Gregory, K., Schleyer, P. v. R. & Snaith, R. (1991). *Adv. Inorg. Chem.* **37**, 47-142.
Hasselbring, R., Pandey, S. K., Roesky, H. W., Stalke, D. & Steiner, A. (1993). *J. Chem. Soc. Dalton Trans.* In the press.
Pandey, S. K. & Roesky, H. W. (1994). *J. Organomet. Chem.* In the press.
Pauer, F. & Stalke, D. (1991). *J. Organomet. Chem.* **418**, 127-145.
Sheldrick, G. M. (1987). *SHELXTL-Plus*. PC Version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467-473.
Sheldrick, G. M. (1994). *J. Appl. Cryst.* In preparation.
Stalke, D., Wedler, M. & Edelmann, F. T. (1992). *J. Organomet. Chem.* **431**, C1-5.
Steiner, A. & Stalke, D. (1993). *Inorg. Chem.* **32**, 1977-1981.
Stoe & Cie (1988*a*). *DIF4. Diffractometer Control Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1988*b*). *REDU4. Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

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# Lithium Tris(trimethylsilyl)silylselenolate Mono(1,2-dimethoxyethane)

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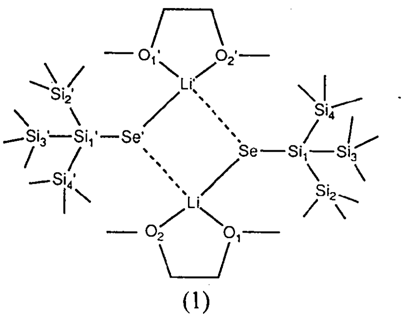
## Abstract

The X-ray crystal structure of (1,2-dimethoxyethane-$\kappa O,O'$)lithium tris(trimethylsilyl)silylselenolate, [Li(C$_4$H$_{10}$O$_2$)][SeSi(C$_3$H$_9$Si)$_3$], was determined and the compound was found to be dimeric in the solid state. Pertinent parameters of the dimer [(1,2-dimethoxyethane)LiSeSi(SiMe$_3$)$_3$]$_2$ are compared to those found in related lithium tellurolates.

## Comment

The solid-state structure of lithium tris(trimethylsilyl)silylselenoate mono(1,2-dimethoxyethane) (1) was determined in order to compare its features with other related silyl chalcogenolates. Compound (1) was synthesized in a manner similar to that described by Bonasia, Gindelberger, Dabbousi & Arnold (1992).



(1)

As for the structure of the analogous compound [(thf)$_2$LiTeSi(SiMe$_3$)$_3$]$_2$ (2), and that of the mono-thf adduct [(thf)LiTeSi(SiMe$_3$)$_3$]$_2$ (3) (Bonasia, Christou & Arnold, 1993), the selenolate was found to be dimeric in the solid state consisting of two asymmetric units related by a center of inversion. The central Li$_2$Se$_2$ four-membered ring in (1) was found to be planar. The Li—Se—Li(i) angle is much more acute than the analagous angle in (2) [90.4 (6)°] but only slightly larger than the Li—Se—Li(i) angle in (3) [70.17 (25)°]. The Li—Se—Li angles in (1) were found to be between similar angles in (2) [123.2 (5), 130.2 (5)°] and in (3) [98.26 (17), 103.47 (17)°], as was the Se—Li—Se(i) angle in (1) [(2) 89.6 (6) and (3) 109.83 (25)°]. The trend observed in these angles might be accounted for by two factors: firstly, the increase in steric interactions caused by the change from a mono-thf adduct to a 1,2-dimethoxyethane (DME) or bis-thf adduct causes an increase in bulk about the Li atom, and, secondly, the decrease in the Li—*E* (*E* = Se, Te) bond length on substitution of Se for Te can effectively bring the bulk of the silyl ligands closer to the bound ethers. In

