Crystal structure of tetrakis(phenylamino)silane, $Si(C_6H_5NH)_4$

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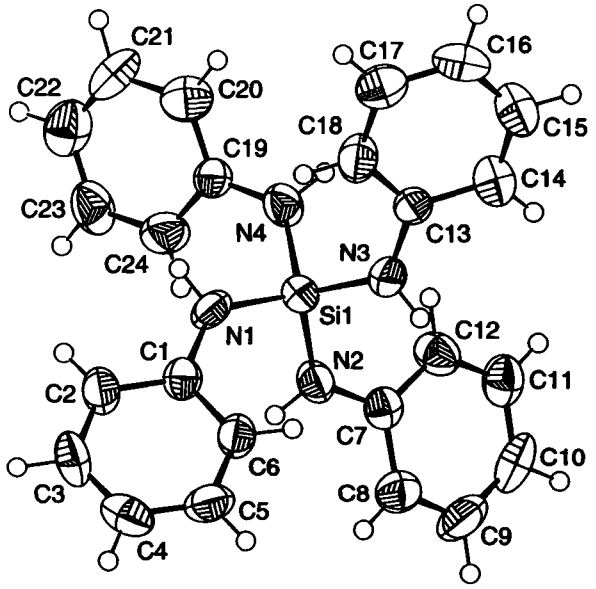

Abstract

$C_{24}H_{24}N_4Si$, monoclinic, $C12/c1$ (no. 15),
$a = 16.217(3)$ Å, $b = 16.409(4)$ Å, $c = 17.302(4)$ Å,
$\beta = 110.14(2)°$, $V = 4322.7$ Å^3, $Z = 8$,
$R_{gt}(F) = 0.077$, $wR_{obs}(F^2) = 0.186$, $T = 293$ K.

Source of material

The compound was synthesized according to the method of Reynolds [1] by reacting silicon tetrachloride with aniline in diethyl ether as a solvent. Crystals were obtained by recrystallizing the crude product from carbon disulfide. A single crystal was sealed in a glass capillary under argon atmosphere for the crystal structure determination.

Discussion

Tetrakis-(phenylamino)silane crystallizes monoclinically with one molecule per asymmetric unit. The Si—N bond lengths correspond to those found in tetrakis(methylamino)silane [2] and tetrakis(pentafluorophenylamino)silane [3]. The N–Si–N bond angles partly show considerable deviations from the tetrahedral angle, resulting in a flattened tetrahedron, which has been similarly found in other silazanes and reflects the center undercoordination of silicon.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.4 × 0.5 × 0.8 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.26 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$ (profile fit)
$2\theta_{max}$:	42°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4303, 2324
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1445
$N(param)_{refined}$:	263
Programs:	SHELXS-86 [4], SHELXL-93 [5]

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

Atom	Site	x	y	z	U_{iso}
H(1N)	8*f*	0.1178	0.2476	0.2027	0.064
H(2N)	8*f*	0.3100	0.3361	0.3061	0.064
H(3N)	8*f*	0.3559	0.1859	0.1942	0.064
H(4N)	8*f*	0.2491	0.0979	0.3098	0.064
H(2)	8*f*	0.0380	0.3734	0.1702	0.064
H(3)	8*f*	0.0200	0.4963	0.1018	0.064
H(4)	8*f*	0.1232	0.5405	0.0475	0.064
H(5)	8*f*	0.2456	0.4624	0.0629	0.064
H(6)	8*f*	0.2657	0.3408	0.1340	0.064
H(8)	8*f*	0.4531	0.4004	0.3372	0.064
H(9)	8*f*	0.5982	0.3927	0.4119	0.064
H(10)	8*f*	0.6602	0.2707	0.4693	0.064
H(11)	8*f*	0.5734	0.1561	0.4529	0.064
H(12)	8*f*	0.4255	0.1634	0.3758	0.064
H(14)	8*f*	0.4078	0.0420	0.1838	0.064
H(15)	8*f*	0.3632	−0.0794	0.1148	0.064
H(16)	8*f*	0.2186	−0.1092	0.0514	0.064
H(17)	8*f*	0.1150	−0.0144	0.0528	0.064
H(18)	8*f*	0.1565	0.1092	0.1171	0.064
H(20)	8*f*	0.1042	0.0449	0.3145	0.064
H(21)	8*f*	0.0036	0.0608	0.3807	0.064
H(22)	8*f*	−0.0015	0.1773	0.4499	0.064
H(23)	8*f*	0.0993	0.2783	0.4578	0.064
H(24)	8*f*	0.2017	0.2634	0.3941	0.064

