

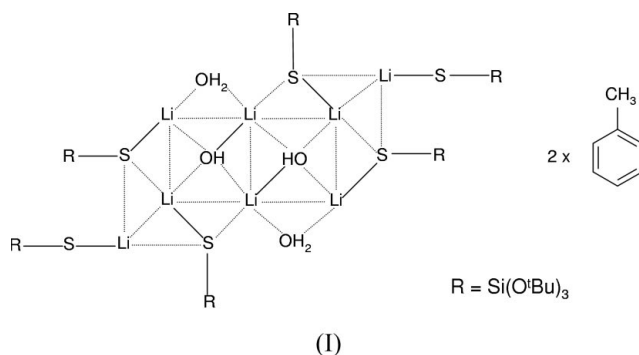
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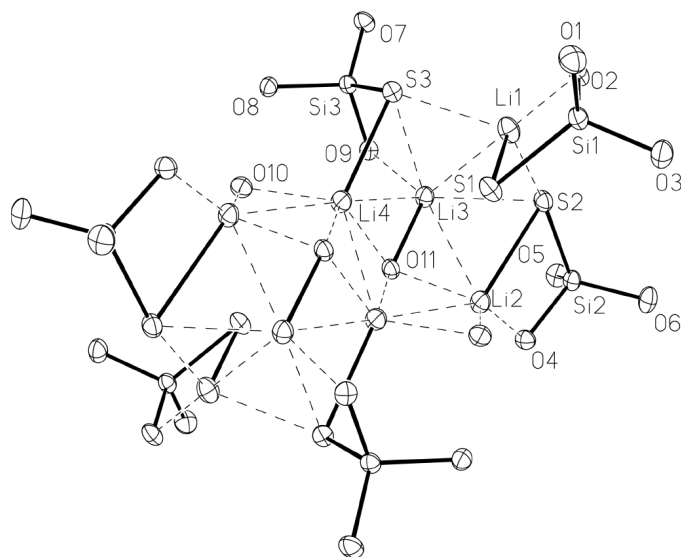
## Key indicators

Single-crystal X-ray study  
*T* = 200 K  
Mean  $\sigma(\text{C}-\text{C})$  = 0.003 Å  
Disorder in solvent or counterion  
*R* factor = 0.036  
*wR* factor = 0.105  
Data-to-parameter ratio = 17.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Diaquadihydroxohexakis(tri-*tert*-butoxysilane-thiolato)octalithium(I)–toluene (1/2)The title compound,  $[\text{Li}_8(\text{C}_{12}\text{H}_{27}\text{O}_3\text{SSi})_6(\text{OH})_2(\text{H}_2\text{O})_2] \cdot 2\text{C}_7\text{H}_8$ , has been obtained by reaction of  $(t\text{BuO})_3\text{SiSH}$  with metallic lithium. The molecule is located on a centre of inversion. The asymmetric unit contains only half a molecule and one disordered toluene molecule.Received 9 September 2002  
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## Comment

Structural studies on thiolates, selenolates and tellurolates of the *s*-block elements have recently been reviewed (Englich & Ruhlandt-Senge, 2000). The number of lithium thiolates cited is about 40 and none of them contain an Li–S–Si fragment. We have attempted to introduce a new type of thiolate ligand,  $(t\text{BuO})_3\text{SiS}^-$ , which, as well as a coordinating S atom, shows chelating properties using O atom(s) (Becker *et al.*, 1996). The title compound, (I), was obtained according to the procedure described in the *Experimental* section. The diaquadihydroxohexakis(tri-*tert*-butoxysilane-thiolato)octalithium(I) core is shown in Fig. 1. The molecule is centrosymmetric. Formally it can be regarded as an adduct of lithium thiolate and lithium hydroxide hydrate. Some similarities to the structure of  $\text{LiOH} \cdot \text{H}_2\text{O}$  (Ojamäe *et al.*, 1994; Hermansson & Thomas, 1982) may be found. Possible sources of  $\text{H}_2\text{O}$  could have been an oxide layer on the metal or traces of water in solvents.Six Li atoms form two edge-sharing rectangles, to which two additional Li atoms are attached terminally. Hydroxyl O atoms lie in positions bridging four Li atoms. Water molecules are bonded to Li atoms as  $\mu_2$  ligands. The two-coordinate atom S1 is in a terminal position, while all the other S atoms are four-coordinate and act as  $\mu_3$  ligands. Eight of the 18 *tert*-butoxy O atoms complete the coordination spheres of the Li atoms. Two disordered [s.o.f. 0.639 (7)/0.361 (7)] toluene molecules are associated with each complex molecule. A disorder model was used for one *tert*-butyl group placed on atom O3. The molecule is a rare example of a lithium thiolate with  $\text{Li} \cdots \text{Li}$  short contacts of *ca.* 2.5 Å, although similar distances can be found in  $\text{LiOH} \cdot \text{H}_2\text{O}$ .



