Fig. 2. Schematic drawings of compound (2), Ph=phenyl, (a) in a 'chair' conformation as found for (1) (the arrows indicate the close phenyl–phenyl contact which would arise), (b) in a conformation with planar Au–Au–P–C–P rings, (c) with non-linear P–Au–P angles as actually occurring in crystals.

The Au–P and Au–Au distances are very similar to those found in (2) (Schmidbaur, Wohlleben, Schubert, Frank & Huttner, 1977). The main differences from (2) are: (i) the almost linear P–Au–P angle [$176 \cdot 6°$, as opposed to $155 \cdot 9°$ in (2)], (ii) the very well maintained planarity of the Au_2P_4 unit [maximum deviations from the least-squares plane are $0 \cdot 04$ Å, as opposed to $0 \cdot 38$ Å in (2)], (iii) the large Au–Cl distances of $3 \cdot 49$ (3) Å which show that the Cl^- ions are not bound covalently [as in (2), where the Au–Cl distance is $2 \cdot 77$ Å] but only associated.

Each of the two water molecules forms two hydrogen bonds to chloride ions of two neighboring unit cells.

If in (2) the Au and P atoms were coplanar as in (1), a close non-bonding contact of approximately $3 \cdot 0$ Å between the axial phenyl rings of the ligands would arise (Fig. 2a). The phenyl rings would come further apart when the methylene C atoms moved into the Au_2P_4 plane (Fig. 2b). This would create, on the other hand, strain in the Au–Au–P–C–P five-membered rings; this strain could be removed upon decreasing the P–Au–P angles (Fig. 2c). Such geometry is actually found for (2). A non-linear P–Au–P structure, of course, is much more susceptible to additional coordination than a linear one. Thus, the structure and bonding in (2) can be explained in terms of steric repulsion. The crystal structure of (1) demonstrates that the linear P–Au–P unit does not have an *a priori* tendency to bend and to bind a chloride ligand. We cannot exclude, however, that the electron-withdrawing effect of the phenyl rings slightly favors the Cl^- coordination in (2), in addition to the steric effect.

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Structure of the Tetrahydrofuran Adduct of Mono-N-lithiohexamethylcyclotrisilazane

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Abstract. Bis-μ-(2,2,4,4,6,6-hexamethylcyclotrisilazanato)-bis[(tetrahydrofuran)lithium(I)], $C_6H_{20}LiN_3Si_3.C_4H_8O$, $M_r = 297 \cdot 55$, triclinic, $P\bar{1}$, $a = 9 \cdot 037$ (1), $b = 12 \cdot 063$ (1), $c = 17 \cdot 779$ (2) Å, $\alpha = 89 \cdot 77$ (1), $\beta = 88 \cdot 62$ (1), $\gamma = 72 \cdot 22$ (1)°, $U = 1845$ Å^3, $Z = 4$, $D_x = 1 \cdot 071$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0 \cdot 71069$ Å, $\mu = 0 \cdot 244$ mm^{-1}, $F(000) = 648$, $T = 290$ K, $R = 0 \cdot 053$ for 4450 observed reflections. There are two crystallographically independent dimers, each lying on an inversion centre. The Si_3N_3 ring has a distorted boat

conformation and the Li-bonded N atom has distorted tetrahedral geometry. The Si–N bond lengths involving this N or the diagonally opposite Si are short [mean 1·697 (3) Å].

Introduction. Lithium salts of cyclotrisilazanes – especially when Si–F-substituted – are important intermediates in ring coupling (Hesse & Klingebiel, 1981) and base-catalyzed isomerization reactions (Clegg, Hesse, Klingebiel, Sheldrick & Skoda, 1980). The title compound was prepared by the reaction of $(Me_2SiNH)_3$ with n-BuLi in n-hexane/THF.

Experimental. Crystals obtained from mother liquor by cooling, $0·71 \times 0·59 \times 0·47$ mm, mounted in capillaries under N_2; Stoe–Siemens AED diffractometer; unit-cell parameters from 2θ values of 40 reflections $(18 < 2\theta < 23°)$; 8686 reflections with $2\theta \le 48°$ and $-10 \le h \le 10$, $-13 \le k \le 13$, $-1 \le l \le 20$; profile analysis (Clegg, 1981); no significant intensity variation for three standard reflections, no absorption corrections; $R_{int} = 0·022$; 7681 unique reflections, 4450 with $F > 4\sigma(F)$; automatic multisolution direct methods; blocked-cascade least squares on F; $w^{-1} = \sigma^2(F) + 0·0002F^2$ (which resulted in a flat analysis of variance); methyl H constrained as rigid group with C–H

$= 0·96$ Å, $H–C–H = 109·5°$, $U(H) = 1·2$ $U_{eq}(C)$, $H(N)$ refined freely with $U(H) = 1·2$ $U_{eq}(N)$; anisotropic thermal parameters for non-H atoms; no extinction corrections; scattering factors from *International Tables for X-ray Crystallography* (1974); 373 parameters, $R = 0·053$, $wR = 0·059$, slope of normal probability plot $= 1·87$, max. $\Delta/\sigma = 0·017$, mean 0·002, largest peak in final difference map $= 0·29$ e Å^{-3}, largest hole $= -0·32$ e Å^{-3}. Programs: *SHELXTL* (Sheldrick, 1978), diffractometer control program (Clegg, 1981).

