# Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

## $U_{\rm eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_i^* \mathbf{a}_i \cdot \mathbf{a}_j.$

	x	у	z	$U_{co}$
Col	0	0.05959 (6)	1/4	0.02601 (15)
Cl2	0.15071 (6)	0.20470 (9)	0.32712 (4)	0.0386 (2)
Cll	-0.05398 (6)	-0.09866 (9)	0.33682 (4)	0.0395 (2)
NI	0.1342 (2)	0.3240 (3)	0.12121 (12)	0.0288 (5)
N3	0.1072 (2)	0.1459 (3)	0.01372 (13)	0.0283 (5)
C4	0.2122 (2)	0.0804 (3)	0.04387 (14)	0.0272 (6)
C6	0.2395 (2)	0.2592 (4)	0.1537 (2)	0.0324 (6)
C2	0.0621 (2)	0.2646 (3)	0.05027 (15)	0.0290 (6)
C5	0.2805 (2)	0.1406 (4)	0.1189 (2)	0.0332 (6)
02	-0.0347(2)	0.3119 (3)	0.01993 (12)	0.0432 (5)
N4	0.2457 (2)	-0.0294(3)	0.0025 (2)	0.0365 (6)
CL	0.0951 (3)	0.4617 (4)	0 1587 (2)	0.0305 (7)

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

Co1-Cl2 Co1-Cl1 N1-C6 N1-C2 N1-C1 N3-C4	2.2628 (10) 2.2791 (8) 1.359 (3) 1.375 (3) 1.464 (4) 1.355 (3)	N3-C2 C4-N4 C4-C5 C6-C5 C2-O2	1.383 (4) 1.309 (4) 1.416 (4) 1.335 (4) 1.217 (3)
C12-C01-C12'  C12-C01-C11  C12'-C01-C11  C11-C01-C11'  C6-N1-C2  C6-N1-C1  C2-N1-C1  C4-N3-C2  C4-N3-C2	117.62 (5) 104.73 (3) 109.05 (3) 111.80 (5) 120.6 (2) 121.6 (2) 117.8 (2) 125.5 (2)	$\begin{array}{c} N4-C4-N3 \\ N4-C4-C5 \\ N3-C4-C5 \\ C5-C6-N1 \\ O2-C2-N1 \\ O2-C2-N3 \\ N1-C2-N3 \\ C6-C5-C4 \end{array}$	119.5 (2) 124.0 (3) 116.4 (2) 123.3 (2) 123.8 (3) 120.9 (2) 115.3 (2) 118.7 (2)
C2-N3-C4-N4 C2-N3-C4-C5 C2-N1-C6-C5 C1-N1-C6-C5 C6-N1-C2-O2 C1-N1-C2-O2 C6-N1-C2-N3	$\begin{array}{r} -178.7 (3) \\ -0.4 (4) \\ 2.1 (4) \\ -175.8 (3) \\ 176.5 (3) \\ -5.5 (4) \\ -4.1 (4) \end{array}$	$\begin{array}{c} C1 - N1 - C2 - N3 \\ C4 - N3 - C2 - O2 \\ C4 - N3 - C2 - N1 \\ N1 - C6 - C5 - C4 \\ N4 - C4 - C5 - C6 \\ N3 - C4 - C5 - C6 \end{array}$	173.9 (2 - 177.3 (3 3.3 (4 1.1 (4 176.4 (3 - 1.9 (4
		··· ··· · · · · · · · · · · · · · · ·	

Data collection: P3/PC Diffractometer Program (Siemens, 1989). Cell refinement: P3/PC Diffractometer Program. Data reduction: SHELXTL-Plus XDISK (Sheldrick, 1987). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL (Sheldrick, 1994). Molecular graphics: SHELXTL-Plus. Software used to prepare material for publication: SHELXL. Absorption coefficients were taken from International Tables for Crystallography (1992, Vol. C). The space group was checked with MISSYM (Le Page, 1987).

