Crystal structure of *N*-1-adamantylbis[bis(trimethylsilyl)amido]-germanimine, C₂₂H₅₁GeN₃Si₄

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Received February 2, 2000, CCDC-No. 1267/397

Abstract

C₂₂H₅₁GeN₃Si₄, triclinic, $P\bar{1}$ (No. 2), $a = 10.0862(6)$ Å, $b = 11.3809(7)$ Å, $c = 14.9594(8)$ Å, $\alpha = 73.849(6)^\circ$, $\beta = 82.902(7)^\circ$, $\gamma = 67.437(7)^\circ$, $V = 1522.9$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.026$, $wR_{\text{ref}}(F^2) = 0.067$, $T = 193$ K.

Source of material

Treatment of germanium bis(trimethylsilyl)amide with 1-adamantyl azide at room temperature furnishes the title compound in 65 % yield.

Discussion

The crystal structure analysis reveals a strictly planar environment of the germanium atom by the nitrogen atoms (sum of angles: 359.8°). The Ge=N bond length of 1.701(1) Å is comparable with those of other germanimines with a similar substitution pattern [1–3]. The angle Ge=N1–C1 angle is widened (130.13°) presumably to reduce repulsion between the bulky substituents.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.45 × 0.63 × 0.90 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	11.77 cm ⁻¹
Diffractometer, scan mode:	Stoe IPDS, 200 exposures, $\Delta\phi = 1^\circ$
$2\theta_{\text{max}}$:	51.98°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	18553, 5531
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 4770
$N(\text{param})_{\text{refined}}$:	271
Programs:	SHELXS-97 [4], SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(2A)	2i	0.8946	0.2856	0.0484	0.033
H(2B)	2i	0.7628	0.3742	0.1013	0.033
H(3)	2i	0.6874	0.3877	-0.0475	0.037
H(4A)	2i	0.5099	0.4010	0.0753	0.042
H(4B)	2i	0.4802	0.3311	0.0045	0.042
H(5)	2i	0.4640	0.2098	0.1599	0.039
H(6A)	2i	0.6266	-0.0020	0.1436	0.042
H(6B)	2i	0.5519	0.0845	0.0459	0.042
H(7)	2i	0.8052	-0.0165	0.0220	0.036
H(8A)	2i	0.9651	0.0394	0.0913	0.032
H(8B)	2i	0.8767	-0.0283	0.1711	0.032
H(9A)	2i	0.6885	0.1930	-0.0805	0.041
H(9B)	2i	0.8495	0.1757	-0.0632	0.041
H(10A)	2i	0.6712	0.1083	0.2550	0.033
H(10B)	2i	0.6250	0.2650	0.2291	0.033
H(11A)	2i	1.1690	-0.1302	0.4083	0.073
H(11B)	2i	1.2461	-0.1916	0.3229	0.073
H(11C)	2i	1.0823	-0.0923	0.3154	0.073
H(12A)	2i	1.2837	0.1367	0.1384	0.053
H(12B)	2i	1.1579	0.0843	0.1370	0.053
H(12C)	2i	1.3216	-0.0153	0.1445	0.053
H(13A)	2i	1.4615	0.0445	0.3061	0.069
H(13B)	2i	1.4868	-0.1038	0.3078	0.069
H(13C)	2i	1.4272	-0.0509	0.3990	0.069
H(14A)	2i	1.3019	-0.0191	0.5080	0.092
H(14B)	2i	1.1391	0.0256	0.5457	0.092
H(14C)	2i	1.2512	0.0720	0.5797	0.092
H(15A)	2i	1.2991	0.3395	0.3456	0.090
H(15B)	2i	1.4104	0.1920	0.3804	0.090
H(15C)	2i	1.3474	0.2830	0.4519	0.090
H(16A)	2i	0.9903	0.4105	0.4235	0.059
H(16B)	2i	1.0535	0.3443	0.5261	0.059
H(16C)	2i	0.9415	0.2981	0.4920	0.059
H(17A)	2i	1.0599	0.3985	0.0898	0.067
H(17B)	2i	1.1712	0.3698	0.1678	0.067
H(17C)	2i	1.1415	0.4970	0.0821	0.067
H(18A)	2i	0.9513	0.6618	0.2957	0.104

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(18B)	2i	1.0717	0.6670	0.2159	0.104
H(18C)	2i	1.1005	0.5399	0.3016	0.104
H(19A)	2i	0.7751	0.6372	0.0745	0.093
H(19B)	2i	0.8673	0.7268	0.0679	0.093
H(19C)	2i	0.7424	0.7282	0.1452	0.093
H(20A)	2i	0.7010	0.6893	0.3304	0.084
H(20B)	2i	0.7555	0.5772	0.4244	0.084

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(20C)	2i	0.5874	0.6478	0.4039	0.084
H(21A)	2i	0.5614	0.6372	0.1825	0.078
H(21B)	2i	0.4542	0.5968	0.2622	0.078
H(21C)	2i	0.5458	0.4978	0.2000	0.078
H(22A)	2i	0.6660	0.2832	0.3732	0.064
H(22B)	2i	0.5648	0.3842	0.4316	0.064
H(22C)	2i	0.7329	0.3134	0.4520	0.064

