

## Lithium triethylammonium bis(trifluoromethanesulfonate)

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## Key indicators

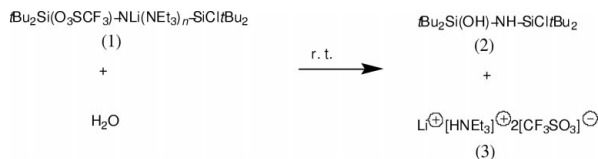
Single-crystal X-ray study  
 T = 173 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
 R factor = 0.029  
 wR factor = 0.058  
 Data-to-parameter ratio = 13.0

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

The title compound,  $\text{Li}^+\cdot\text{HNEt}_3^+\cdot 2\text{CF}_3\text{SO}_3^-$ , is composed of two symmetry-independent trifluorosulfonate anions, an  $\text{Li}^+$  cation and a triethylammonium cation. The Li cation is tetrahedrally coordinated by four O atoms from four different  $\text{CF}_3\text{SO}_3^-$  anions. The triethylammonium cation is bonded *via* an  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond to one of the O atoms that is not coordinating to the Li cation.

## Comment

Recently, we have shown that silanimines can be synthesized by  $\text{CF}_3\text{SO}_3\text{Li}$  elimination from silylamides  $\text{R}_2\text{SiTf}-\text{NLi}-\text{R}'$  (Tf = trifluoromethanesulfonate) (Bolte & Lerner, 2001; Lerner, 1994; Wiberg & Lerner, 1996). Hydrolysis of silylamides  $\text{R}_2\text{SiTf}-\text{NLi}-\text{R}'$  leads to the formation of the silanols  $\text{R}_2\text{SiOH}-\text{NH}-\text{R}'$  and lithium trifluorosulfonate (Lerner, 1994). We report here the X-ray crystal structure analysis of the trifluorosulfonate double salt (3). The synthesis of (3) was achieved by hydrolysis of silylamide (1), as indicated in the *Scheme*.



The asymmetric unit of (3) is composed of two symmetry-independent trifluorosulfonate anions, an  $\text{Li}^+$  cation and a triethylammonium cation. The  $\text{Li}^+$  cation is tetrahedrally coordinated by four O atoms from four different trifluorosulfonate anions. As a result, a ladder-like structure is built up (Fig. 2). The triethylammonium cation is bonded *via* an  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond to one of the O atoms that is not coordinating to the Li cation. The sixth O atom shows no short intermolecular contact.

## Experimental

Water (25  $\mu\text{l}$ ) was added, with stirring at ambient temperature, to a solution of  $\text{tBu}_2\text{Si}(\text{O}_3\text{SCF}_3)-\text{NLi}(\text{NEt}_3)_n-\text{SiCl}t\text{Bu}_2$  (1.42 mmol) in benzene (10 ml). Colourless crystals of (3) were grown by storing this solution at room temperature for several days.

## Crystal data

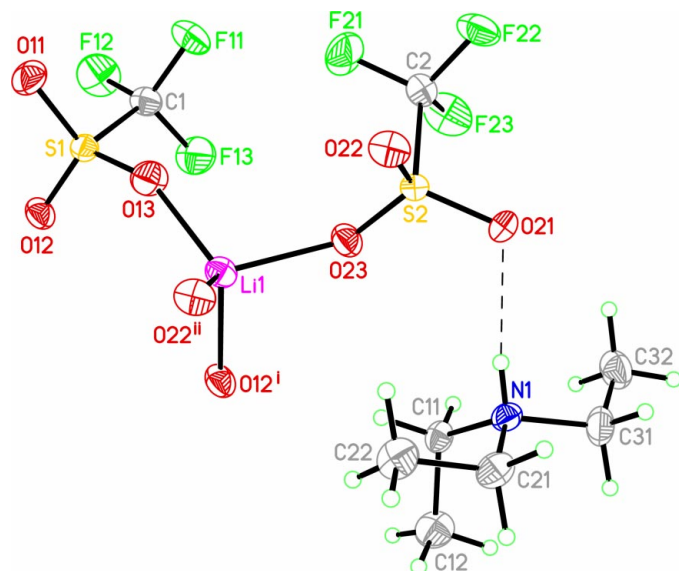
$\text{Li}^+\cdot\text{C}_6\text{H}_{16}\text{N}^+\cdot 2\text{CF}_3\text{SO}_3^-$   
 $M_r = 407.28$   
 Monoclinic,  $P2_1/n$   
 $a = 10.1400$  (13)  $\text{\AA}$   
 $b = 8.6742$  (9)  $\text{\AA}$   
 $c = 19.650$  (2)  $\text{\AA}$   
 $\beta = 100.834$  (9) $^\circ$   
 $V = 1697.5$  (3)  $\text{\AA}^3$   
 $Z = 4$