**Figure 1**  
A view of the core of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 25% probability level. All C and H atoms have been omitted.

## Experimental

The work was carried out using standard vacuum–nitrogen line and Schlenk techniques. The title compound was synthesized by direct reaction of a diethyl ether solution of  $(t\text{BuO})_3\text{SiSH}$  (Piekos & Wojnowski, 1962) (2 mmol) with lithium powder. The mixture was stirred magnetically for 2 d. The excess of metal was separated by twofold filtration and the solvent evaporated to dryness. Recrystallization from toluene afforded colourless crystals suitable for X-ray diffraction analysis.

### Crystal data

$[\text{Li}_8(\text{C}_{12}\text{H}_{27}\text{O}_3\text{SSi})_6(\text{OH})_2(\text{H}_2\text{O})_2] \cdot 2\text{C}_7\text{H}_8$	$D_x = 1.083 \text{ Mg m}^{-3}$
$M_r = 1986.7$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 8000 reflections
$a = 13.088 (3) \text{ \AA}$	$\theta = 4.1\text{--}26.1^\circ$
$b = 27.188 (5) \text{ \AA}$	$\mu = 0.23 \text{ mm}^{-1}$
$c = 17.525 (4) \text{ \AA}$	$T = 200 (2) \text{ K}$
$\beta = 102.25 (3)^\circ$	Thick plate, colourless
$V = 6094 (2) \text{ \AA}^3$	$0.6 \times 0.3 \times 0.2 \text{ mm}$
$Z = 2$	

### Data collection

Stoe IPDS diffractometer	$R_{\text{int}} = 0.035$
$\varphi$ scans <i>i.e.</i> $\varphi \omega$ etc	$\theta_{\text{max}} = 25.9^\circ$
47490 measured reflections	$h = -16 \rightarrow 16$
11732 independent reflections	$k = -33 \rightarrow 33$
10031 reflections with $I > 2\sigma(I)$	$l = -21 \rightarrow 21$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 0.8026P]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.105$	$(\Delta/\sigma)_{\text{max}} = 0.03$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
11732 reflections	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$
669 parameters	
H-atom parameters constrained	

**Table 1**

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

Li1–S1	2.449 (3)	S2–Li3	2.433 (2)
Li1–S3	2.514 (3)	Li3–O11	1.926 (2)
Li1–S2	2.541 (3)	Li3–S3	2.647 (2)
Li1···Li3	3.070 (4)	Li3···Li4	2.680 (3)
S1–Si1	2.0546 (6)	S3–Si3	2.0644 (6)
Li2–O11	1.912 (3)	S3–Li4	2.459 (2)
Li2–O10 <sup>i</sup>	1.971 (3)	O11–Li4 <sup>i</sup>	1.953 (2)
Li2–S2	2.579 (2)	O11–Li4	1.959 (3)
Li2···Li4 <sup>i</sup>	2.581 (3)	Li4–O10	2.033 (3)
Li2···Li3	2.673 (3)	Li4···Li4 <sup>i</sup>	2.451 (5)
S2–Si2	2.0649 (7)		
Si1–S1–Li1	75.57 (6)	Li2···Li3···Li4	81.90 (9)
O11–Li2···Li4 <sup>i</sup>	48.78 (8)	Li4 <sup>i</sup> ···Li4···Li2 <sup>i</sup>	88.37 (12)
O11–Li2···Li3	46.09 (8)	Li4 <sup>i</sup> ···Li4···Li3	94.23 (13)
O10 <sup>i</sup> –Li2···Li3	130.04 (12)	Li2 <sup>i</sup> ···Li4···Li3	164.31 (14)
S2–Li2···Li3	55.17 (7)	Li4 <sup>i</sup> ···Li4···Li2	47.33 (9)
Li4 <sup>i</sup> ···Li2···Li3	91.48 (10)		

Symmetry code: (i)  $1 - x, -y, 2 - z$ .

H atoms were located in a difference Fourier maps, then positioned geometrically and allowed to ride on their respective parent atoms.

Data collection: *IPDS* (Stoe & Cie, 1999); cell refinement: *IPDS*; data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *RESVIEW* (Schwenk, 1998); software used to prepare material for publication: *SHELXL97*.

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