Crystal structure of di(*N*-lithiummethylamino)bis(dimethylamino)silane, $Si(NLiCH_3)_2(N(CH_3)_2)_2$

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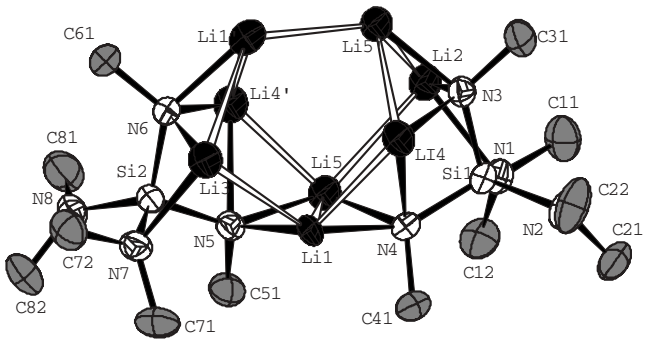

Abstract

$C_{12}H_{36}Li_4N_8Si_2$, monoclinic, *C*12/*c*1 (No. 15), a = 19.468(4) Å, b = 12.870(3) Å, c = 20.568(4) Å, β = 117.88(3)°, V = 4555.3 Å^3, Z = 8, $R_{gt}(F)$ = 0.052, $wR_{ref}(F^2)$ = 0.148, T = 293 K.

Source of material

$Si(NLiCH_3)_2(N(CH_3)_2)_2$ was obtained by reacting silicon tetrachloride first with lithiated dimethylamine and subsequently with lithiated monomethylamine in hexane. Transparent colourless crystals were grown from hexane solution at 238 K. All products are sensitive to air and moisture, so that they are to be handled under dry and oxygen free argon.

Experimental details

All non-hydrogen atoms were refined anisotropically, hydrogen atoms were calculated isotropically with fixed positions.

Table 1. Data collection and handling.

Crystal:	colorless irregular, size 0.2 × 0.5 × 1.0 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.66 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{max}$:	45°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11253, 2991
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2209
$N(param)_{refined}$:	237
Programs:	SHELXS-97 [3], SHELXL-97 [4], DIAMOND [5]

Discussion

$Si(NLiCH_3)_2(N(CH_3)_2)_2$ crystallizes monoclinic in the space group *C*2/*c* and is, in contrast to dimeric $Si(NLiR)_2(N(CH_3)_2)_2$, R = CMe_3 [1], $SiMe_3$ [2], tetrameric in the solid state. In each tetramer eight lithium atoms cluster together to form the structure motive of realgar, As_4S_4. d(Li—Li) = 239.3(8) pm (shortest), 280.7(8) pm (longest). Together with half of the nitrogen atoms a topological interesting Li_8N_8-skeleton is built up: d(Li—N) = 201.5(6) pm (shortest), 229.6(6) pm (longest).

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(11A)	8*f*	0.3325	0.3281	0.1592	0.127
H(11B)	8*f*	0.3975	0.2853	0.1417	0.127
H(11C)	8*f*	0.4202	0.3389	0.2175	0.127
H(12A)	8*f*	0.2921	0.2274	0.2337	0.116
H(12B)	8*f*	0.3739	0.2583	0.2974	0.116
H(12C)	8*f*	0.3485	0.1414	0.2850	0.116
H(21A)	8*f*	0.1800	0.1646	0.0112	0.117
H(21B)	8*f*	0.2332	0.2353	0.0780	0.117
H(21C)	8*f*	0.1935	0.1367	0.0905	0.117
H(22A)	8*f*	0.2179	0.0532	–0.0282	0.154
H(22B)	8*f*	0.2560	–0.0371	0.0287	0.154
H(22C)	8*f*	0.3064	0.0277	0.0021	0.154
H(31A)	8*f*	0.4333	0.0804	0.0468	0.115
H(31B)	8*f*	0.5119	0.1266	0.1071	0.115
H(31C)	8*f*	0.4338	0.1865	0.0844	0.115
H(41A)	8*f*	0.2916	–0.1140	0.2292	0.098
H(41B)	8*f*	0.2491	–0.0671	0.1497	0.098
H(41C)	8*f*	0.2670	0.0033	0.2179	0.098
H(51A)	8*f*	0.3897	–0.0767	0.3651	0.098
H(51B)	8*f*	0.4572	–0.1229	0.4373	0.098
H(51C)	8*f*	0.3883	–0.1942	0.3845	0.098
H(61A)	8*f*	0.6997	–0.2643	0.4142	0.131
H(61B)	8*f*	0.6795	–0.3110	0.3369	0.131
H(61C)	8*f*	0.6569	–0.3716	0.3902	0.131
H(71A)	8*f*	0.3888	–0.4223	0.3614	0.125
H(71B)	8*f*	0.3675	–0.4519	0.2802	0.125
H(71C)	8*f*	0.3628	–0.3358	0.3008	0.125
H(72A)	8*f*	0.5073	–0.5167	0.3923	0.119
H(72B)	8*f*	0.5687	–0.4712	0.3710	0.119
H(72C)	8*f*	0.4948	–0.5281	0.3116	0.119
H(81A)	8*f*	0.6423	–0.2374	0.5684	0.147
H(81B)	8*f*	0.5976	–0.1484	0.5124	0.147
H(81C)	8*f*	0.6654	–0.2074	0.5074	0.147
H(82A)	8*f*	0.5745	–0.3531	0.5598	0.132
H(82B)	8*f*	0.5349	–0.4255	0.4907	0.132
H(82C)	8*f*	0.4890	–0.3311	0.4995	0.132

