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THE CRYSTAL STRUCTURE OF *N*-LITHIOHEXAMETHYLDISILAZANE, $[LiN(SiMe_3)_2]_3$

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Summary

The crystal structure of $[LiN(SiMe_3)_2]_3$ has been determined from single-crystal X-ray diffraction data collected by counter methods. *N*-Lithiohexamethyldisilazane crystallizes in the monoclinic space group $P2_1/c$ with unit cell parameters a 8.848(4), b 31.868(9), c 12.325(4) Å, β = 104.93(3)°, and ρ_{calc} 0.99 g cm^{-3} for Z = 4 trimeric units. Least-squares refinement gave a final conventional R value of 0.050 for 2449 independent observed reflections. In the solid state the compound exists in a trimeric configuration with a planar Li_3N_3 ring. The average Li—N length is 2.00(2) Å, and the ring angles are N—Li—N = 147(3)°, and Li—N—Li = 92(2)°. A greater degree of covalency in the title compound compared to $Na[N(SiMe_3)_2]$ and $K[N(SiMe_3)_2] \cdot 2\ C_4H_8O_2$ can be inferred from the Si—N bond length of 1.729(4) Å.

Introduction

The properties of the alkali metal disilazanes have been the subject of numerous investigations and the results of three crystal structures have appeared. The potassium salt is insoluble in hydrocarbon media, and the X-ray structure [2] was done on the bisdioxane adduct: $K[N(SiMe_3)_2] \cdot 2\ C_4H_8O_2$. It is revealed a Si—N—Si angle of 136(1)°, 7° larger than any other disilazane derivative. *N*-Sodiohexamethyldisilazane is dimeric in solution [3], but exists as a polymer in the solid state [1]. The Si—N—Si angle is 125.6(1)°. The lithium analogue is also reported to be a dimer in hydrocarbon solution [3], but according to a preliminary note by Moots et al. [4], is a trimer in the crystalline solid. Here the Si—N—Si angle is 118°.

The communication [4] of the structure of $[LiN(SiMe_3)_2]_3$ was based on film data, and no coordinates or standard deviations in the structural parameters were reported. Although it was stated that the compound possesses a planar Li_3N_3 ring, careful scrutiny of the molecular illustration in the original manu-

TABLE 1

CRYSTAL DATA

Mol. formula:	$[LiN(SiMe_3)_2]_3$
Mol. wt.:	501.98
Linear abs. coeff. μ:	2.53 cm^{-1}.
Calc. density:	0.99 g cm^{-3}
Max. crystal dimensions:	0.30 × 0.30 × 0.40 mm
Space group:	monoclinic, $P2_1/c$
Molecules/unit cell:	4
Cell constants [a]:	a = 8.848(4) Å
	b = 31.868(9) Å
	c = 12.325(4) Å
	β = 104.33(3)°
Cell volume:	3357.9 $Å^3$

[a] Mo-K_α radiation, λ = 0.71069 Å. Ambient temperature of 23 ± 1°C.

script and in a subsequent review article [3], lead to the inference of a decidedly nonplanar ring. In order to understand subtle but important features of the bonding in this compound, we have carried out a detailed structural analysis.

Experimental

N-Lithiohexamethyldisilazane was recrystallized under a nitrogen atmosphere from benzene, and the colorless, air-sensitive parallelepipeds were sealed in thin-walled glass capillaries. Final lattice parameters as determined from a least squares refinement of the angular settings of 15 reflections ($2\theta > 20°$) accurately centered on an Enraf-Nonius CAD-4 diffractometer are given in Table 1.

Data were collected on the diffractometer with graphite crystal monochromated molybdenum radiation. The diffracted intensities were collected by the $\omega - 2\theta$ scan technique with a take-off angle of 3.5°. The scan rate was variable and was determined by a fast 20° min^{-1} prescan. Calculated speeds for the slow scan (based on the net intensity in the prescan) ranged from 7 to 0.4° min^{-1}. Other diffractometer parameters and the method of estimation of standard deviations have been previously described [5]. As a check on the stability of the instrument and crystal, two reflections were measured after every 40 reflections; no significant variation was noted.

One independent quadrant of data was measured out to $2\theta = 50°$; a slow scan was performed on a total of 2449 unique reflections. Since these data were scanned at a speed which would yield a net count of 4000, the calculated standard deviations were all very nearly equal. No reflection was subjected to a slow scan unless a net count of 30 was obtained in the prescan. Based on these considerations, the data set of 2449 reflections (used in the subsequent structure determination and refinement) was considered observed, and consisted of those for which $I \geqslant 3\sigma(I)$. The intensities were corrected for Lorentz and polarization effects, but not for absorption (μ = 2.53 cm^{-1}).