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#### References

Campayo, L., Navarro, P., Cano, F. H. & Foces-Foces, C. (1987). J. Chem. Soc. Perkin Trans. 2, pp. 569–573.
 Le Page, Y. (1987). J. Appl. Cryst. 20, 264–269.

Lindroos, S. & Lumme, P. (1991). *Acta Cryst.* C47, 872-874. Rossi, M., Caradonna, J. P., Marzilli, L. G. & Kistenmacher, T. J. (1979).

Adv. Mol. Relaxation Interact. Processes, **15**, 103-117. Sheldrick, G. M. (1987). SHELXTL-Plus. Release 3.4 for Nicolet R3m/V crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Sheldrick, G. M. (1990). Acta Cryst. A46, 467-473.

Sheldrick, G. M. (1994). J. Appl. Cryst. In preparation.

Siemens (1989). P3/PC Diffractometer Program. Version 3.13. Siemens Analytical Instruments Inc., Madison, Wisconsin, USA.

Søtofte, I. & Nielsen, K. (1981). Acta Chem. Scand. Ser. A, 35, 739-745. Taylor, R. & Kennard, O. (1982). J. Mol. Struct. 78, 1-28.

Acta Cryst. (1994). C50, 671-674

# $[Li{N(Me_3SiNPPh_2)_2}]_2.2.5C_7H_8$

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#### Abstract

The structure of the title compound bis{ $\mu$ -*P*,*P'*-imido-1:2 $\kappa^2 N''$ -bis[*P*,*P*-diphenyl-*N*-(trimethylsilyl)phosphine imido](1-)-1 $\kappa$ *N*, 1 $\kappa$ *P*:2 $\kappa$ *N'*, 2 $\kappa$ *P'*}dilithium(*Li*—*Li*), [Li{N[(CH<sub>3</sub>)<sub>3</sub>SiNP(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>]<sub>2</sub>}]<sub>2</sub>.2.5C<sub>7</sub>H<sub>8</sub> is reported. There is one molecule of the dimer in the asymmetric unit, two disordered molecules of the toluene solvent close to a centre of symmetry and another disordered solvent molecule situated on a twofold axis.

#### Comment

Organolithium compounds have received increasing attention in the past few years (see Gregory, Schleyer & Snaith, 1991, for a recent review), especially the amidolithium compounds ( $R_2$ NLi), mainly because of their use as proton abstractors in both organic and inorganic syntheses. The title compound, (I) (Pandey & Roesky, 1994; Steiner & Stalke, 1993), is a derivative of the monomer [{Ph<sub>2</sub>P(Me<sub>3</sub>SiN)<sub>2</sub>}Li(thf)<sub>2</sub>], and is stabilized by a butterfly-like ligand conformation with pseudononcrystallographic  $C_2$  symmetry and an Li—Li distance of 2.79 (1) Å.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71841 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1122]

# $[Li{N[(CH_3)_3SiNP(C_6H_5)_2]_2}]_2.2.5C_7H_8$

Lil

NI

Si 1 Cl

C2

C3 **P**1

C4

C5

C6 C7

C8

C9

C10 CII

C12

C13 C14

C15

N2 P2 C16 C17

C18

C19 C20

C21

C22 C23 C24

C25 C26 C27 N3 Si2 C28 C29





Fig. 1. Plot of (I), showing 50% probability displacement ellipsoids and atomic labelling.

The full-matrix anisotropic refinement finally converged to R = 0.056. The refinement of the disordered solvent molecules was carried out by means of a constraintrestraint model, refining the occupation factors of the two components along the twofold axis and the other two components close to a centre of symmetry as p and 1-p.

