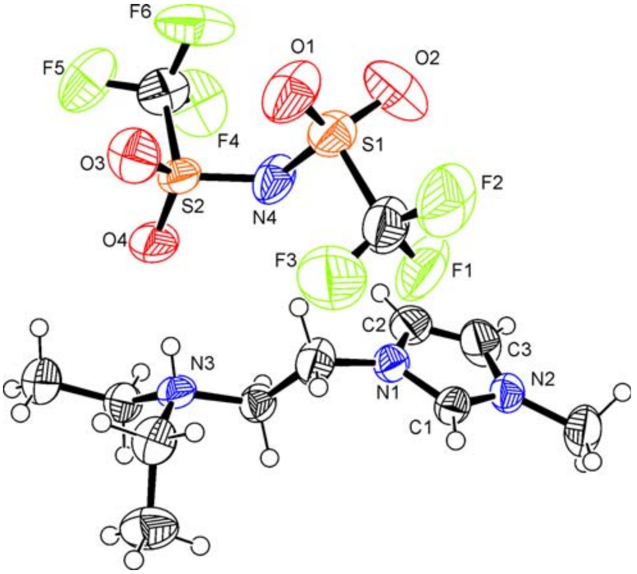


Fig. 6 ORTEP plot of 1-(2-(diethylamino)ethyl)-3-methylimidazolium bis(bis(trifluoromethylsulfonyl)imide) (**4**) showing 50% probability ellipsoids. Disordered anions have been omitted for clarity

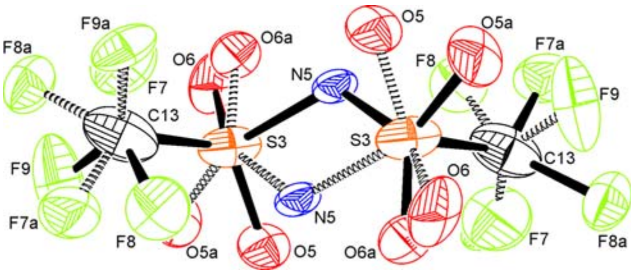


Fig. 7 Anion disorder in 1-(2-(diethylamino)ethyl)-3-methylimidazolium bis(bis(trifluoromethylsulfonyl)imide) (**4**) with 30% probability ellipsoids. The position of the second component is indicated by the *dashed lines*

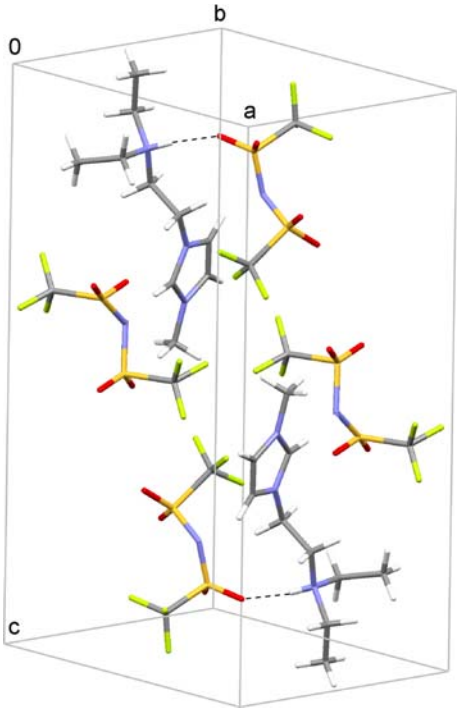
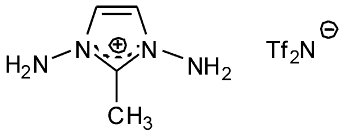


Fig. 8 Short N–H···O contacts in 1-(2-(diethylamino)ethyl)-3-methylimidazolium bis(bis(trifluoromethylsulfonyl)imide) (**4**). Only one component of the disordered anions is shown

(Fig. 7). The ordered anion exhibits a torsion angle of −162.2°. The packing consists of pairs of hydrogen-bonded anions and cations (Fig. 8).

Conclusion

Ironically, crystallography is the most powerful tool for the elucidation of interactions in ionic 'liquids' or low-melting salts. Three markedly different hydrogen bonding architectures were observed in the crystal structures of three new protic imidazolium triflimides. Short interionic contacts, conformational flexibility, and disorder phenomena were identified.



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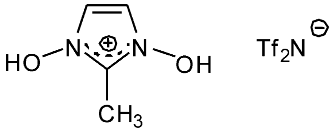

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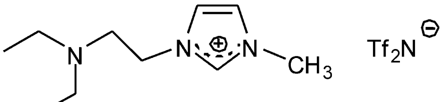

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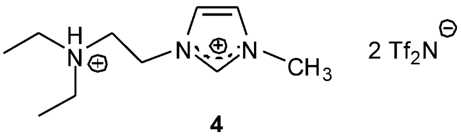

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