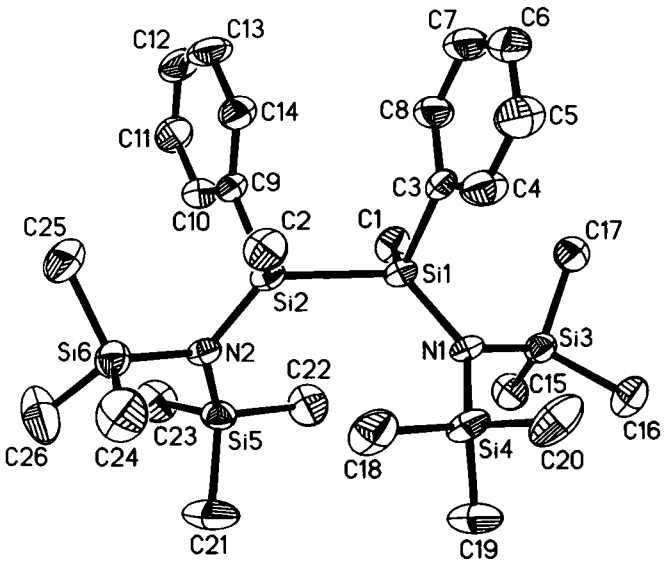
Crystal structure of 1,2-bis[bis(trimethylsilyl)amino]-1,2-dimethyl-1,2-diphenyl-disilane, $\langle\{[(CH_3)_3Si]_2N\}(CH_3)(C_6H_5)Si\rangle_2$

E. Popowski*, I. Rietz, H. Reinke and M. Köckerling

Universität Rostock, Institut für Chemie, Albert-Einstein-Str. 3a, 18051 Rostock, Germany

Received December 4, 2004, accepted and available on-line January 11, 2005; CCDC no. 1267/1439



Abstract

$C_{26}H_{52}N_2Si_6$, triclinic, $P\bar{1}$ (no. 2), $a = 11.179(1)$ Å, $b = 11.907(1)$ Å, $c = 14.761(2)$ Å, $\alpha = 88.94(1)°$, $\beta = 78.80(1)°$, $\gamma = 64.14(1)°$, $V = 1729.5$ Å^3, $Z = 2$, $R_{gt}(F) = 0.068$, $wR_{ref}(F^2) = 0.180$, $T = 293$ K.

Source of material

The disilane was prepared by reaction of $[(Me_3Si)_2N]MePhSiLi$ (Me = CH_3, Ph = C_6H_5) with $[(Me_3Si)_2N]MePhSiCl$ at –78 °C in tetrahydrofuran [1]. Colorless single crystals of the compound were obtained by crystallization from *n*-pentane.

Discussion

The Si1—Si2 bond distance (2.416(2) Å) of the disilane is slightly longer in comparison to disilanes with relatively little steric strain (2.33 Å – 2.37 Å [2,3]), due to the steric bulkiness of both $(Me_3Si)_2N$ substituents. The Si—N bond distances, ranging from 1.747(4) Å to 1.775(5) Å, are within the normal range for acyclic silazanes [2,4]. The sum of the bond angles around the nitrogen atoms (N1: 360°, N2: 359.9°) shows that the geometry at the nitrogen atoms is planar. It is interesting to note that the conformation of the two methyl groups at the Si atoms is approximately *trans* (dihedral angle 149.3°), whereas that of the larger phenyl and bis(trimethylsilyl)amino substituents is nearly *gauche* (∠N1–Si1–Si2–N2 = 50.7°, ∠C3–Si1–Si2–C9 = 68.6°).

* Correspondence author
(e-mail: eckhard.popowski@chemie.uni-rostock.de)

Table 1. Data collection and handling.

