

[*trans*-Pt{OC(CH₃)N(CH₃)₂}₂-*trans*-Cl₂] has been reported (Roat, Yolles & Rheingold, 1986). The title compound was obtained by the treatment of di- μ -chloro-bis[*o*-(*N*-methyliminomethyl)phenyl]dipalladium(II) with AgBF₄ in *N,N*-dimethylformamide (Wu & Heck, 1987).

The crystals were a gift of Dr R. Heck.

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1,3-Di-*tert*-butyl-2,2-dimethyl-5,8-diphenyl-1,3,5,6,7,8-hexaaza-2-sila-4-germaspiro[3.4]oct-6-ene

BY HANS PREUT, RONALD C. OBLOH AND WILHELM P. NEUMANN

Fachbereich Chemie der Universität Dortmund, Postfach 500500, D-4600 Dortmund 50, Federal Republic of Germany

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Abstract. C₂₂H₃₄GeN₆Si, $M_r = 483.23$, monoclinic, $C2/c$, $a = 23.493(8)$, $b = 17.160(22)$, $c = 16.895(20)$ Å, $\beta = 124.44(6)^\circ$, $V = 5617(11)$ Å³, $Z = 8$, $D_x = 1.143$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.13$ mm⁻¹, $F(000) = 2032$, $T = 291(1)$ K, final $R = 0.072$ for 1820 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. In the molecule the central Ge atom is part of a five-membered Ge–N–N–N–N ring and a four-membered Ge–N–Si–N ring. Four N atoms are bound to Ge with Ge–N distances in the range 1.81(1) to 1.83(1) Å and N–Ge–N angles in the range 82.6(6) to 124.7(5)°. The nearly planar rings form a dihedral angle of 88.9(6)°. The two C [Si–C 1.86(2) Å] and two N [Si–N 1.75(1) Å] atoms bound to Si form bond angles C–Si–C, C–Si–N, N–Si–N in the range 87.5(5) to 115.8(7)°.

Experimental. The title compound has been obtained as a by-product from the known gerylene (Veith & Grosser, 1982) and phenyl azide at 298 K. Colourless crystals from THF by slow concentration at 298 K. The major product is 1,3,7,9-tetra-*tert*-butyl-2,2,8,8-tetramethyl-5,10-diphenyl-1,3,5,7,9,10-hexaaza-2,8-disila-4,6-digermadispiro[3.1.3.1]decane. The crystal structure of its tin analogue has been published (Preut, Obloh & Neumann, 1987). Crystal size $\sim 0.1 \times 0.2 \times 0.2$ mm, $\omega/2\theta$ scan, scan speed 1.1–3.3° min⁻¹ in θ , Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 25.6^\circ$; six standard reflections recorded every 2.5 h, only random deviations; 7442 reflections measured, $1.5 \leq \theta \leq 22.0^\circ$, $-24 \leq h \leq 24$, $0 \leq k \leq 18$, $-17 \leq l \leq 17$; after

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averaging ($R_{\text{int}} = 0.03$): 3446 unique reflections, 1820 with $F \geq 4.0\sigma(F)$; Lorentz–polarization correction and absorption correction *via* ψ scans; max./min. transmission 1.00/0.95; systematic absences (hkl) $h + k = 2n + 1$, ($h0l$) $h = 2n + 1$, $l = 2n + 1$ conform to space groups $C2/c$ and Cc ; structure solution *via* Patterson function in space group $C2/c$, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C–H 0.96 Å); phenyl groups were treated as rigid groups (C–C 1.395 Å, C–C–C 120°); refine-

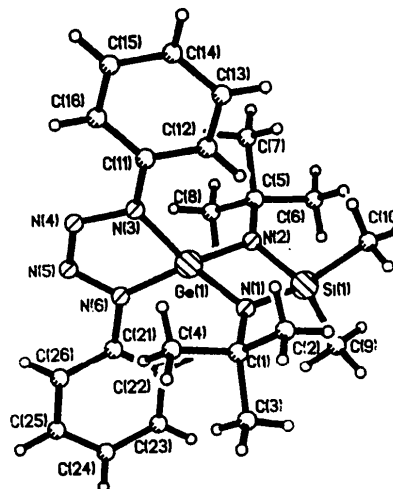


Fig. 1. View of the molecule, showing the atom-numbering scheme.

