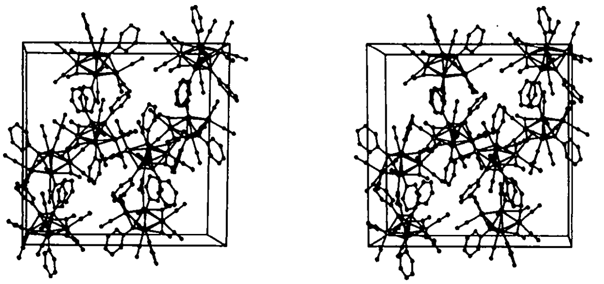

Fig. 3. Unit-cell packing diagram as viewed down the *a* axis.

unique ($R_{int} = 2{\cdot}31\%$), 7583 observed with $F_o > 6\sigma(F_o)$, 2507 unobserved reflections. Direct-methods (*SOLV*) structure solution; least-squares refinement on 776 parameters; all non-H atoms anisotropic, H atoms idealized and updated (C—H = 0·96 Å, $U = 1{\cdot}2\ U$ of attached C); phenyl rings rigid planar hexagons (C—C = 1·395 Å). $R_F = 2{\cdot}99\%$, $wR_F = 4{\cdot}13\%$, $S = 0{\cdot}955$, $w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0{\cdot}001$; $(\Delta/\sigma)_{max} = 0{\cdot}733$; $\Delta\rho_{max} = 0{\cdot}47$, $\Delta\rho_{min} = -0{\cdot}45$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); *SHELXTL* computer program (Sheldrick, 1984).

Atomic and equivalent isotropic thermal parameters are given in Table 1 and selected bond lengths in Table 2. Fig. 1 shows the labeled molecular structure of the compound and Fig. 2 the tetraruthenium cluster framework with bridging CO groups. A unit-cell packing diagram is shown in Fig. 3.*

Related literature. The title compound is the phenyl analog of a structure previously reported by Blohm & Gladfelter (1986).

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* Lists of structure factors, anisotropic thermal parameters and full lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44518 (70 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(*N*,*N*-dimethylformamide)[*o*-(*N*-methyliminomethyl)phenyl]palladium(II) Tetrafluoroborate

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Abstract. $[PdC_6H_4(CH_2NCH_3)(C_3H_7NO)_2]^+.BF_4^-$, $M_r = 458{\cdot}5$, triclinic, $P\bar{1}$, $a = 8{\cdot}098\ (2)$, $b = 9{\cdot}934\ (3)$, $c = 12{\cdot}860\ (2)$ Å, $\alpha = 67{\cdot}46\ (2)$, $\beta = 85{\cdot}41\ (2)$, $\gamma = 80{\cdot}22\ (2)°$, $V = 941{\cdot}5\ (4)$ Å^3, $Z = 2$, $D_x = 1{\cdot}617$ g cm^{-3}, $\mu = 10{\cdot}08$ cm^{-1} (Mo), $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $F(000) = 462$, $T = 296$ K, $R_F = 5{\cdot}01\%$ for 3141 observed reflections and 227 parameters. The Pd^{II} coordination environment is nearly square planar with the Pd atom 0·08 Å above the plane. Pd—N 2·018 (5), Pd—C 1·949 (5), Pd—O 2·077 (4) and 2·178 (4) Å. The molecules associate as invertamer pairs with Pd···Pd = 3·488 (1) Å.

Experimental. Yellow crystals of (1) from chloroform and diethyl ether, 0·3 × 0·3 × 0·4 mm, Nicolet *R*3*m* diffractometer with graphite monochromator, Wyckoff scans, lattice parameters from least-squares fit of 21 reflections ($23 \leq 2\theta \leq 33°$), absorption correction unnecessary ($\mu = 10{\cdot}08$ cm^{-1}, uniform crystal shape), $2\theta_{max} = 52°$ ($h = \pm 10$, $k = \pm 13$, $l = +16$), standard reflections 500, 250 and 129, variation ≤ 2%. 3783 reflections collected, 3554 unique, $R_{int} = 2{\cdot}67\%$, 3141 observed with $F_o \geq 3\sigma(F_o)$, 369 unobserved reflections, sharpened Patterson structure solution, least-squares refinement on 227 parameters, all non-hydrogen atoms anisotropic, H atoms idealized and updated [C—H = 0·96 Å, $U = 1{\cdot}2\ U$ of attached C], BF_4^- geometry