Crystal:	colorless prism fragment, size 0.2 x 0.24 x 0.44 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.58 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω
$2\theta_{max}$:	45.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5265, 4434
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2597
$N(param)_{refined}$:	307
Programs:	SHELX-97 [5], DIAMOND [6]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	0.2117	0.1441	0.0569	0.100
H(1B)	2*i*	0.2222	0.0092	0.0457	0.100
H(1C)	2*i*	0.3442	0.0270	0.0688	0.100
H(2A)	2*i*	0.1769	–0.2374	0.3678	0.114
H(2B)	2*i*	0.0597	–0.1346	0.3270	0.114
H(2C)	2*i*	0.1238	–0.0957	0.3990	0.114
H(4A)	2*i*	–0.0801	0.1745	0.3446	0.101
H(5A)	2*i*	–0.3045	0.2123	0.3548	0.124
H(6A)	2*i*	–0.3681	0.1661	0.2278	0.120
H(7A)	2*i*	–0.2118	0.0744	0.0953	0.111
H(8A)	2*i*	0.0108	0.0332	0.0857	0.089
H(10A)	2*i*	0.4521	–0.2737	0.0737	0.083
H(11A)	2*i*	0.4452	–0.3844	–0.0494	0.104
H(12A)	2*i*	0.2496	–0.4008	–0.0565	0.112
H(13A)	2*i*	0.0636	–0.3118	0.0613	0.109
H(14A)	2*i*	0.0698	–0.2032	0.1870	0.087
H(15A)	2*i*	0.3609	0.2449	0.0982	0.115
H(15B)	2*i*	0.3719	0.3185	0.1808	0.115
H(15C)	2*i*	0.3007	0.3913	0.1020	0.115
H(16A)	2*i*	–0.0173	0.4928	0.3163	0.129
H(16B)	2*i*	0.0558	0.5520	0.2434	0.129
H(16C)	2*i*	0.1268	0.4792	0.3222	0.129
H(17A)	2*i*	0.0648	0.2905	0.0817	0.124
H(17B)	2*i*	0.0258	0.4339	0.0903	0.124
H(17C)	2*i*	–0.0569	0.3788	0.1592	0.124
H(18A)	2*i*	0.3156	–0.0053	0.4016	0.152
H(18B)	2*i*	0.1610	0.0313	0.4382	0.152
H(18C)	2*i*	0.2412	0.0676	0.4988	0.152
H(19A)	2*i*	0.4044	0.2023	0.3518	0.148
H(19B)	2*i*	0.3307	0.2571	0.4538	0.148
H(19C)	2*i*	0.2937	0.3418	0.3713	0.148
H(20A)	2*i*	–0.0479	0.2930	0.4541	0.166
H(20B)	2*i*	–0.0046	0.4018	0.4388	0.166
H(20C)	2*i*	0.0324	0.3172	0.5214	0.166
H(21A)	2*i*	0.5678	–0.0994	0.3829	0.175
H(21B)	2*i*	0.7064	–0.1268	0.3161	0.175
H(21C)	2*i*	0.6821	–0.2371	0.3616	0.175
H(22A)	2*i*	0.4464	0.0380	0.2196	0.143

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(22B)	2*i*	0.4917	–0.0368	0.1230	0.143
H(22C)	2*i*	0.5960	–0.0086	0.1638	0.143
H(23A)	2*i*	0.6778	–0.3016	0.0941	0.151
H(23B)	2*i*	0.7547	–0.3702	0.1719	0.151
H(23C)	2*i*	0.7791	–0.2600	0.1262	0.151
H(24A)	2*i*	0.4486	–0.2640	0.4788	0.171
H(24B)	2*i*	0.4410	–0.3920	0.4902	0.171

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(24D)	2*i*	0.3150	–0.2732	0.4716	0.171
H(25A)	2*i*	0.4388	–0.4792	0.2262	0.169
H(25D)	2*i*	0.3086	–0.4152	0.3050	0.169
H(25B)	2*i*	0.4346	–0.5339	0.3236	0.169
H(26D)	2*i*	0.6992	–0.4841	0.2524	0.201
H(26A)	2*i*	0.6726	–0.5330	0.3501	0.201
H(26B)	2*i*	0.7026	–0.4159	0.3410	0.201