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si(1)	8*f*	0.36463(5)	0.08144(7)	0.15744(5)	0.0394(5)	0.0428(5)	0.0399(6)	0.0076(4)	0.0130(4)	0.0019(4)
Si(2)	8*f*	0.52367(5)	–0.26924(7)	0.37585(5)	0.0432(6)	0.0449(6)	0.0419(6)	–0.0021(4)	0.0201(4)	0.0076(4)
N(1)	8*f*	0.3791(2)	0.1951(2)	0.2110(2)	0.056(2)	0.044(2)	0.054(2)	0.008(1)	0.020(2)	–0.001(1)
N(4)	8*f*	0.3617(2)	–0.0248(2)	0.2050(2)	0.032(1)	0.051(2)	0.049(2)	0.004(1)	0.018(1)	0.004(1)
N(3)	8*f*	0.4469(2)	0.0635(2)	0.1491(1)	0.044(2)	0.048(2)	0.040(2)	0.005(1)	0.020(1)	0.006(1)
N(2)	8*f*	0.2835(2)	0.1034(2)	0.0742(2)	0.050(2)	0.059(2)	0.049(2)	0.017(1)	0.006(2)	–0.002(2)
N(5)	8*f*	0.4644(2)	–0.1642(2)	0.3461(2)	0.049(2)	0.046(2)	0.042(2)	0.003(1)	0.026(1)	0.005(1)
N(6)	8*f*	0.5865(2)	–0.2501(2)	0.3412(2)	0.035(2)	0.048(2)	0.049(2)	0.003(1)	0.018(1)	0.010(1)
N(7)	8*f*	0.4721(2)	–0.3844(2)	0.3343(2)	0.052(2)	0.045(2)	0.065(2)	–0.008(1)	0.032(2)	0.001(1)
N(8)	8*f*	0.5680(2)	–0.2897(3)	0.4693(2)	0.061(2)	0.069(2)	0.045(2)	–0.009(2)	0.018(2)	0.016(2)
C(11)	8*f*	0.3826(3)	0.2954(3)	0.1797(3)	0.103(4)	0.048(2)	0.089(3)	0.001(2)	0.033(3)	0.001(2)
C(12)	8*f*	0.3455(3)	0.2065(4)	0.2611(3)	0.085(3)	0.077(3)	0.078(3)	0.017(2)	0.045(3)	–0.013(2)
C(21)	8*f*	0.2170(2)	0.1651(4)	0.0625(3)	0.052(2)	0.095(3)	0.073(3)	0.023(2)	0.017(2)	0.010(2)
C(22)	8*f*	0.2643(3)	0.0306(4)	0.0141(3)	0.099(4)	0.097(4)	0.059(3)	0.035(3)	–0.009(3)	–0.018(3)
C(31)	8*f*	0.4574(3)	0.1191(4)	0.0919(3)	0.081(3)	0.089(3)	0.068(3)	0.020(2)	0.041(2)	0.029(2)
C(41)	8*f*	0.2857(2)	–0.0531(3)	0.2000(2)	0.044(2)	0.071(3)	0.079(3)	0.003(2)	0.027(2)	0.009(2)
C(51)	8*f*	0.4211(2)	–0.1371(3)	0.3868(2)	0.074(3)	0.075(3)	0.063(3)	0.008(2)	0.045(2)	0.003(2)
C(61)	8*f*	0.6623(2)	–0.3039(4)	0.3734(3)	0.050(2)	0.111(4)	0.105(4)	0.026(2)	0.040(3)	0.053(3)
C(71)	8*f*	0.3908(3)	–0.3999(4)	0.3177(3)	0.070(3)	0.083(3)	0.106(4)	–0.025(2)	0.050(3)	–0.007(3)
C(72)	8*f*	0.5144(3)	–0.4838(3)	0.3540(3)	0.100(4)	0.051(2)	0.096(4)	0.004(2)	0.053(3)	0.006(2)
C(81)	8*f*	0.6229(3)	–0.2144(4)	0.5185(3)	0.098(3)	0.110(3)	0.080(2)	–0.020(2)	0.036(2)	0.012(2)
C(82)	8*f*	0.5392(3)	–0.3553(4)	0.5081(3)	0.121(4)	0.087(3)	0.069(3)	–0.009(3)	0.055(3)	0.019(3)
Li(1)	8*f*	0.3987(3)	–0.1729(4)	0.2367(3)	0.042(3)	0.048(3)	0.045(3)	0.002(2)	0.019(3)	–0.002(2)
Li(2)	4*e*	1/2	0.1487(6)	1/4	0.045(4)	0.054(5)	0.046(5)	0	0.016(4)	0
Li(3)	4*e*	1/2	–0.3343(6)	1/4	0.051(5)	0.048(4)	0.054(5)	0	0.024(4)	0
Li(4)	8*f*	0.4228(3)	–0.0935(4)	0.1441(3)	0.052(3)	0.045(3)	0.052(3)	0.004(3)	0.023(3)	–0.003(3)
Li(5)	8*f*	0.4514(3)	–0.0151(4)	0.3089(3)	0.043(3)	0.052(3)	0.048(3)	–0.002(2)	0.018(3)	0.000(3)

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