The mean values of the distances and angles for the Li environment are: Li-N(Si) 1.99 (1), Li-N(P) 2.25 (5), Li—P 2.64 (1) Å, (Si)N—Li—N(Si) 140 (4), (P)N—Li— N(P) 103 (3), (P)N-Li-N(Si) 74 (1) and (Si)N-Li-N(P) 134 (6)°, showing distorted square-planar coordination of the Li atoms, in good agreement with other published results for similar lithium-ring compounds (Pauer & Stalke, 1991; Edelmann, Knösel, Pauer & Stalke, 1992; Stalke, Wedler & Edelmann, 1992; Hasselbring, Pandey, Roesky, Stalke & Steiner, 1993).

 $D_x = 1.192 \text{ Mg m}^{-3}$ 

#### **Experimental**

# Crystal data $[Li_2(C_{30}H_{38}N_3P_2Si_2)_2]$ .-2.5C7H8

$2.5C_7H_8$	Mo $K\alpha$ radiation
$M_r = 1361.72$	$\lambda = 0.71073 \text{ Å}$
Monoclinic	Cell parameters from 60
C2/c	reflections
a = 51.814 (10) Å	$\theta = 20-25^{\circ}$
b = 12.232 (2) Å	$\mu = 0.208 \text{ mm}^{-1}$
c = 23.984 (5) Å	T = 187 (2) K
$\beta = 92.91 (3)^{\circ}$	Transparent needles

$V = 15181.2 (50) \text{ Å}^3$ Z = 8	$0.2 \times 0.2 \times 0.1 \text{ mm}$ Colourless
Data collection	
Stoe Siemens AED four- circle diffractometer Profile data from $2\theta/\omega$ scans Absorption correction: empirical $T_{min} = 0.761, T_{max} =$ 0.779 8169 measured reflections 7038 independent reflections	5113 observed reflections $[I > 2\sigma(I)]$ $R_{int} = 0.0235$ $\theta_{max} = 20.00^{\circ}$ $h = -49 \rightarrow 49$ $k = -11 \rightarrow 11$ $l = -3 \rightarrow 23$ 3 standard reflections frequency: 90 min intensity variation: 3%
Refinement	
Refinement on $F^2$ R(F) = 0.0557 $wR(F^2) = 0.1343$ S = 1.079 7037 reflections 908 parameters	$(\Delta/\sigma)_{\text{max}} = -0.237$ $\Delta\rho_{\text{max}} = 0.325 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.476 \text{ e } \text{\AA}^{-3}$ Extinction correction: none Atomic scattering factors from International Tables
Calculated weights	for Crystallography (1992,

 $w = 1/[\sigma^2(F_o^2) + (0.0712P)^2]$ 

+ 69.5000P] where  $P = (F_o^2 + 2F_c^2)/3$ 

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

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6.1.1.4)