Table 1. *Atomic coordinates* $(\times 10^4)$ *and isotropic thermal parameters* $(\text{Å}^2 \times 10^3)$

	x	y	z	U_{eq}*
Si(1)	4749 (1)	3104 (1)	4073 (1)	53 (1)
C(1)	3921 (5)	4262 (3)	3362 (2)	88 (2)
C(2)	3148 (5)	2484 (4)	4338 (2)	103 (2)
N(1)	5473 (3)	3620 (2)	4821 (1)	48 (1)
Si(2)	6896 (1)	2668 (1)	5303 (1)	59 (1)
C(3)	8636 (4)	3193 (4)	5320 (3)	100 (2)
C(4)	6420 (6)	2441 (3)	6306 (2)	97 (2)
N(2)	7422 (4)	1287 (2)	4900 (2)	84 (1)
Si(3)	7551 (1)	921 (1)	3975 (1)	70 (1)
C(5)	9498 (5)	779 (4)	3531 (3)	111 (2)
C(6)	7170 (6)	−496 (3)	3854 (3)	113 (2)
N(3)	6186 (4)	2051 (2)	3583 (1)	63 (1)
Li(1)	5982 (6)	5101 (4)	4535 (3)	58 (2)
O(1)	7423 (3)	5208 (2)	3742 (1)	81 (1)
C(2a)	7978 (7)	6162 (4)	3616 (3)	143 (3)
C(3a)	8893 (7)	5982 (5)	2955 (3)	150 (3)
C(4a)	9104 (7)	4784 (5)	2720 (3)	152 (3)
C(5a)	8204 (7)	4343 (4)	3205 (3)	139 (3)
Si(1')	37 (1)	2015 (1)	9110 (1)	56 (1)
C(1')	1242 (5)	935 (3)	8412 (2)	95 (2)
C(2')	1297 (5)	2869 (4)	9441 (3)	109 (2)
N(1')	−677 (3)	1375 (2)	9821 (1)	48 (1)
Si(2')	−3017 (1)	3967 (1)	8971 (1)	59 (1)
C(3')	−4810 (4)	3953 (3)	8479 (2)	85 (2)
C(4')	−2864 (5)	5463 (3)	8909 (2)	92 (2)
N(2')	−1443 (3)	2935 (2)	8596 (1)	59 (1)
Si(3')	−2330 (1)	2178 (1)	10273 (1)	56 (1)
C(5')	−3876 (4)	1453 (3)	10208 (2)	88 (2)
C(6')	−2127 (5)	2435 (3)	11297 (2)	88 (2)
N(3')	−3014 (4)	3562 (2)	9891 (2)	69 (1)
Li(1')	811 (6)	220 (4)	10494 (3)	56 (2)
O(1')	1966 (3)	489 (2)	11332 (1)	77 (1)
C(2b)	2264 (5)	1558 (4)	11503 (3)	112 (2)
C(3b)	3448 (8)	1310 (5)	12061 (3)	160 (4)
C(4b)	3826 (7)	112 (5)	12215 (4)	195 (4)
C(5b)	2815 (6)	−367 (4)	11841 (3)	112 (2)

* U_{eq} defined as one third of the trace of the orthogonalized $\mathbf{U}_{ij}$ tensor.

Table 2. *Bond lengths* (Å) *and bond angles* (°)