The function $w(|F_o| - |F_c|)^2$ was minimized *. No corrections were made for

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* Crystallographic programs used on a UNIVAC 1110 include ORFLS (structure factor calculation and least-squares refinement) [6a], ORFFE (distances and angles with esd's) [6b], ORTEP (thermal ellipsoid drawings) [6c], FOURIER [6d], and BPL (least-squares planes) [6e].

(Continued on p. 234)

TABLE 2

FINAL FRACTIONAL COORDINATES AND THERMAL PARAMETERS ^{a} FOR *N*-LITHIOHEXAMETHYLDISILAZANE

Atom	*x/a*	*y/b*	*z/c*	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Si(1)	0.6118(2)	0.0754(1)	0.3433(2)	0.0142(3)	0.0011(1)	0.0061(2)	−0.0003(1)	0.0026(2)	0.0004(1)
Si(2)	0.9520(2)	0.0895(1)	0.3818(2)	0.0122(3)	0.0012(1)	0.0074(2)	0.0008(1)	0.0016(2)	0.0006(1)
Si(3)	0.7995(2)	0.0861(1)	−0.0714(2)	0.0134(2)	0.0010(1)	0.0069(2)	0.0003(1)	0.0037(2)	−0.0002(1)
Si(4)	0.4629(2)	0.1060(1)	−0.1022(2)	0.0107(3)	0.0011(1)	0.0064(2)	−0.0007(1)	0.0020(2)	−0.0004(1)
Si(5)	0.5738(2)	0.2345(1)	0.1947(2)	0.0105(2)	0.0008(1)	0.0079(2)	0.0003(1)	0.0019(2)	−0.0002(1)
Si(6)	0.8909(2)	0.2352(1)	0.1514(2)	0.0116(2)	0.0009(1)	0.0074(2)	−0.0004(1)	0.0022(2)	−0.0002(1)
N(1)	0.7638(5)	0.0988(1)	0.3015(4)	0.0134(7)	0.0010(1)	0.0060(4)	0.0002(2)	0.0020(4)	0.0005(1)
N(2)	0.6529(5)	0.1091(1)	−0.0216(4)	0.0122(7)	0.0009(1)	0.0065(4)	0.0002(2)	0.0031(4)	−0.0003(1)
N(3)	0.7274(4)	0.2077(1)	0.1635(4)	0.0113(7)	0.0008(1)	0.0080(5)	0.0001(2)	0.0016(4)	−0.0001(1)
Li(1)	0.7060(13)	0.0882(4)	0.1361(10)	0.0202(19)	0.0012(2)	0.0094(10)	−0.0006(4)	0.0048(11)	−0.0004(3)
Li(2)	0.6785(12)	0.1700(4)	0.0279(10)	0.0122(15)	0.0018(2)	0.0093(11)	−0.0001(4)	0.0021(10)	0.0001(3)
Li(3)	0.7631(12)	0.1601(4)	0.2747(10)	0.0165(18)	0.0012(2)	0.0100(11)	0.0005(4)	0.0022(11)	0.0002(3)
C(1)	0.4329(8)	0.0763(3)	0.2224(6)	0.0182(12)	0.0020(1)	0.0086(7)	−0.0018(3)	0.0016(7)	0.0003(2)
C(2)	0.5604(9)	0.1024(3)	0.4626(6)	0.0223(14)	0.0024(2)	0.0085(7)	0.0002(4)	0.0057(8)	0.0000(2)
C(3)	0.6481(9)	0.0194(3)	0.3833(7)	0.0278(17)	0.0017(1)	0.0121(9)	−0.0010(4)	0.0050(10)	0.0009(2)
C(4)	1.0816(9)	0.1340(3)	0.3566(7)	0.0190(14)	0.0020(1)	0.0139(9)	−0.0005(3)	0.0003(9)	0.0017(3)
C(5)	1.0372(9)	0.0394(3)	0.3486(7)	0.0261(16)	0.0021(1)	0.0129(9)	0.0033(4)	0.0065(10)	0.0019(3)
C(6)	0.9743(8)	0.0888(3)	0.5352(6)	0.0175(12)	0.0024(1)	0.0078(7)	0.0008(3)	−0.0006(7)	0.0006(2)
C(7)	0.9712(9)	0.0763(3)	0.0496(6)	0.0203(14)	0.0026(2)	0.0107(8)	0.0028(4)	0.0043(8)	0.0008(3)
C(8)	0.8738(8)	0.1182(2)	−0.1738(6)	0.0187(12)	0.0012(1)	0.0098(7)	0.0001(3)	0.0067(7)	0.0002(2)
C(9)	0.7448(10)	0.0347(3)	−0.1408(8)	0.0309(18)	0.0012(1)	0.0179(11)	0.0003(4)	0.0129(11)	−0.0009(3)
C(10)	0.3458(7)	0.1481(2)	−0.0554(6)	0.0132(10)	0.0018(1)	0.0110(7)	0.0004(3)	0.0023(7)	0.0000(2)
C(11)	0.4389(8)	0.1142(3)	−0.2548(6)	0.0167(12)	0.0020(1)	0.0094(7)	−0.0011(3)	−0.0002(7)	−0.0005(2)
C(12)	0.3631(8)	0.0541(2)	−0.0915(7)	0.0227(14)	0.0013(1)	0.0123(8)	−0.0016(3)	0.0042(8)	0.0000(2)
C(13)	0.4387(8)	0.2612(3)	0.0730(6)	0.0175(12)	0.0018(1)	0.0111(8)	0.0014(3)	0.0023(8)	0.0003(2)
C(14)	0.4554(7)	0.1971(2)	0.2550(6)	0.0175(12)	0.0013(1)	0.0116(8)	−0.0003(3)	0.0064(8)	−0.0006(2)
C(15)	0.6347(8)	0.2761(2)	0.3044(6)	0.0196(12)	0.0013(1)	0.0129(8)	0.0009(3)	0.0066(8)	−0.0003(2)
C(16)	1.0006(7)	0.2013(2)	0.0732(6)	0.0158(11)	0.0015(1)	0.0080(6)	−0.0005(3)	0.0042(6)	−0.0001(2)
C(17)	0.8507(9)	0.2852(2)	0.0749(7)	0.0222(14)	0.0014(1)	0.0164(10)	−0.0010(3)	0.0071(9)	0.0006(3)
C(18)	1.0308(8)	0.2500(2)	0.2880(6)	0.0166(11)	0.0015(1)	0.0115(8)	−0.0012(3)	0.0037(7)	−0.0014(2)