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ge(1)	2i	0.95411(2)	0.25456(2)	0.27259(1)	0.02054(9)	0.0196(1)	0.0214(1)	−0.00683(7)	−0.00390(6)	−0.00433(7)
Si(1)	2i	1.23654(5)	0.02742(5)	0.29184(4)	0.0229(2)	0.0248(2)	0.0360(3)	−0.0058(2)	−0.0034(2)	−0.0058(2)
Si(2)	2i	1.17287(5)	0.20140(6)	0.42802(4)	0.0280(2)	0.0455(3)	0.0247(3)	−0.0148(2)	−0.0057(2)	−0.0092(3)
Si(3)	2i	0.94961(6)	0.53592(5)	0.19323(4)	0.0560(3)	0.0250(3)	0.0284(3)	−0.0193(2)	0.0016(2)	−0.0049(2)
Si(4)	2i	0.69477(5)	0.48842(5)	0.31576(4)	0.0311(2)	0.0299(3)	0.0293(3)	−0.0005(2)	−0.0044(2)	−0.0118(2)
N(1)	2i	0.9173(1)	0.1482(1)	0.2284(1)	0.0234(7)	0.0219(7)	0.0323(9)	−0.0068(5)	−0.0084(6)	−0.0076(7)
N(2)	2i	1.1256(1)	0.1690(1)	0.3311(1)	0.0221(6)	0.0257(7)	0.0249(8)	−0.0077(5)	−0.0058(5)	−0.0038(7)
N(3)	2i	0.8665(2)	0.4315(1)	0.2647(1)	0.0330(7)	0.0192(7)	0.0249(8)	−0.0069(6)	−0.0025(6)	−0.0052(7)
C(1)	2i	0.8134(2)	0.1737(2)	0.1592(1)	0.0204(7)	0.0201(8)	0.0272(9)	−0.0068(6)	−0.0039(6)	−0.0069(8)
C(2)	2i	0.7990(2)	0.2941(2)	0.0777(1)	0.0304(8)	0.0221(8)	0.030(1)	−0.0098(7)	−0.0048(7)	−0.0049(8)
C(3)	2i	0.6959(2)	0.3081(2)	0.0046(1)	0.0344(9)	0.0268(9)	0.029(1)	−0.0089(7)	−0.0084(7)	−0.0032(9)
C(4)	2i	0.5480(2)	0.3215(2)	0.0510(2)	0.0270(8)	0.037(1)	0.040(1)	−0.0037(7)	−0.0127(8)	−0.0123(9)
C(5)	2i	0.5608(2)	0.2005(2)	0.1303(1)	0.0204(8)	0.041(1)	0.038(1)	−0.0109(7)	−0.0030(7)	−0.014(1)
C(6)	2i	0.6187(2)	0.0769(2)	0.0923(2)	0.0342(9)	0.039(1)	0.040(1)	−0.0204(8)	−0.0061(8)	−0.012(1)
C(7)	2i	0.7668(2)	0.0637(2)	0.0467(1)	0.0327(9)	0.0268(9)	0.033(1)	−0.0100(7)	−0.0037(7)	−0.0140(9)
C(8)	2i	0.8684(2)	0.0508(2)	0.1197(1)	0.0247(8)	0.0221(8)	0.034(1)	−0.0058(6)	−0.0041(7)	−0.0100(8)
C(9)	2i	0.7541(2)	0.1848(2)	−0.0332(1)	0.038(1)	0.037(1)	0.030(1)	−0.0123(8)	−0.0037(8)	−0.0115(9)
C(10)	2i	0.6635(2)	0.1868(2)	0.2033(1)	0.0241(8)	0.0316(9)	0.029(1)	−0.0101(7)	−0.0010(7)	−0.0095(8)
C(11)	2i	1.1764(2)	−0.1133(2)	0.3404(2)	0.045(1)	0.028(1)	0.067(2)	−0.0115(9)	−0.004(1)	−0.002(1)
C(12)	2i	1.2517(2)	0.0624(2)	0.1626(1)	0.0263(9)	0.045(1)	0.041(1)	−0.0129(8)	0.0017(8)	−0.021(1)
C(13)	2i	1.4256(2)	−0.0272(2)	0.3309(2)	0.0272(9)	0.049(1)	0.052(1)	−0.0043(9)	−0.0084(9)	−0.008(1)
C(14)	2i	1.2221(3)	0.0523(3)	0.5271(2)	0.055(1)	0.072(2)	0.033(1)	−0.003(1)	−0.011(1)	0.001(1)
C(15)	2i	1.3256(3)	0.2611(3)	0.3979(2)	0.048(1)	0.092(2)	0.069(2)	−0.043(1)	0.006(1)	−0.042(2)
C(16)	2i	1.0217(2)	0.3286(2)	0.4727(2)	0.043(1)	0.052(1)	0.030(1)	−0.0180(9)	−0.0041(8)	−0.017(1)
C(17)	2i	1.0982(2)	0.4388(2)	0.1252(2)	0.054(1)	0.039(1)	0.041(1)	−0.025(1)	0.012(1)	−0.005(1)
C(18)	2i	1.0275(4)	0.6099(3)	0.2594(2)	0.116(2)	0.069(2)	0.055(2)	−0.070(2)	0.008(2)	−0.018(2)
C(19)	2i	0.8180(3)	0.6733(2)	0.1104(2)	0.087(2)	0.031(1)	0.044(2)	−0.009(1)	0.004(1)	0.006(1)
C(20)	2i	0.6833(3)	0.6158(2)	0.3758(2)	0.058(1)	0.047(1)	0.060(2)	−0.001(1)	−0.003(1)	−0.035(1)
C(21)	2i	0.5464(2)	0.5641(2)	0.2299(2)	0.041(1)	0.051(1)	0.048(1)	0.007(1)	−0.015(1)	−0.015(1)
C(22)	2i	0.6606(2)	0.3511(2)	0.4035(2)	0.034(1)	0.056(1)	0.036(1)	−0.0165(9)	0.0031(8)	−0.010(1)

Acknowledgments. Financial support of our work by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie is gratefully acknowledged.

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