## $U_{\rm eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_i^* \mathbf{a}_i \cdot \mathbf{a}_j.$

x	v	Z	Uea
0.1327 (2)	0.2765 (7)	-0.0504 (4)	0.034 (2)
0.12539 (8)	0.1560 (3)	-0.1061 (2)	0.0289 (11)
0.14541 (3)	0.09058 (14)	-0.14945 (7)	0.0403 (5)
0.14356 (12)	0.1440 (6)	-0.2220(2)	0.059 (2)
0.13916 (14)	-0.0591 (5)	-0.1549 (3)	0.067 (2)
0.17912 (11)	0.1120 (6)	-0.1206 (3)	0.059 (2)
0.09536 (3)	0.17637 (11)	-0.10836 (6)	0.0278 (4)
0.08305 (10)	0.2148 (4)	-0.1775 (2)	0.0278 (13)
0.08927 (10)	0.3176 (5)	-0.1964 (2)	0.0348 (15)
0.08091 (11)	0.3539 (5)	-0.2488(2)	0.041 (2)
0.06627 (12)	0.2856 (5)	-0.2835 (2)	0.048 (2)
0.06037 (12)	0.1824 (5)	-0.2657 (2)	0.047 (2)
0.06894 (10)	0.1457 (5)	-0.2135 (2)	0.0359 (15)
0.07783 (11)	0.0545 (4)	-0.0885 (2)	0.0303 (14)
0.05142 (12)	0.0401 (5)	-0.0994(2)	0.042 (2)
0.03910 (13)	-0.0530 (5)	-0.0812 (3)	0.052 (2)
0.0529 (2)	-0.1314 (5)	-0.0523 (3)	0.056 (2)
0.0789 (2)	-0.1173 (5)	0.0407 (3)	0.056 (2)
0.09142 (12)	-0.0253 (5)	-0.0584 (2)	0.042 (2)
0.09037 (8)	0.2745 (3)	-0.0649 (2)	0.0271 (11)
0.06680 (3)	0.35724 (11)	-0.05332 (6)	0.0263 (4)
0.03608 (10)	0.3015 (4)	-0.0799 (2)	0.0286 (14)
0.02724 (10)	0.3053 (4)	-0.1356 (2)	0.0331 (14)
0.00418 (11)	0.2561 (5)	-0.1529 (3)	0.042 (2)
-0.01005 (11)	0.2002 (5)	-0.1150 (3)	0.046 (2)
-0.00152 (11)	0.1953 (5)	-0.0597 (3)	0.042 (2)
0.02125 (10)	0.2463 (4)	-0.0426 (2)	0.0327 (14)
0.07081 (10)	0.4833 (4)	-0.0917 (2)	0.0275 (13)
0.05019 (11)	0.5432 (5)	-0.1141 (2)	0.038 (2)
0.05401 (13)	0.6413 (5)	-0.1413 (2)	0.048 (2)
0.07847 (13)	0.6814 (5)	-0.1452 (2)	0.046 (2)
0.09929 (12)	0.6251 (5)	-0.1214 (2)	0.042 (2)
0.09543 (11)	0.5267 (5)	-0.0949 (2)	0.0338 (15)
0.06724 (7)	0.3759 (3)	0.0113 (2)	0.0267 (11)
0.05441 (3)	0.46368 (13)	0.05685 (6)	0.0321 (4)
0.01974 (11)	0.4358 (5)	0.0688 (2)	0.050(2)
0.07283 (11)	0.4460 (5)	0.1248 (2)	0.042(2)