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*	0.2588(1)	0.2170(1)	0.2534(1)	0.043(1)	0.036(1)	0.048(1)	0.002(1)	0.018(1)	−0.001(1)
N(1)	8*f*	0.1655(4)	0.2702(4)	0.2023(4)	0.036(4)	0.053(4)	0.058(4)	−0.008(3)	0.021(3)	0.006(4)
N(2)	8*f*	0.3318(4)	0.2883(4)	0.3060(4)	0.043(4)	0.042(4)	0.054(4)	0.008(3)	0.012(3)	−0.002(3)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(3)	8*f*	0.3094(4)	0.1637(4)	0.1980(4)	0.048(4)	0.047(4)	0.070(5)	–0.003(3)	0.034(4)	–0.007(4)
N(4)	8*f*	0.2251(4)	0.1451(4)	0.3071(4)	0.062(4)	0.045(4)	0.057(4)	0.008(3)	0.034(4)	0.004(3)
C(1)	8*f*	0.1532(5)	0.3446(5)	0.1597(5)	0.038(5)	0.049(5)	0.041(5)	0.003(4)	0.009(4)	0.001(4)
C(2)	8*f*	0.0805(5)	0.3912(5)	0.1493(5)	0.052(5)	0.054(6)	0.061(6)	0.013(5)	0.026(5)	0.006(5)
C(3)	8*f*	0.0694(6)	0.4647(6)	0.1078(6)	0.057(6)	0.065(7)	0.077(7)	0.031(5)	0.023(5)	0.008(6)
C(4)	8*f*	0.1310(6)	0.4912(5)	0.0756(5)	0.075(7)	0.039(5)	0.060(6)	–0.001(5)	0.006(5)	–0.003(4)
C(5)	8*f*	0.2039(6)	0.4447(5)	0.0850(6)	0.059(6)	0.052(6)	0.072(7)	–0.015(5)	0.025(5)	0.006(5)
C(6)	8*f*	0.2157(5)	0.3718(5)	0.1272(5)	0.046(5)	0.052(6)	0.058(6)	0.008(4)	0.025(5)	0.009(4)
C(7)	8*f*	0.4223(5)	0.2823(5)	0.3496(4)	0.047(5)	0.048(5)	0.036(4)	0.004(5)	0.019(4)	0.003(4)
C(8)	8*f*	0.4770(6)	0.3510(5)	0.3607(5)	0.058(6)	0.054(6)	0.067(6)	–0.002(5)	0.028(5)	0.011(5)
C(9)	8*f*	0.5633(6)	0.3464(7)	0.4047(6)	0.052(6)	0.083(8)	0.094(8)	–0.026(6)	0.035(6)	–0.013(7)
C(10)	8*f*	0.6004(6)	0.2736(8)	0.4393(6)	0.034(5)	0.116(9)	0.067(6)	0.002(7)	0.022(5)	0.001(7)
C(11)	8*f*	0.5489(6)	0.2053(6)	0.4292(5)	0.049(6)	0.071(7)	0.061(6)	0.013(5)	0.017(5)	0.009(5)
C(12)	8*f*	0.4601(5)	0.2100(5)	0.3835(5)	0.055(6)	0.047(6)	0.056(5)	0.003(5)	0.007(5)	0.002(5)
C(13)	8*f*	0.2858(5)	0.0892(4)	0.1581(5)	0.046(5)	0.039(5)	0.045(5)	0.000(4)	0.024(4)	0.005(4)
C(14)	8*f*	0.3483(6)	0.0316(6)	0.1571(5)	0.058(6)	0.061(6)	0.060(6)	0.012(5)	0.019(5)	0.000(5)
C(15)	8*f*	0.3211(7)	–0.0415(6)	0.1160(6)	0.067(7)	0.061(7)	0.072(7)	0.019(5)	0.020(5)	–0.002(5)
C(16)	8*f*	0.2355(7)	–0.0594(5)	0.0778(5)	0.090(8)	0.040(5)	0.050(6)	–0.014(6)	0.021(6)	–0.001(4)
C(17)	8*f*	0.1743(6)	–0.0030(6)	0.0788(5)	0.062(6)	0.065(6)	0.055(6)	–0.013(6)	0.017(5)	–0.002(5)
C(18)	8*f*	0.1993(5)	0.0711(5)	0.1180(5)	0.043(5)	0.065(6)	0.062(6)	0.000(5)	0.017(5)	–0.018(5)
C(19)	8*f*	0.1645(5)	0.1519(5)	0.3472(5)	0.037(5)	0.043(5)	0.047(5)	0.001(4)	0.010(4)	0.003(4)
C(20)	8*f*	0.1044(6)	0.0926(6)	0.3436(5)	0.064(6)	0.063(6)	0.049(5)	–0.011(5)	0.010(5)	–0.007(5)
C(21)	8*f*	0.0432(6)	0.1025(7)	0.3832(6)	0.046(6)	0.100(9)	0.070(7)	–0.021(6)	0.026(5)	0.002(6)
C(22)	8*f*	0.0402(6)	0.1709(7)	0.4246(6)	0.071(7)	0.091(8)	0.060(7)	0.010(6)	0.034(5)	0.019(6)
C(23)	8*f*	0.1000(6)	0.2307(6)	0.4288(6)	0.087(7)	0.064(7)	0.067(7)	0.019(6)	0.047(6)	0.003(5)
C(24)	8*f*	0.1617(6)	0.2217(6)	0.3905(5)	0.075(6)	0.053(6)	0.073(6)	–0.008(5)	0.040(5)	0.004(5)

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