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)	2*i*	0.1742(2)	0.0712(2)	0.2063(1)	0.056(1)	0.053(1)	0.0413(9)	–0.0313(9)	–0.0095(8)	–0.0011(8)
Si(2)	2*i*	0.2721(2)	–0.1417(2)	0.2502(1)	0.060(1)	0.056(1)	0.053(1)	–0.038(1)	–0.0110(9)	0.0048(8)
Si(3)	2*i*	0.1500(2)	0.3334(2)	0.2046(1)	0.064(1)	0.053(1)	0.061(1)	–0.032(1)	–0.017(1)	0.0047(9)
Si(4)	2*i*	0.1861(2)	0.2082(2)	0.3820(1)	0.105(2)	0.075(1)	0.045(1)	–0.061(1)	–0.015(1)	0.0001(9)
Si(5)	2*i*	0.5705(2)	–0.1769(2)	0.2363(1)	0.061(1)	0.071(1)	0.070(1)	–0.040(1)	–0.023(1)	0.015(1)
Si(6)	2*i*	0.4761(2)	–0.3566(2)	0.3311(1)	0.083(2)	0.060(1)	0.068(1)	–0.040(1)	–0.023(1)	0.017(1)
N(1)	2*i*	0.1732(5)	0.1987(4)	0.2665(3)	0.057(3)	0.053(3)	0.043(3)	–0.036(3)	–0.010(2)	–0.003(2)
N(2)	2*i*	0.4376(5)	–0.2192(4)	0.2716(3)	0.061(3)	0.058(3)	0.047(3)	–0.039(3)	–0.015(2)	0.004(2)
C(1)	2*i*	0.2470(7)	0.0617(6)	0.0788(4)	0.085(5)	0.065(4)	0.049(4)	–0.036(4)	–0.005(4)	0.001(3)
C(2)	2*i*	0.1421(7)	–0.1538(6)	0.3480(5)	0.079(5)	0.082(5)	0.083(5)	–0.053(4)	–0.010(4)	0.021(4)
C(3)	2*i*	–0.0084(6)	0.0997(5)	0.2147(4)	0.060(4)	0.043(4)	0.058(4)	–0.025(3)	–0.012(3)	–0.008(3)
C(4)	2*i*	–0.1053(8)	0.1533(7)	0.2935(5)	0.067(5)	0.107(6)	0.080(5)	–0.044(5)	–0.003(4)	–0.029(5)
C(5)	2*i*	–0.2409(8)	0.1773(8)	0.3000(6)	0.070(6)	0.124(7)	0.107(7)	–0.045(5)	0.011(5)	–0.047(6)
C(6)	2*i*	–0.2778(8)	0.1486(7)	0.2250(7)	0.064(6)	0.090(6)	0.153(9)	–0.038(5)	–0.026(6)	–0.021(6)
C(7)	2*i*	–0.1853(9)	0.0949(8)	0.1461(6)	0.083(6)	0.103(7)	0.103(7)	–0.045(5)	–0.034(5)	–0.019(5)
C(8)	2*i*	–0.0519(7)	0.0704(6)	0.1406(5)	0.071(5)	0.084(5)	0.073(5)	–0.034(4)	–0.026(4)	–0.007(4)
C(9)	2*i*	0.2631(7)	–0.2269(5)	0.1465(4)	0.062(4)	0.047(4)	0.062(4)	–0.031(3)	–0.020(4)	0.004(3)
C(10)	2*i*	0.3732(7)	–0.2815(6)	0.0728(5)	0.074(5)	0.069(5)	0.074(5)	–0.034(4)	–0.029(4)	0.001(4)
C(11)	2*i*	0.3690(9)	–0.3472(7)	–0.0018(5)	0.099(6)	0.078(5)	0.077(5)	–0.032(5)	–0.024(5)	–0.017(4)
C(12)	2*i*	0.253(1)	–0.3575(7)	–0.0059(6)	0.134(8)	0.083(6)	0.089(6)	–0.059(6)	–0.052(6)	–0.005(5)
C(13)	2*i*	0.142(1)	–0.3046(8)	0.0639(6)	0.119(8)	0.105(7)	0.095(6)	–0.076(6)	–0.058(6)	0.012(5)
C(14)	2*i*	0.1465(7)	–0.2393(6)	0.1398(5)	0.081(5)	0.071(5)	0.084(5)	–0.046(4)	–0.027(4)	0.012(4)
C(15)	2*i*	0.3160(7)	0.3205(6)	0.1384(5)	0.081(5)	0.075(5)	0.090(5)	–0.047(4)	–0.020(4)	0.027(4)
C(16)	2*i*	0.0689(8)	0.4826(6)	0.2810(5)	0.110(6)	0.065(5)	0.086(5)	–0.042(5)	–0.017(5)	–0.005(4)
C(17)	2*i*	0.0315(7)	0.3627(6)	0.1242(5)	0.092(6)	0.072(5)	0.091(5)	–0.035(4)	–0.039(5)	0.013(4)
C(18)	2*i*	0.2314(9)	0.0572(7)	0.4368(4)	0.170(9)	0.096(6)	0.054(4)	–0.070(6)	–0.029(5)	0.009(4)
C(19)	2*i*	0.3199(8)	0.2584(8)	0.3908(5)	0.131(7)	0.141(7)	0.079(5)	–0.098(6)	–0.054(5)	0.029(5)
C(20)	2*i*	0.0214(9)	0.3185(7)	0.4584(5)	0.158(9)	0.116(7)	0.068(5)	–0.086(7)	0.022(5)	–0.033(5)
C(21)	2*i*	0.6401(9)	–0.1577(9)	0.3363(5)	0.129(8)	0.177(9)	0.106(6)	–0.109(7)	–0.062(6)	0.027(6)
C(22)	2*i*	0.5201(7)	–0.0282(7)	0.1787(6)	0.077(6)	0.090(6)	0.141(7)	–0.059(5)	–0.020(5)	0.039(5)
C(23)	2*i*	0.7129(7)	–0.2911(7)	0.1461(5)	0.072(6)	0.117(7)	0.104(6)	–0.041(5)	–0.002(5)	0.007(5)
C(24)	2*i*	0.413(1)	–0.3166(8)	0.4583(5)	0.175(9)	0.114(7)	0.062(5)	–0.072(7)	–0.025(6)	0.027(5)
C(25)	2*i*	0.4061(9)	–0.4586(7)	0.2917(6)	0.154(9)	0.081(6)	0.146(8)	–0.078(6)	–0.061(7)	0.029(5)
C(26)	2*i*	0.6596(8)	–0.4598(8)	0.3170(7)	0.089(7)	0.101(7)	0.20(1)	–0.032(6)	–0.035(7)	0.069(7)