(Table continued)

TABLE 2 (*continued*)

Atom	x/a	y/b	z/c	β_{11}
H(1) [C(1)]	0.418	0.099	0.168	5.7
H(2) [C(1)]	0.452	0.056	0.163	5.0
H(3) [C(1)]	0.363	0.059	0.228	5.4
H(4) [C(2)]	0.613	0.097	0.523	6.5
H(5) [C(2)]	0.529	0.132	0.451	7.6
H(6) [C(2)]	0.490	0.096	0.476	6.2
H(7) [C(3)]	0.564	0.013	0.399	3.9
H(8) [C(3)]	0.686	0.018	0.457	7.9
H(9) [C(3)]	0.657	−0.000	0.327	7.5
H(10) [C(4)]	1.074	0.136	0.271	5.2
H(11) [C(4)]	1.064	0.161	0.392	6.9
H(12) [C(4)]	1.114	0.120	0.415	6.4
H(13) [C(5)]	1.001	0.012	0.374	3.9
H(14) [C(5)]	1.107	0.039	0.372	4.7
H(15) [C(5)]	1.041	0.039	0.263	7.2
H(16) [C(6)]	0.973	0.117	0.572	4.0
H(17) [C(6)]	1.051	0.078	0.570	5.0
H(18) [C(6)]	0.915	0.078	0.567	6.0
H(19) [C(7)]	0.954	0.049	0.084	5.2
H(20) [C(7)]	0.990	0.090	0.124	6.3
H(21) [C(7)]	1.050	0.077	0.026	6.1
H(22) [C(8)]	0.808	0.125	−0.236	5.6
H(23) [C(8)]	0.932	0.100	−0.203	4.9
H(24) [C(8)]	0.917	0.146	−0.145	4.2
H(25) [C(9)]	0.730	0.014	−0.092	5.3
H(26) [C(9)]	0.671	0.039	−0.216	7.1
H(27) [C(9)]	0.820	0.023	−0.168	6.5
H(28) [C(10)]	0.269	0.146	−0.096	6.6
H(29) [C(10)]	0.376	0.178	−0.075	3.6
H(30) [C(10)]	0.357	0.140	0.024	4.6
H(31) [C(11)]	0.456	0.089	−0.285	2.8
H(32) [C(11)]	0.360	0.116	−0.286	2.8