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C30	0.05614 (12)	0.6084 (4)	0.0338 (2)	0.047 (2)	Li1—P1	2.627 (9)	Li2—N5	2.302 (9)
L12	0.0956 (2)	0.2712 (7)	0.0297 (4)	0.034 (2)	Li1—P4	2.632 (9)	Li2—P3	2.656 (9)
N4	0.10479 (7)	0.1664 (3)	0.0911 (2)	0.0280 (11)	Li1—Li2	2.786 (12)		
Si3	0.08415 (3)	0.07679 (13)	0.11976 (6)	0.0345 (4)	N6 131 N1	144 4 (5)	N3 132 N4	126 6 (5)
C31	0.05188 (11)	0.0954 (5)	0.0834 (3)	0.055 (2)		1791(5)	N3 L12 N2	72 5 (2)
C32	0.09356 (12)	-0.0684 (4)	0.1112 (3)	0.047 (2)		746(3)	NJ-LIZ-NZ	13.3 (3)
C33	0.08087 (13)	0.1024 (5)	0.1961 (2)	0.056 (2)		74.0 (3)	N4-LIZ-NZ	139.0 (3)
P3	0.13464 (3)	0.18522 (11)	0.09106 (6)	0.0273 (4)		74.5 (5) 120.6 (5)	NJ-LIZ-NJ	139.1 (3)
C34	0.15169 (10)	0.0585 (4)	0.0794 (2)	0.0281 (14)	NI-LII-NS	130.6 (5)	N4-L12-N5	72.9(3)
C35	0.15485 (11)	0.0212 (5)	0.0255 (2)	0.039 (2)	N2-LII-N5	106.0 (4)	N2—L12—N5	101.0 (4)
C36	0.16523 (12)	-0.0806(5)	0.0160 (3)	0.051 (2)	N6-LII-PI	153.7 (5)	N3—Li2—P2	36.1 (2)
C37	0.17243 (12)	-0.1473 (5)	0.0603 (3)	0.053 (2)	NI-LII-PI	36.7 (2)	N4-Li2-P2	156.5 (5)
C38	0.16956 (11)	-0.1117(5)	0.1141 (3)	0.048 (2)	N2-L11-P1	37.9 (2)	N2—Li2—P2	37.5 (2)
C39	0.15912 (11)	-0.0103(5)	0.1237 (2)	0.039 (2)	N5Li1P1	126.4 (4)	N5—Li2—P2	128.1 (4)
C40	0.14810(11)	0.2324 (4)	0.1578 (2)	0.0301 (14)	N6—Li1—P4	36.6 (2)	N3—Li2—P3	156.1 (4)
C41	0.13173 (12)	0.2814 (5)	0.1947 (2)	0.039 (2)	N1—Li1—P4	155.9 (4)	N4—Li2—P3	35.8 (2)
C42	0.14120 (15)	0.3210(5)	0.2456 (3)	0.052(2)	N2—Li1—P4	125.4 (4)	N2—Li2—P3	127.5 (4)
C43	0.1670 (2)	0.3117 (5)	0.2602 (3)	0.061(2)	N5-Li1-P4	37.9 (2)	N5—Li2—P3	37.1 (2)
C44	0.18367 (13)	0 2631 (5)	0.2262(3)	0.052(2)	P1—Li1—P4	160.6 (4)	P2-Li2-P3	163.5 (4)
C45	0.17410(12)	0 2236 (4)	0.1733(2)	0.032(2)	N6—Li1—Li2	106.3 (4)	N3—Li2—Li1	111.1 (4)
N5	0 12000 (7)	0.2200 (1)	0.1755(2)	0.0762(11)	N1—Li1—Li2	109.2 (4)	N4—Li2—Li1	112.2 (4)
D/	0.13999(7) 0.15905(3)	0.2761(3)	0.0410(2)	0.0203(11)	N2—Li1—Li2	52.6 (3)	N2—Li2—Li1	50.4 (3)
Г4 С46	0.13803(3)	0.37041(11)	0.05271(0)	0.0277(4)	N5—Li1—Li2	53.3 (3)	N5—Li2—Li1	50.7 (3)
C40	0.19110 (10)	0.3308 (3)	0.0331(2)	0.0324(14)	P1—Li1—Li2	80.8 (3)	P2—Li2—Li1	81.6 (3)
C47	0.20210(11)	0.2500 (5)	0.0443(2)	0.040(2)	P4—Li1—Li2	79.8 (3)	P3-Li2-Li1	81.9 (3)
C48	0.22810(13)	0.2309(0)	0.0557(3)	0.057(2)	<b>TT</b> 0			•
C49	0.24328 (13)	0.3118 (8)	0.0780(3)	0.0/4 (2)	The refinement o	t the disorde	red solvent mol	ecules was car-
C50	0.23313 (13)	0.4132(7)	0.0883 (3)	0.069 (2)	ried out using a	constraint-res	straint model. T	he two rings of
C51	0.20719(12)	0.4327 (6)	0.0770(3)	0.051 (2)	the toluene mole	cules close to	a centre of sv	mmetry, one of
C52	0.14/6/(10)	0.4893 (4)	0.0757(2)	0.0298 (14)	them having two		for a ventile of of	0.50(1) and
C53	0.13221 (10)	0.5703 (5)	0.0512(2)	0.0359 (15)	them having two	components		ors 0.39 (1) and
C54	0.12343 (11)	0.6552 (5)	0.0833 (3)	0.044 (2)	0.41(1), were re	fined constra	ining them to re	gular hexagons,
C55	0.12972 (12)	0.6608 (5)	0.1396(3)	0.045 (2)	restraining planar	ty and chemi	cally equivalent	1,3 and 1,4 dis-
C56	0.14506 (12)	0.5808 (5)	0.1644 (2)	0.040 (2)	tances to be equa	for the Me	group and restr	aining displace-
C5/	0.15389 (11)	0.4963 (5)	0.1326 (2)	0.0378 (15)	mont personators	long the hor	d directions for	all the C storms
N6	0.15552 (7)	0.4045 (3)	-0.0314(2)	0.0285 (11)				an me c atoms.
514	0.1/384(3)	0.4/3/9(13)	-0.07636(6)	0.0359 (4)	The toluene mole	cule along th	e twofold axis, a	also having two