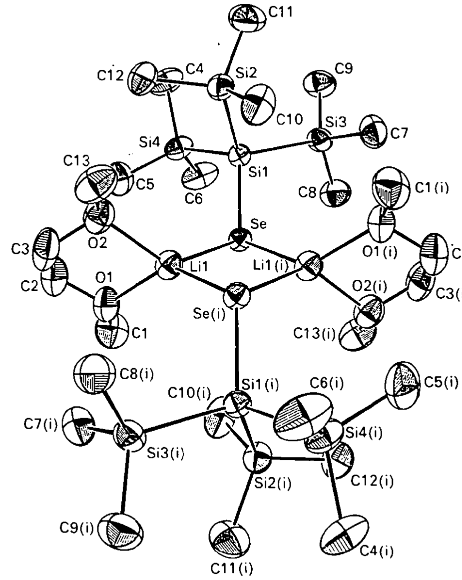


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecular structure of [Li(C$_4$H$_{10}$O$_2$)][SeSi(C$_3$H$_9$Si)$_3$] showing 50% probability displacement ellipsoids and the atom-numbering scheme. The H atoms have been omitted for clarity.

order to relieve the strain imposed by these two factors, the molecule adopts larger *E*—Li—*E*(i) and Li—*E*—Si angles, and in the cases of (2) and (3), a change in bond length is observed (2.85 and 2.743 Å, respectively). The angles about the pseudo-tetrahedral Li atom in (1) range from 81.8 (3) to 129.8 (3)°, the smallest of which is the DME bite angle. The dihedral angle between the plane defined by atoms O1, O2 and Li, and the plane defined by atoms Se, Li and Se(i) is 68.6 (2)°. It appears that this twisting is also primarily due to steric effects, the methyl groups on the DME ligands being pushed away from the bulk of the silyl groups.

## Experimental

### *Crystal data*

$[Li(C_4H_{10}O_2)][SeSi(C_3H_9Si)_3]$
$M_r$ = 423.76
Monoclinic
$P2_1/a$
$a$ = 12.628 (2) Å
$b$ = 14.679 (4) Å
$c$ = 13.644 (3) Å
$\beta$ = 102.25 (2)°
$V$ = 2472 (1) Å$^3$
$Z$ = 4

$D_x$ = 1.139 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\lambda$ = 0.71073 Å
Cell parameters from 40 reflections
$\theta$ = 6.75–14.55°
$\mu$ = 1.69 mm$^{-1}$
$T$ = 183 K
Plates
0.3 × 0.3 × 0.2 mm
Clear

### *Data collection*

Enraf-Nonius CAD-4 diffractometer
$\theta$-2$\theta$ scans
Absorption correction: none
3567 measured reflections
3567 independent reflections
2340 observed reflections $[I < 3\sigma(I)]$

$\theta_{max}$ = 22.5°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 15$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 90 min
intensity variation: 3.2%

### *Refinement*

Refinement on $F$
$R$ = 0.028
$wR$ = 0.030
$S$ = 1.129
2340 reflections
190 parameters

Weights: non-poisson contribution
$\Delta\rho_{max}$ = 0.397 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.85 e Å$^{-3}$
Atomic scattering factors from Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| Se | 0.37365 (1) | 0.44566 (1) | 0.40007 (1) | 2.501 (7) |
| Si(1) | 0.30818 (8) | 0.50411 (7) | 0.24462 (7) | 2.51 (2) |
| Si(2) | 0.38068 (8) | 0.64790 (7) | 0.22284 (8) | 3.26 (2) |
| Si(3) | 0.35538 (9) | 0.40269 (8) | 0.12832 (8) | 3.48 (3) |
| Si(4) | 0.11975 (8) | 0.52042 (8) | 0.21755 (9) | 3.55 (3) |
| Li | 0.4155 (5) | 0.5635 (4) | 0.5424 (5) | 3.2 (1) |
| O(1) | 0.2952 (2) | 0.5565 (2) | 0.6181 (2) | 4.13 (6) |
| O(2) | 0.3789 (2) | 0.6985 (2) | 0.5413 (2) | 4.37 (7) |
| C(1) | 0.2249 (3) | 0.4817 (3) | 0.6245 (3) | 5.4 (1) |
| C(2) | 0.2399 (3) | 0.6393 (3) | 0.6153 (3) | 5.1 (1) |
| C(3) | 0.3206 (4) | 0.7147 (3) | 0.6175 (3) | 5.3 (1) |
| C(4) | 0.0686 (3) | 0.5972 (3) | 0.1081 (3) | 5.3 (1) |
| C(5) | 0.0828 (3) | 0.5703 (3) | 0.3318 (4) | 5.8 (1) |
| C(6) | 0.0470 (3) | 0.4092 (3) | 0.1903 (4) | 5.8 (1) |
| C(7) | 0.5039 (3) | 0.4108 (3) | 0.1354 (3) | 5.3 (1) |
| C(8) | 0.3235 (4) | 0.2839 (3) | 0.1614 (3) | 5.1 (1) |
| C(9) | 0.2823 (4) | 0.4252 (3) | −0.0038 (3) | 5.1 (1) |
| C(10) | 0.5270 (3) | 0.6485 (3) | 0.2866 (4) | 5.2 (1) |
| C(11) | 0.3689 (4) | 0.6732 (3) | 0.0867 (3) | 5.8 (1) |
| C(12) | 0.3104 (3) | 0.7413 (3) | 0.2763 (3) | 4.2 (1) |
| C(13) | 0.4572 (4) | 0.7680 (3) | 0.5383 (4) | 5.4 (1) |