H(33) [C(11)]	0.455	0.144	−0.275	5.8
H(34) [C(12)]	0.310	0.067	−0.115	4.3
H(35) [C(12)]	0.413	0.027	−0.099	5.0
H(36) [C(12)]	0.373	0.044	−0.012	4.1
H(37) [C(13)]	0.410	0.241	0.007	4.1
H(38) [C(13)]	0.373	0.271	0.092	4.2
H(39) [C(13)]	0.472	0.281	0.022	5.8
H(40) [C(14)]	0.426	0.173	0.205	4.3
H(41) [C(14)]	0.512	0.187	0.330	5.6
H(42) [C(14)]	0.390	0.208	0.290	4.4
H(43) [C(15)]	0.652	0.263	0.381	5.7
H(44) [C(15)]	0.672	0.297	0.277	5.1
H(45) [C(15)]	0.568	0.289	0.320	3.7
H(46) [C(16)]	0.944	0.194	−0.006	3.5
H(47) [C(16)]	1.025	0.176	0.115	4.8
H(48) [C(16)]	1.071	0.216	0.057	4.3
H(49) [C(17)]	0.922	0.300	0.067	4.4
H(50) [C(17)]	0.840	0.276	0.002	7.9
H(51) [C(17)]	0.764	0.291	0.057	5.2
H(52) [C(18)]	1.105	0.263	0.265	4.2
H(53) [C(18)]	1.002	0.272	0.335	5.2
H(54) [C(18)]	1.066	0.224	0.318	6.1

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[a] Anisotropic thermal parameters defined by $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

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extinction. Neutral atom scattering factors were taken from the compilations of Cromer and Waber [7] for Li, Si, N, and C; those for H were from "International Tables for X-ray Crystallography" [8].

Structure solution and refinement

The structure was solved by the straight-forward application of the direct methods program MULTAN [9]. Several cycles of least squares refinement of the positional and isotropic thermal parameters of the 30 nonhydrogen atoms afforded a reliability factor of $R_1 = \Sigma(|F_0| - |F_c|)/\Sigma|F_0| = 0.146$. Conversion to anisotropic thermal parameters and further refinement gave $R_1 = 0.066$. The positions of the 54 hydrogen atoms were determined from a difference Fourier map, and refined for two cycles with a damping factor of 0.2. More cycles of refinement of the nonhydrogen atoms led to final values of $R_1 = 0.050$ and $R_2 = [\Sigma w(|F_0| - |F_c|)^2/\Sigma w(F_0)^2]^{1/2} = 0.054$. The weighting scheme was based on unit weights, and unobserved reflections were not included. The largest parameter shifts in the final cycle of refinement were less than 0.01 of their estimated standard deviations. The estimated standard deviation of an observation of unit weight was 0.94. A final difference Fourier map showed no unaccounted electron density. The final values of the positional and thermal parameters are given in Table 2.

Discussion

The structure and atom numbering scheme for $[LiN(SiMe_3)_2]_3$ are presented in Fig. 1. The packing of the trimers into the unit cell is shown in Fig. 2. The Li_3N_3 ring is planar to within 0.01 Å, and shows average values of Li—N = 2.00(2) Å, N—Li—N = 147(3)°, and Li—N—Li = 92(2)° (Table 3). In the polymeric structure of $Na[N(SiMe_3)_2]$ [1], the Na—N length, 2.355(3) Å, agrees well with

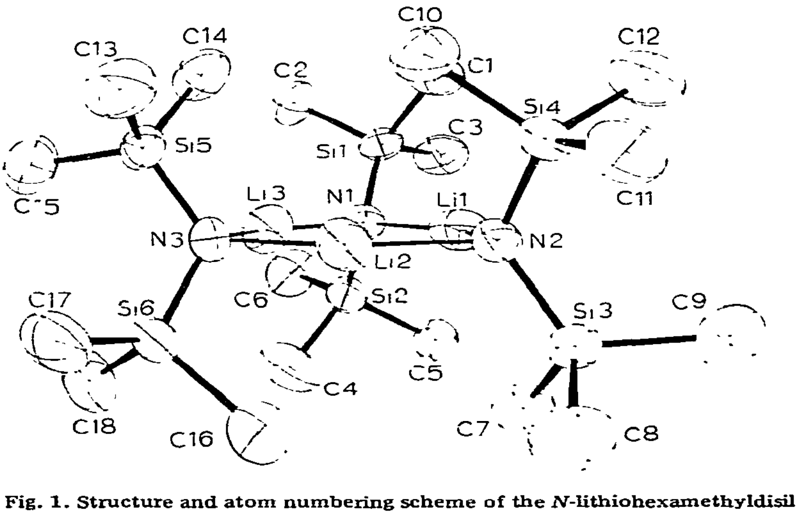

Fig. 1. Structure and atom numbering scheme of the *N*-lithiohexamethyldisilazane trimer.