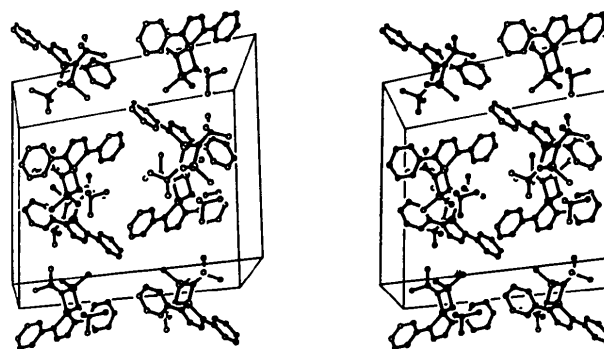
Table 1. Atomic coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	U_{eq}
Ge(1)	0.26764 (9)	0.58237 (9)	0.3078 (1)	4.3 (1)
Si(1)	0.2401 (2)	0.4673 (3)	0.3815 (3)	5.3 (3)
N(1)	0.2169 (6)	0.5658 (6)	0.3564 (8)	4.1 (7)
N(2)	0.2925 (6)	0.4816 (6)	0.3403 (8)	4.5 (8)
N(3)	0.3304 (6)	0.6610 (8)	0.345 (1)	5.5 (8)
N(4)	0.3170 (8)	0.7087 (8)	0.268 (1)	7 (1)
N(5)	0.2656 (8)	0.6879 (9)	0.188 (1)	6 (1)
N(6)	0.2328 (7)	0.6226 (7)	0.188 (1)	5.3 (7)
C(1)	0.1702 (9)	0.6192 (9)	0.362 (1)	5 (1)
C(2)	0.194 (1)	0.620 (1)	0.467 (1)	12 (2)
C(3)	0.0943 (9)	0.597 (1)	0.291 (2)	10 (1)
C(4)	0.1759 (9)	0.6999 (9)	0.333 (1)	8 (2)
C(5)	0.3422 (9)	0.434 (1)	0.336 (1)	7 (1)
C(6)	0.341 (2)	0.352 (1)	0.365 (2)	20 (4)
C(7)	0.412 (1)	0.463 (2)	0.410 (3)	23 (4)
C(8)	0.330 (2)	0.438 (2)	0.243 (2)	32 (5)
C(9)	0.1690 (9)	0.3958 (9)	0.311 (1)	8 (1)
C(10)	0.2876 (9)	0.442 (1)	0.511 (1)	8 (1)
C(12)†	0.3926 (6)	0.6448 (6)	0.5168 (9)	6 (1)
C(13)	0.4451	0.6658	0.6105	8 (1)
C(14)	0.4889	0.7278	0.6267	8 (1)
C(15)	0.4801	0.7688	0.5492	8 (1)
C(16)	0.4276	0.7479	0.4556	7 (1)
C(11)	0.3839	0.6859	0.4394	5 (1)
C(22)†	0.1405 (7)	0.5327 (7)	0.0928 (9)	8 (1)
C(23)	0.0779	0.5126	0.0082	9 (2)
C(24)	0.0455	0.5647	-0.0687	10 (2)
C(25)	0.0756	0.6368	-0.0609	10 (2)
C(26)	0.1381	0.6569	0.0237	7 (1)
C(21)	0.1706	0.6049	0.1005	6 (1)

† Phenyl groups were refined as rigid bodies and e.s.d.'s are given only for one C of each group.