Table 2. *Selected geometric parameters* (Å, °)

Atoms labelled with (i) are in the second asymmetric unit of the dimer.

| | | | |
|---|---|---|---|
| Se—Li | 2.570 (6) | Si(2)—C(12) | 1.864 (4) |
| Se—Li(i) | 2.614 (6) | Si(3)—C(7) | 1.861 (5) |
| Se(i)—Li | 2.570 (6) | Si(3)—C(8) | 1.866 (4) |
| Se—Si(1) | 2.275 (1) | Si(3)—C(9) | 1.872 (4) |
| Li—O(1) | 2.013 (7) | Si(4)—C(4) | 1.872 (4) |
| Li—O(2) | 2.034 (7) | Si(4)—C(5) | 1.868 (5) |
| Si(1)—Si(2) | 2.345 (1) | Si(4)—C(6) | 1.872 (5) |
| Si(1)—Si(3) | 2.343 (1) | O(2)—C(3) | 1.415 (5) |
| Si(1)—Si(4) | 2.342 (1) | O(1)—C(1) | 1.426 (5) |
| Si(2)—C(10) | 1.868 (4) | O(1)—C(2) | 1.399 (5) |
| Si(2)—C(11) | 1.869 (5) | O(2)—C(13) | 1.428 (5) |
| Se—Li—O(1) | 107.2 (3) | O(1)—Li—Se(i) | 105.5 (3) |
| Se—Li—O(2) | 129.8 (3) | Si(1)—Se—Li | 115.2 (1) |
| Se—Li—Se(i) | 103.0 (2) | Si(1)—Se—Li(i) | 116.41 (14) |
| O(1)—Li—O(2) | 81.8 (3) | Li—Se—Li(i) | 77.0 (2) |
| O(1)—Li—Se(i) | 132.5 (3) | | |

The structure was solved using the heavy-atom method with the heavy atoms identified from a Patterson map with isotropic refinement. Subsequently Se, Si, then all non-H atoms were refined anisotropically. All H atoms were assigned ideal positions but not refined. All calculations were carried out using *MolEN* (Fair, 1990). Data collection and cell refinement were carried out on a DEC MicroVAX II controlled diffractometer using *CAD-4 Software* (Enraf-Nonius, 1977). Data reduction, structure solution and structure refinement used *MolEN* and *SDP* (Frenz, 1985).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry including H-atom geometry, least-squares-planes data and r.m.s. amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71479 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1075]

## References

Bonasia, P. J., Christou, V. & Arnold, J. (1993). *J. Am. Chem. Soc.* In the press.

Bonasia, P. J., Gindelberger, D. E., Dabbousi, B. O. & Arnold, J. (1992). *J. Am. Chem. Soc.* **114**, 5209-5214.

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf-Nonius, Delft, The Netherlands.

Frenz, B. A. (1985). *Structure Determination Package. SDP/PDP User's Guide*. Version 3.0. Enraf-Nonius, Delft, The Netherlands.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.