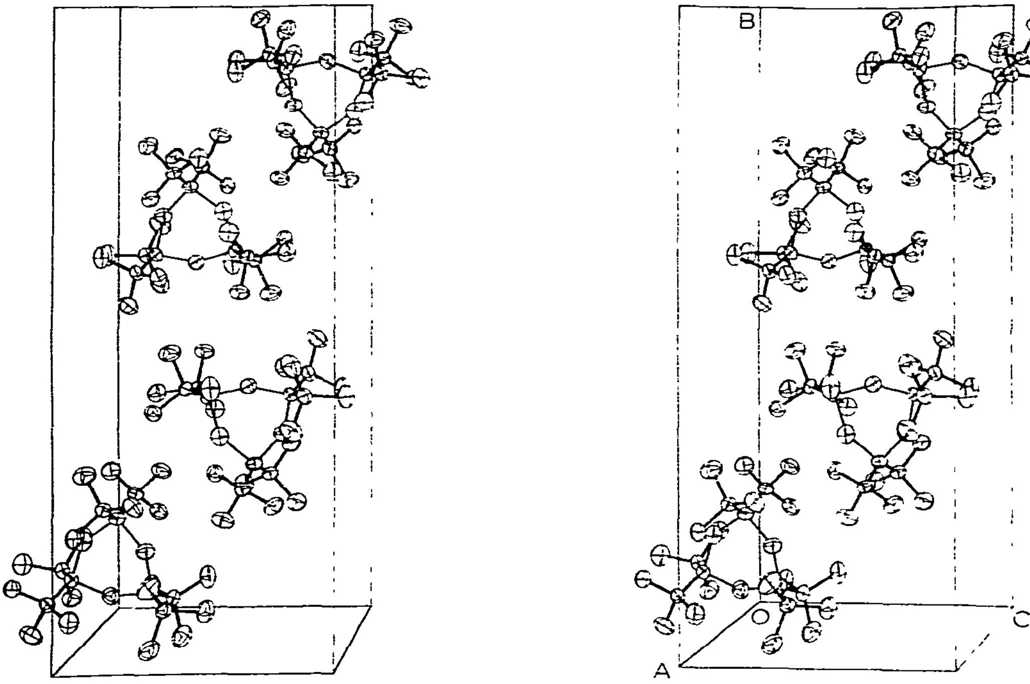

Fig. 2. Stereoscopic view of the packing of the trimers in the unit cell with the atoms represented by their 50% probability ellipsoids for thermal motion.

this result after a correction of 0.35 Å for the difference in Na$^+$ and Li$^+$ radii [10a]. Angles of N—Na—N = 150.2(1)° and Na—N—Na = 102.0(1)° are also comparable to those found in the trimeric structure.

The two important parameters of the hexamethyldisilazane ion fit well into a bonding scheme which involves an appreciable covalent contribution to the Li—N bond. The Si—N bond length 1.729(4) Å is comparable to the 1.735(12) Å found in $H[N(SiMe_3)_2]$ [11] and 1.73(3) Å in $Fe[N(SiMe_3)_2]_3$ [12]. It is significantly longer than the 1.690(5) Å in $Na[N(SiMe_3)_2]$, and the 1.64(1) Å in $K[N(SiMe_3)_2] \cdot 2C_4H_8O_2$ [2]. The average Si—N—Si bond angle, 118.6(9)° is much smaller than the 125.6(1)° for the Na salt, and 136(1)° for that of K. A similar argument has been used to attribute a greater degree of covalency in $Sc[N(SiMe_3)_2]_3$ [13] compared to $Eu[N(SiMe_3)_2]_3$ [13].