C58	0.20821 (11)	0.4297 (6)	-0.0/48(3)	0.062 (2)	components [occu	pation factor	s 0.68 (3) and 0.	.32 (3)], was re-
C39	0.17392(13)	0.0243(5)	-0.0628 (3)	0.056 (2)	fined restraining d	isplacement r	parameters along	the bond direc-
C60	0.15988 (12)	0.4445 (5)	-0.1481 (2)	0.047(2)	tions and chemica	lly equivalent	1213and14	distances for all
C61	0	0.8337 (09)	- 1/4	0.280 (21)	the C stome The	U stoms work	a second with a s	ulsunees for an
C62	0 0161 (12)	0.7340 (30)	- 1/4	0.204 (14)	the Catolins. The	n atoms were	e renned with a r	lang model for
C03	-0.0101(13)	0.0020 (34)	-0.2208 (32)	0.202(14)	the positional para	meters and w	ith a common isc	stropic displace-
C64	-0.0104(7)	0.5755 (54)	-0.2221 (18)	0.198 (14)	ment parameter fo	r the methyl l	H atoms and a di	fferent common
C61/	0	0.3233(30)	- 1/4	0.202(13)	isotropic factor fo	r the nhenvl H	H atoms	
C62'	0	0.9237 (39)	- 1/4	0.308 (19)	Data collection	and call r	finamant wara	norformed up
C62	0.0141.(4)	0.8224 (31)	-1/4	0.220(12)				performed us-
C64'	-0.0141(4)	0.7722 (20)	-0.2102(9)	0.200(12)	ing DIF4 (Stoe	& Cie, 198	(88 <i>a</i> ). Data red	uction was by
C65'	-0.0140(0)	0.0003 (23)	-0.2170(14)	0.166(12) 0.188(12)	REDU4 (Stoe &	Cie, 1988	b). The structu	re was solved
C66	0 2233 (3)	-0.0164(14)	-1/4 0.2136(7)	0.188(12) 0.334(12)	with SHELXS86 (	Sheldrick, 19	990) and refined	with SHELXL
C67	0.2233(3)	-0.0104(14)	0.2150(7)	0.334(12)	(Sheldrick 1994)	Molecular	graphics were	prepared using
C68	0.2494(2) 0.2484(2)	0.0350(10)	0.2232(3)	0.240(0)	CULLYTI DL (C		graphics were	richared using
C60	0.2404 (2)	0.1300 (10)	0.1336 (4)	0.213 (0)	SHELAIL-Plus (S	neidrick, 198	() and the mate	rial for publica-
C70	0.2700 (3)	0.1500 (3)	0.2130(3)	0.246 (0)	tion was prepared	with SHELX	L.	
C71	0.2888 (2)	0.1550 (11)	0.2328 (3)	0.220 (0)				
C72	0.2677(2)	0.0332(11)	0.2782(4)	0.208 (6)	This was to see		handha Daardaal	
C73	0.2027(3)	-0.0048(3)	0.2044(3)	0.236 (0)	This work was	supported	by the Deutsch	e Forschungs-
C74	0.2370 (4)	0.1470 (21)	0.3110(10)	0.210(10)	gemeinschaft an	d the Fond	s der Chemisc	hen Industrie.
C75	0.2370 (4)	0.1377 (10)	0.4008 (7)	0.237(10)	One of the auth	ors (IEVD)	(III) would like	e to thank the
C76	0.2226 (3)	0.1338 (13)	0.4223 (9)	0.243(9)				
C77	0.2230 (4)	0.1639 (19)	0.3092 (8)	0.230(10)	Universidad de	Oviedo (Spa	ain) for a shor	t-stay grant in
C78	0.2500 (4)	0.2737(17)	0.3002 (0)	0.240 (9)	Göttingen and G	eorge Sheld	rick's Crystallo	graphy Group
C70	0.2320 (4)	0.3237 (13)	0.4045(11)	0.243 (9)	for their haln du	ring this star	- <b>j</b> = <b>i</b> - <b>c</b>	0 P
C73'	0.2320 (4)	0.2750 (10)	0.4378 (9)	0.240 (10)	tor men help du	ing this stay	y.	
C7A'	0.2374(7)	0.2251 (33)	0.4095 (10)	0.290 (14)				
C75'	0.2791(3)	0.1072 (23)	0.3039(13)	0.311 (13)	Lists of structure fa	ctors, anisotror	oic displacement n	arameters. H-atom
C76'	0.2272 (4)	0.1273 (24)	0.3310(13)	0.310(13)	coordinates and com	nlete geometr	have been denosit	ted with the British
C77'	0.2550(0)	0.0047 (21)	0.2017(14)	0.270 (14)	Library Document	Supply Centre	as Supplementar	v Publication No.
C78'	0.2780 (5)	0 1592 (25)	0 3027 (14)	0.302(15)	SUP 71712 (32 nn	) Coniec may	he obtained through	igh The Technicol
C79'	0 2696 (5)	0 2041 (23)	0.3517(14)	0.330 (16)	Editor Internetional	J. Copies may		agn the rechindan
2.7	0.2070 (0)	0.2011 (20)	0.5517 (17)	0.000 (10)	Eunor, international	ULLOID OF CITYS	unograpny, 5 ADD	cy square, Unester