Si(1)–C(1)	1·871 (4)	Si(1)–C(2)	1·871 (5)
Si(1)–N(1)	1·697 (3)	Si(1)–N(3)	1·733 (3)
N(1)–Si(2)	1·689 (2)	N(1)–Li(1)	2·036 (6)
N(1)–Li(1a)	2·031 (5)	Si(2)–C(3)	1·866 (5)
Si(2)–C(4)	1·863 (4)	Si(2)–N(2)	1·739 (3)
N(2)–H(2)	0·777 (34)	N(2)–Si(3)	1·697 (3)
Si(3)–C(5)	1·871 (5)	Si(3)–C(6)	1·859 (5)
Si(3)–N(3)	1·694 (3)	N(3)–H(3)	0·737 (28)
Li(1)–O(1)	1·927 (6)	Li(1)–N(1a)	2·031 (5)
Li(1)–Li(1a)	2·463 (11)	O(1)–C(2a)	1·404 (6)
O(1)–C(5a)	1·422 (5)	C(2a)–C(3a)	1·400 (8)
C(3a)–C(4a)	1·460 (8)	C(4a)–C(5a)	1·384 (9)
Si(1')–C(1')	1·872 (4)	Si(1')–C(2')	1·859 (6)
Si(1')–N(1')	1·694 (3)	Si(1')–N(2')	1·733 (3)
N(1')–Si(3')	1·696 (2)	N(1')–Li(1')	2·025 (5)
N(1')–Li(1b)	2·046 (6)	Si(2')–C(3')	1·864 (4)
Si(2')–C(4')	1·854 (4)	Si(2')–N(2')	1·702 (3)
Si(2')–N(3')	1·705 (3)	N(2')–H(2')	0·772 (27)
Si(3')–C(5')	1·867 (5)	Si(3')–C(6')	1·871 (3)
Si(3')–N(3')	1·735 (3)	N(3')–H(3')	0·716 (31)
Li(1')–O(1')	1·924 (6)	Li(1')–N(1'a)	2·046 (6)
Li(1')–Li(1b)	2·475 (11)	O(1')–C(2b)	1·431 (5)
O(1')–C(5b)	1·420 (5)	C(2b)–C(3b)	1·439 (8)
C(3b)–C(4b)	1·406 (9)	C(4b)–C(5b)	1·403 (9)
C(1)–Si(1)–N(1)	112·3 (2)	C(2)–Si(1)–N(1)	113·2 (2)
C(1)–Si(1)–N(3)	104·7 (1)	C(2)–Si(1)–N(3)	108·5 (2)
N(1)–Si(1)–N(3)	111·6 (1)	Si(1)–N(1)–Si(2)	117·7 (1)
Si(1)–N(1)–Li(1)	110·2 (2)	Si(2)–N(1)–Li(1)	113·2 (2)
Si(1)–N(1)–Li(1a)	118·9 (2)	Si(2)–N(1)–Li(1a)	114·2 (2)
Li(1)–N(1)–Li(1a)	74·5 (3)	N(1)–Si(2)–C(3)	109·5 (2)
N(1)–Si(2)–C(4)	115·8 (2)	N(1)–Si(2)–N(2)	111·4 (1)
C(3)–Si(2)–N(2)	109·1 (2)	C(4)–Si(2)–N(2)	104·8 (2)
Si(2)–N(2)–H(2)	111·3 (26)	Si(3)–N(2)–Si(3)	128·5 (2)
H(2)–N(2)–Si(3)	120·0 (25)	N(2)–Si(3)–C(5)	113·5 (2)
N(2)–Si(3)–C(6)	110·0 (2)	N(2)–Si(3)–N(3)	103·1 (1)
C(5)–Si(3)–C(6)	109·7 (2)	C(6)–Si(3)–N(3)	113·2 (2)
Si(1)–N(3)–Si(3)	125·5 (2)	Si(1)–N(3)–H(3)	114·8 (24)
Si(3)–N(3)–H(3)	119·4 (24)	N(1)–Li(1)–O(1)	124·7 (3)
N(1)–Li(1)–N(1a)	105·5 (3)	O(1)–Li(1)–N(1a)	129·6 (3)
N(1)–Li(1)–Li(1a)	52·6 (2)	O(1)–Li(1)–Li(1a)	175·0 (4)
Li(1)–O(1)–C(2a)	125·3 (3)	Li(1)–O(1)–C(5a)	128·2 (3)
C(2a)–O(1)–C(5a)	106·4 (4)	O(1)–C(2a)–C(3a)	109·6 (4)
C(2a)–C(3a)–C(4a)	106·4 (5)	C(3a)–C(4a)–C(5a)	107·0 (5)
O(1)–C(5a)–C(4a)	109·8 (4)	C(1')–Si(1')–N(1')	112·6 (2)
C(2')–Si(1')–N(1')	113·0 (2)	C(1')–Si(1')–N(2')	105·2 (1)
C(2')–Si(1')–N(2')	108·0 (2)	N(1')–Si(1')–N(2')	111·4 (1)
Si(1')–N(1')–Si(3')	117·7 (1)	Si(1')–N(1')–Li(1')	119·5 (2)
Si(3')–N(1')–Li(1')	113·7 (2)	Si(1')–N(1')–Li(1b)	111·3 (2)
Si(3')–N(1')–Li(1b)	111·7 (2)	Li(1')–N(1')–Li(1b)	74·9 (3)
C(3')–Si(2')–N(2')	110·1 (2)	C(4')–Si(2')–N(2')	113·5 (2)
C(3')–Si(2')–N(3')	113·1 (2)	C(4')–Si(2')–N(3')	109·7 (2)
N(2')–Si(2')–N(3')	102·8 (1)	Si(1')–N(2')–Si(2')	125·0 (1)
Si(1')–N(2')–H(2')	116·4 (22)	Si(2')–N(2')–H(2')	118·4 (21)
N(1')–Si(3')–C(5')	110·5 (1)	N(1')–Si(3')–C(6')	115·1 (2)
N(1')–Si(3')–N(3')	111·3 (1)	C(5')–Si(3')–N(3')	108·7 (2)
C(6')–Si(3')–N(3')	104·5 (2)	Si(2')–N(3')–Si(3')	128·6 (2)
Si(2')–N(3')–H(3')	120·1 (26)	Si(3')–N(3')–H(3')	110·9 (27)
N(1')–Li(1')–O(1')	129·8 (3)	N(1')–Li(1')–N(1'a)	105·1 (3)
O(1')–Li(1')–N(1'a)	124·7 (3)	N(1')–Li(1')–Li(1b)	53·0 (2)
O(1')–Li(1')–Li(1b)	174·3 (4)	Li(1')– O(1')–C(2b)	126·0 (3)
Li(1')–O(1')–C(5b)	126·0 (3)	C(2b)–O(1')–C(5b)	107·7 (3)
O(1')–C(2b)–C(3b)	107·5 (4)	C(2b)–C(3b)–C(4b)	106·5 (6)
C(3b)–C(4b)–C(5b)	110·0 (5)	O(1')–C(5b)–C(4b)	107·1 (4)