The nature of the nonbonded methyl—methyl repulsions around the Li_3N_3 ring can be assessed via a comparison of $Li_3[N(SiMe_3)_2]_3$ with other compounds of formulation $M[N(SiMe_3)_2]_3$. The N···N separation of 3.85(1) Å for M = Li_3 is the same as that found in $Eu[N(SiMe_3)_2]_3$, but is much longer than the 3.08 Å calculated for $Al[N(SiMe_3)_2]_3$ [14]. Even so, it is likely that methyl—methyl repulsions exert a small effect on the bonding parameters of the ring, since the H···H distances on separate anions around the ring are as small as 2.12 Å *. The sum of the van der Waals radii for two hydrogen atoms is 2.4 Å [10b].

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* The average C—H distance is 0.91(10) Å, and the closest H···H appraoch between hydrogen atoms on separate trimers is 2.53 Å.

TABLE 3

INTERATOMIC DISTANCES (Å) AND ANGLES (°) FOR *N*-LITHIOHEXAMETHYLDISILAZANE

Bond distances			
Li(1)—N(1)	1.998(12)	Si(1)—N(1)	1.728(4)
Li(1)—N(2)	1.994(13)	Si(2)—N(1)	1.729(4)
Li(2)—N(2)	2.015(14)	Si(3)—N(2)	1.734(4)
Li(2)—N(3)	2.022(13)	Si(4)—N(2)	1.721(4)
Li(3)—N(3)	2.015(12)	Si(5)—N(3)	1.730(5)
Li(3)—N(1)	1.983(12)	Si(6)—N(3)	1.730(5)
Si(1)—C(1)	1.874(7)	Si(3)—C(7)	1.861(7)
Si(1)—C(2)	1.858(7)	Si(3)—C(8)	1.872(7)
Si(1)—C(3)	1.855(8)	Si(3)—C(9)	1.855(8)
Si(2)—C(4)	1.898(8)	Si(4)—C(10)	1.874(7)
Si(2)—C(5)	1.855(8)	Si(4)—C(11)	1.855(8)
Si(2)—C(6)	1.849(7)	Si(4)—C(12)	1.893(7)
Si(5)—C(13)	1.865(7)	Si(6)—C(16)	1.877(7)
Si(5)—C(14)	1.863(7)	Si(6)—C(17)	1.865(8)
Si(5)—C(15)	1.871(7)	Si(6)—C(18)	1.874(7)
Si(1)···Si(2)	2.958(2)	N(1)···N(2)	3.862(9)
Si(3)···Si(4)	2.971(2)	N(1)···N(3)	3.844(9)
Si(5)···Si(6)	2.988(2)	N(2)···N(3)	3.839(9)
Li(1)···Li(2)	2.898(18)	Li(1)···Li(3)	2.826(16)
Li(2)···Li(3)	2.956(16)		
Bond angles			
N(1)—Li(1)—N(2)	150.7(7)	Li(1)—N(2)—Li(2)	92.6(5)
N(2)—Li(2)—N(3)	144.0(7)	Li(2)—N(3)—Li(3)	94.1(5)
N(3)—Li(3)—N(1)	148.1(7)	Li(1)—N(1)—Li(3)	90.4(5)
Si(1)—N(1)—Si(2)	117.7(3)	Si(1)—N(1)—Li(1)	102.6(4)
Si(3)—N(2)—Si(4)	118.6(3)	Si(1)—N(1)—Li(3)	120.3(4)
Si(5)—N(3)—Si(6)	119.5(3)	Si(2)—N(1)—Li(1)	120.8(4)
Si(2)—N(1)—Li(3)	102.9(4)	Si(5)—N(3)—Li(2)	117.9(4)
Si(3)—N(2)—Li(1)	101.2(4)	Si(5)—N(3)—LI(3)	102.4(4)
Si(3)—N(2)—Li(2)	118.4(4)	Si(6)—N(3)—Li(2)	103.8(4)
Si(4)—N(2)—Li(1)	119.5(4)	Si(6)—N(3)—Li(3)	116.8(4)
Si(4)—N(2)—Li(2)	104.3(4)		

The lithium atom is coordinated to only the two nitrogen atoms. The shortest Li···H length is 2.56(1) Å. This results in the short Li—N bond distance of 2.00(1) Å compared to 2.096(6) Å for the four-coordinated Li atom in stilbene bis(lithium · TMEDA) [15], and to 2.22(4) Å for the five-coordinated species in stilbene bis(lithium · PMDTA) [15].

From the crystal structure data, a clear reason for the existence of trimers in the solid state but dimers in solution fails to emerge. In lieu of persuasive electronic or steric effects, it is tempting to assume that packing effects favor the trimeric configuration in the crystalline substance, and that entropy provides the driving force for the dimers in solution.

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