Table 2. Selected geometric parameters (Å, °)

Li1—N6	2.001 (10)	N2—Li2	2.273 (9)
Li1—N1	2.012 (10)	P2—Li2	2.646 (9)
Li1-N2	2.203 (9)	N3—Li2	1.983 (9)
Li1—N5	2.221 (9)	Li2—N4	1.991 (9)

Edelmann, F. T., Knösel, F., Pauer, F. & Stalke, D. (1992). J. Organomet. Chem. 438, 1-10.

CH1 2HU, England. [CIF reference: AL1068]

References

- 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. A46, 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 (1988a). *DIF4*. *Diffractometer Control Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.
- Stoe & Cie (1988b). 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  $(1,2\text{-dimethoxyethane}-\kappa O, O')$  lithium tris(trimethylsilyl)silylselenolate, [Li(C<sub>4</sub>-H<sub>10</sub>O<sub>2</sub>)][SeSi(C<sub>3</sub>H<sub>9</sub>Si)<sub>3</sub>], was determined and the compound was found to be dimeric in the solid state. Pertinent parameters of the dimer [(1,2-dimethoxyethane)LiSeSi(SiMe<sub>3</sub>)<sub>3</sub>]<sub>2</sub> 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).

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As for the structure of the analogous compound  $[(thf)_2LiTeSi(SiMe_3)_3]_2$  (2), and that of the mono-thf adduct [(thf)LiTeSi(SiMe<sub>3</sub>)<sub>3</sub>]<sub>2</sub> (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<sub>2</sub>Se<sub>2</sub> fourmembered ring in (1) was found to be planar. The Li-Se-Li(i) angle is much more acute than the analagous angle in (2)  $[90.4 (6)^{\circ}]$  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),  $103.47 (17)^{\circ}$ ], 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 bisthf 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 silvl ligands closer to the bound ethers. In





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