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

U_{eq} = one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Pd	1953·0 (4)	420·2 (4)	145·8 (3)	54·8 (1)
N(1)	3549 (5)	−1027 (5)	−347 (4)	66 (2)
N(2)	−1193 (6)	3193 (5)	1485 (4)	71 (2)
N(3)	2892 (7)	−1930 (5)	3760 (4)	73 (2)
O(1)	309 (5)	2131 (4)	381 (3)	66 (1)
O(2)	2442 (5)	−474 (4)	1944 (3)	73 (2)
C(1)	4515 (8)	−2380 (6)	427 (6)	88 (3)
C(2)	3705 (8)	−693 (7)	−1396 (5)	78 (3)
C(3)	2692 (7)	633 (7)	−2102 (5)	71 (3)
C(4)	2671 (9)	1181 (9)	−3260 (6)	92 (3)
C(5)	1696 (11)	2482 (10)	−3833 (6)	108 (4)
C(6)	712 (9)	3271 (8)	−3248 (5)	93 (3)
C(7)	686 (8)	2707 (7)	−2081 (5)	75 (2)
C(8)	1698 (6)	1389 (6)	−1480 (4)	60 (2)
C(9)	−96 (6)	2164 (6)	1321 (5)	63 (2)
C(10)	−1630 (10)	3118 (9)	2630 (7)	101 (4)
C(11)	−2067 (8)	4407 (7)	566 (7)	90 (3)
C(12)	2784 (8)	−1690 (6)	2691 (5)	70 (2)
C(13)	2547 (12)	−733 (7)	4152 (6)	109 (4)
C(14)	3295 (12)	−3401 (7)	4609 (6)	120 (4)
B	6561 (4)	−3464 (4)	−2873 (3)	68 (3)
F(1)	6439 (8)	−3429 (6)	−1867 (4)	215 (5)
F(2)	6074 (9)	−4664 (5)	−2843 (5)	215 (5)
F(3)	5616 (6)	−2318 (5)	−3558 (4)	152 (3)
F(4)	8115 (5)	−3446 (8)	−3226 (5)	264 (8)

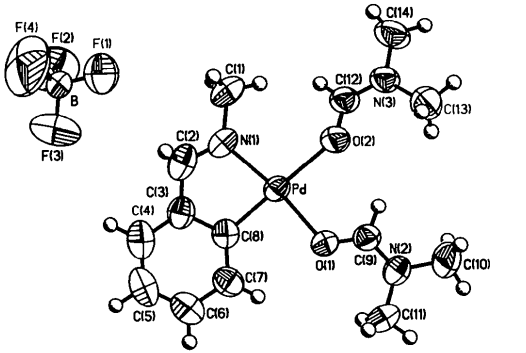


Fig. 1. Molecular structure and labeling scheme for the title compound. The thermal ellipsoids are drawn at the 40% probability level.

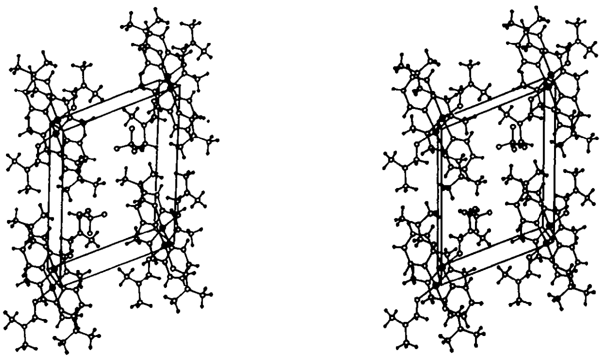


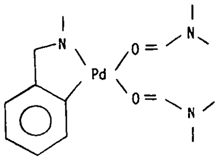
Fig. 2. Unit-cell packing diagram as viewed down the *a* axis (the *c* axis is vertical).

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Pd—N(1)	2·018 (5)	Pd—O(1)	2·077 (4)
Pd—O(2)	2·178 (4)	Pd—C(8)	1·949 (5)
N(1)—C(1)	1·467 (7)	N(1)—C(2)	1·260 (9)
N(2)—C(9)	1·306 (8)	N(2)—C(10)	1·463 (10)
N(2)—C(11)	1·450 (7)	N(3)—C(12)	1·310 (8)
N(3)—C(13)	1·439 (10)	N(3)—C(14)	1·452 (7)
O(1)—C(9)	1·239 (7)	O(2)—C(12)	1·224 (6)
C(2)—C(3)	1·438 (7)	C(3)—C(4)	1·376 (9)
C(3)—C(8)	1·415 (9)	C(4)—C(5)	1·362 (11)
C(5)—C(6)	1·400 (13)	C(6)—C(7)	1·386 (9)
C(7)—C(8)	1·394 (7)	B—F(1)	1·303 (6)
B—F(2)	1·304 (7)	B—F(3)	1·304 (5)
B—F(4)	1·303 (5)		