References

1. Rietz, I.; Popowski, E.; Reinke, H.; Michalik, M.: Reaktionen von Bis(trimethylsily)amino-substituierten Chlorsilanen $[(Me_3Si)_2N]Me_{2-n}Ph_nSiCl$ ($n = 0, 1, 2$) mit Lithium – Darstellung von Bis(trimethylsilyl)amino-substituierten Silyllithiumverbindungen und Disilanen. J. Organomet. Chem. **556** (1998) 67-64.
2. Sheldrick, W. S.: Structural Chemistry of organic silicon compounds. In: The Chemistry of Organic Silicon Compounds (Eds. S. Patai, Z. Rappoport), pp. 227-303. J. Wiley and Sons, Chichester 1989.
3. Lukevics, E.; Pudova, O.: Molecular Structure of Compounds with Silicon-Silicon Bonds. Main Group Met. Chem. **21** (1998) 123-183.
4. Sklover, V. E.; Struchkov, Yu. T.: Recent Developments in Structural Chemistry of Organosilicon Compounds. J. Organomet. Chem. **322** (1987) 269-298.
5. Sheldrick, G. M.: SHELX-97. Programs for the Analysis of Crystal Structures. University of Göttingen, Germany 1997.
6. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 2.0f. Crystal Impact GbR, Bonn, Germany 1998.