Table 2. Bond distances (\AA) and angles ($^\circ$)

Ge(1)---Si(1)	2.604 (6)	N(3)-N(4)	1.41 (2)
Ge(1)-N(1)	1.81 (2)	N(3)-C(11)	1.43 (2)
Ge(1)-N(2)	1.81 (1)	N(4)-N(5)	1.26 (2)
Ge(1)-N(3)	1.83 (1)	N(5)-N(6)	1.36 (2)
Ge(1)-N(6)	1.83 (1)	N(6)-C(21)	1.41 (2)
Si(1)-N(1)	1.75 (1)	C(1)-C(2)	1.53 (3)
Si(1)-N(2)	1.74 (2)	C(1)-C(3)	1.53 (2)
Si(1)-C(9)	1.86 (2)	C(1)-C(4)	1.50 (2)
Si(1)-C(10)	1.86 (2)	C(5)-C(6)	1.50 (3)
N(1)-C(1)	1.47 (2)	C(5)-C(7)	1.48 (3)
N(2)-C(5)	1.46 (3)	C(5)-C(8)	1.43 (5)
N(2)-Ge(1)-N(1)	83.9 (5)	N(5)-N(4)-N(3)	114.1 (15)
N(3)-Ge(1)-N(1)	124.7 (6)	N(6)-N(5)-N(4)	115.3 (14)
N(3)-Ge(1)-N(2)	122.9 (7)	N(5)-N(6)-Ge(1)	114.6 (11)
N(6)-Ge(1)-N(1)	124.1 (7)	C(21)-N(6)-Ge(1)	129.0 (11)
N(6)-Ge(1)-N(2)	124.2 (6)	C(21)-N(6)-N(5)	115.2 (13)
N(6)-Ge(1)-N(3)	82.6 (6)	C(2)-C(1)-N(1)	106.5 (15)
N(2)-Si(1)-N(1)	87.8 (7)	C(3)-C(1)-N(1)	112.4 (14)
C(9)-Si(1)-N(1)	115.8 (8)	C(3)-C(1)-C(2)	113.8 (18)
C(9)-Si(1)-N(2)	114.6 (7)	C(4)-C(1)-N(1)	110.4 (17)
C(10)-Si(1)-N(1)	113.8 (7)	C(4)-C(1)-C(2)	109.3 (14)
C(10)-Si(1)-N(2)	114.5 (8)	C(4)-C(1)-C(3)	104.6 (15)
C(10)-Si(1)-C(9)	109.1 (8)	C(6)-C(5)-N(2)	110.1 (21)
Si(1)-N(1)-Ge(1)	93.8 (7)	C(7)-C(5)-N(2)	108.0 (18)
C(1)-N(1)-Ge(1)	129.4 (10)	C(7)-C(5)-C(6)	104.7 (21)
C(1)-N(1)-Si(1)	136.7 (11)	C(8)-C(5)-N(2)	111.0 (20)
Si(1)-N(2)-Ge(1)	94.4 (6)	C(8)-C(5)-C(6)	112.5 (22)
C(5)-N(2)-Ge(1)	130.5 (12)	C(8)-C(5)-C(7)	110.2 (28)
C(5)-N(2)-Si(1)	135.1 (11)	C(12)-C(11)-N(3)	117.8 (11)
N(4)-N(3)-Ge(1)	113.3 (11)	C(16)-C(11)-N(3)	122.2 (12)
C(11)-N(3)-Ge(1)	129.3 (11)	C(22)-C(21)-N(6)	117.5 (12)
C(11)-N(3)-N(4)	116.8 (12)	C(26)-C(21)-N(6)	122.5 (12)

Fig. 2. Stereoscopic view of the unit cell (b nearly vertical, a nearly horizontal).

ment on F with 1820 reflections and 248 refined parameters; $w = 1.0/[\sigma^2(F) + 0.0005F^2]$; $S = 2.45$, $R = 0.072$, $wR = 0.068$, $(\Delta/\sigma)_{\max} = 0.1$ for the common U of H atoms and 0.01 for the remaining parameters; no extinction correction; largest peak in final ΔF map $\pm 1.3 (3) e \text{\AA}^{-3}$; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf-Nonius *SDP-Plus* (Frenz, 1985) for data reduction, *SHELXTL-Plus* (Sheldrick, 1987) for structure solution, refinement and plots and *PARST* (Nardelli, 1983) for least-squares planes.

The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit cell in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths and angles are given in Table 2.

Related literature. Preut *et al.* (1987).

* Lists of H-atom coordinates, least-squares planes and dihedral angles, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44525 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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