Discussion. Atomic coordinates, bond lengths and angles are given in Tables 1 and 2.* Corresponding bond lengths and angles of the two crystallographically independent molecules are insignificantly different. The molecular structure, illustrated in Fig. 1, shows the compound to be a dimer of the lithiumamide with terminal THF molecules completing the three-coordinated planar Li-atom environment. The Li-bonded N atom has distorted tetrahedral geometry. Bond lengths and angles are comparable to those found in [(Me₃Si)₂NLi.OEt₂]₂ (Lappert, Slade, Singh, Atwood, Rogers & Shakir, 1983). The Si_3N_3 ring has a strongly distorted boat conformation [torsion angles Si(1)—N(1)—Si(2)—N(2)—Si(3)—N(3)—Si(1): −36·7 (2), 1·6 (9), 37·6 (9), −29·7 (10), −15·1 (19), 47·6 (17)°; Bucourt & Hainant, 1965]. This change in conformation from the almost planar $(RR'SiNH)_3$ rings (Clegg, Sheldrick & Stalke, 1984) to an approximate boat conformation may be caused by the anionic character of the N atom. The Si—N bond lengths in the ring involving either this N or the diagonally opposite Si are short [mean 1·697 (3) Å], whereas the middle Si—N distances are normal [mean 1·735 (3) Å]. The H-bonded N atoms have virtually planar geometry [mean N—H 0·75 (7) Å, freely refined].

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* Lists of structure factors, anisotropic temperature factors and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42928 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
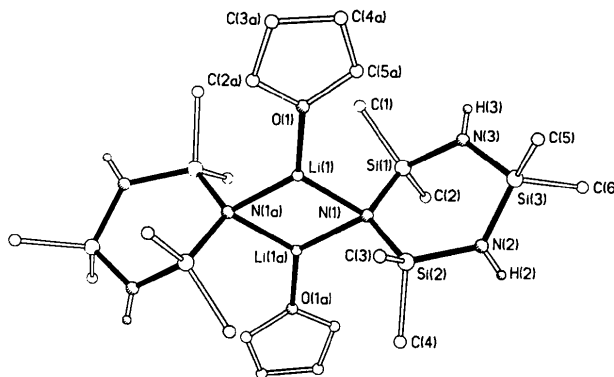

Fig. 1. Molecular structure of the dimer. H atoms of the methyl groups and of the THF molecules are omitted, and the unique atoms plus three symmetry-generated atoms N(1a), Li(1a) and O(1a) are labelled.

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Structure of Decacarbonyl-tetra-μ-hydrido-μ-[methylenebis(diphenylphosphine)-*P*,*P'*]-*tetrahedro*-tetraruthenium

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Abstract. [Ru₄(C₂₅H₂₂P₂)(CO)₁₀H₄], $M_r = 1072·8$, monoclinic, $P2_1/c$, $a = 10·887$ (2), $b = 19·751$ (3), $c = 18·278$ (2) Å, $\beta = 105·89$ (1)°, $V = 3780·1$ (2) Å³, $Z = 4$, $D_x = 1·89$ g cm⁻³, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 16·65$ cm⁻¹, $F(000) = 2088$, $T = 118$ (1) K, $R = 0·030$ for 5569 observed reflections. The structure consists of a closed tetrahedral metal core involving four hydrido-bridged metal–metal bonds [Ru(1)–Ru(3)

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