N(1)—Pd—O(1)	170·8 (2)	N(1)—Pd—O(2)	100·6 (2)
O(1)—Pd—O(2)	88·2 (1)	N(1)—Pd—C(8)	80·5 (2)
O(1)—Pd—C(8)	90·5 (2)	O(2)—Pd—C(8)	173·1 (2)
Pd—N(1)—C(1)	124·3 (4)	Pd—N(1)—C(2)	115·6 (4)
C(1)—N(1)—C(2)	120·1 (6)	C(9)—N(2)—C(10)	120·1 (5)
C(9)—N(2)—C(11)	122·4 (6)	C(10)—N(2)—C(11)	117·5 (6)
C(12)—N(3)—C(13)	120·9 (5)	C(12)—N(3)—C(14)	122·3 (6)
C(13)—N(3)—C(14)	116·7 (6)	Pd—O(1)—C(9)	123·3 (3)
Pd—O(2)—C(12)	137·5 (5)	N(1)—C(2)—C(3)	117·0 (6)
C(2)—C(3)—C(4)	125·9 (7)	C(2)—C(3)—C(8)	112·7 (5)
C(4)—C(3)—C(8)	121·3 (5)	C(3)—C(4)—C(5)	120·2 (8)
C(4)—C(5)—C(6)	120·2 (7)	C(5)—C(6)—C(7)	120·1 (6)
C(6)—C(7)—C(8)	120·5 (6)	Pd—C(8)—C(3)	114·1 (3)
Pd—C(8)—C(7)	128·2 (5)	C(3)—C(8)—C(7)	117·7 (5)
N(2)—C(9)—O(1)	123·9 (4)	N(3)—C(12)—O(2)	124·9 (6)
F(1)—B—F(2)	109·5 (5)	F(1)—B—F(3)	109·5 (4)
F(2)—B—F(3)	109·4 (5)	F(1)—B—F(4)	109·5 (5)
F(2)—B—F(4)	109·5 (6)	F(3)—B—F(4)	109·4 (4)

fixed to tetrahedron, $d(\mathrm{BF}) = 1{\cdot}34$ Å. $R_F = 5{\cdot}01\%$, $wR_F = 6{\cdot}92\%$, $S = 1{\cdot}224$, $w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0{\cdot}002$, $(\Delta/\sigma)_{\max} = 0{\cdot}077$, $\Delta\rho_{\max} = 0{\cdot}81$, $\Delta\rho_{\min} = -0{\cdot}57$ e Å^{-3}, atomic scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELXTL* computer program (Sheldrick, 1984).*



(1)

Atomic and thermal parameters are given in Table 1, bond lengths and angles are listed in Table 2. Fig. 1 shows the labelled molecular structure of the compound, and Fig. 2 the packing arrangement.

Related literature. No related palladium structures containing oxygen-bound amides could be located in the Cambridge Database. The platinum structure

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* Lists of anisotropic thermal parameters, observed and calculated structure factors and bond distances and angles involving H have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44498 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

[*trans*-Pt{$OC(CH_3)N(CH_3)_2$}$_2$-*trans*-Cl_2] 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 $AgBF_4$ in *N*,*N*-dimethylformamide (Wu & Heck, 1987).

The crystals were a gift of Dr R. Heck.

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Acta Cryst. (1988). C**44**, 574–575

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

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Abstract. $C_{22}H_{34}GeN_6Si$, $M_r = 483{\cdot}23$, monoclinic, $C2/c$, $a = 23{\cdot}493\,(8)$, $b = 17{\cdot}160\,(22)$, $c = 16{\cdot}895\,(20)$ Å, $\beta = 124{\cdot}44\,(6)°$, $V = 5617\,(11)$ Å^3, $Z = 8$, $D_x = 1{\cdot}143$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}13$ mm^{-1}, $F(000) = 2032$, $T = 291\,(1)$ K, final $R = 0{\cdot}072$ for 1820 unique observed [$F \geq 4{\cdot}0\sigma(F)$] 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 germylene (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 ~0·1 × 0·2 × 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 $2\theta = 25{\cdot}6°$; six standard reflections recorded every 2·5 h, only random deviations; 7442 reflections measured, $1{\cdot}5 \leq \theta \leq 22{\cdot}0°$, $-24 \leq h \leq 24$, $0 \leq k \leq 18$, $-17 \leq l \leq 17$; after averaging ($R_{\text{int}} = 0{\cdot}03$): 3446 unique reflections, 1820 with $F \geq 4{\cdot}0\sigma(F)$; 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-

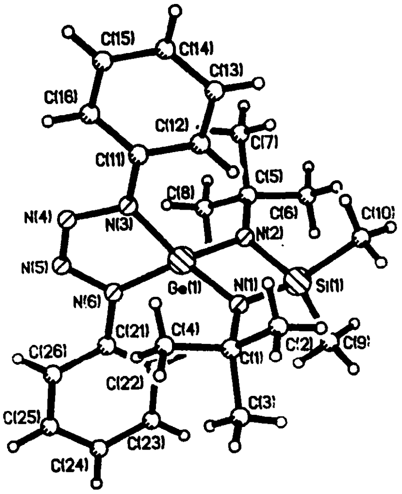

Fig. 1. View of the molecule, showing the atom-numbering scheme.

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