$D_x = 1.594 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 28862 reflections  
 $\theta = 2.3\text{--}24.7^\circ$   
 $\mu = 0.40 \text{ mm}^{-1}$   
 T = 173 (2) K  
 Block, colourless  
 $0.34 \times 0.28 \times 0.26 \text{ mm}$

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**Figure 1**  
Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

#### Data collection

Stoe IPDS-II two-circle diffractometer	2866 independent reflections
$\omega$ scans	1690 reflections with $I > 2\sigma(I)$
Absorption correction: empirical ( <i>MULABS</i> ; Spek, 1990; Blessing, 1995)	$R_{\text{int}} = 0.045$
$T_{\text{min}} = 0.876$ , $T_{\text{max}} = 0.903$	$\theta_{\text{max}} = 24.7^\circ$
17 018 measured reflections	$h = -11 \rightarrow 11$
	$k = -10 \rightarrow 10$
	$l = -22 \rightarrow 23$

#### Refinement

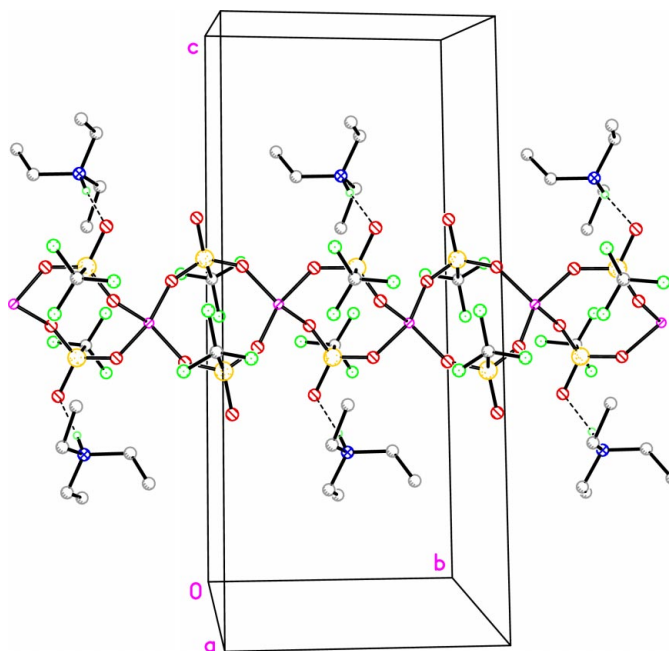
Refinement on $F^2$	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.021P)^2]$
$wR(F^2) = 0.058$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.90$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2866 reflections	$\Delta\rho_{\text{max}} = 0.23 \text{ e } \text{\AA}^{-3}$
221 parameters	$\Delta\rho_{\text{min}} = -0.26 \text{ e } \text{\AA}^{-3}$

**Table 1**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1 \cdots O21$	0.84 (2)	2.04 (3)	2.870 (3)	173 (2)

All H atoms could be located in a difference Fourier synthesis. The H atom bonded to the N atom was refined freely. All others were refined with fixed individual displacement parameters [ $U_{\text{iso}}(\text{H}) =$



**Figure 2**

Packing diagram of the title compound; view on to the  $bc$  plane; only the H atoms bonded to N are shown. Colour code: C shaded black circles, H small green open circles, F dotted green circles, Li pink circles, N blue circles, O red circles, and S yellow circles.

$1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})]$  using a riding model with  $\text{C}-\text{H} = 0.99 \text{ \AA}$  or methyl  $\text{C}-\text{H} = 0.98 \text{ \AA}$ .

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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