

### 3-*tert*-Butyl-5-methyl-2,3-diphenyl-1,2-diaza-3-sila-5-cyclopentene

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**Abstract.** C<sub>19</sub>H<sub>24</sub>N<sub>2</sub>Si,  $M_r = 308.5$ , monoclinic,  $P2_1/c$ ,  $a = 8.135$  (2),  $b = 11.612$  (3),  $c = 18.990$  (4) Å,  $\beta = 96.683$  (5)°,  $Z = 4$ ,  $D_o = 1.15$ ,  $D_c = 1.150$  Mg m<sup>-3</sup>. The central SiN<sub>2</sub>C<sub>2</sub> ring has an envelope conformation, the Si atom lying 0.26 Å out of the plane of the other four atoms.

**Introduction.** This structure was determined as part of a study of Si–N derivatives. Crystals suitable for X-ray diffraction were obtained from light petroleum ether. Initial cell dimensions were obtained from oscillation and Weissenberg photographs (Cu  $K\alpha$  radiation). Systematic absences ( $h0l$  for odd  $l$ ;  $0k0$  for odd  $k$ ) define the space group  $P2_1/c$  uniquely.

Intensities were collected with a Stoe two-circle diffractometer from two crystals of maximum dimension 0.5 mm (layers  $h0l$  to  $h15l$ ,  $0kl$  and  $1kl$ ;  $\omega$  scans, approximate constant-count mode; Mo  $K\alpha$  radiation). Cell dimensions were refined by least-squares methods from the observed  $\omega$  angles for all  $h0l$  and  $0kl$  reflexions. No absorption corrections were applied ( $\mu = 0.096$  mm<sup>-1</sup>). After refinement of interlayer scale factors and merging of equivalent reflexions, 4248 independent reflexions with  $I > 2\sigma(I)$  were used for structure solution and refinement.

The structure was solved by multiresolution  $\sum_2$  sign expansion. The figures of merit NQUEST (DeTitta, Edmonds, Langs & Hauptman, 1975) and  $R(\alpha)$  (Roberts, Pettersen, Sheldrick, Isaacs & Kennard, 1973) both unambiguously identified the best solution, which gave an  $E$  map missing only one atom.  $\sum w\Delta^2$  was minimized in the refinement  $\{\Delta = |F_o| - |F_c|, w = [\sigma^2(F) + g|F_o|^2]^{-1}; g$  refined to 0.00048}. All H atoms were located by difference syntheses and included in the refinement, but with rigid methyl groups and geometrical constraints (C–H 0.96 Å; H–C–H 109.5°; H on external angle bisector for aromatic C). Group isotropic thermal parameters were refined for the H atoms attached to each C atom; all non-H atoms were refined anisotropically. Scattering factors were those of Cromer & Waber (1974) and Cromer & Ibers (1974).

The final  $R = 0.056$ , with  $R_w = (\sum w\Delta^2 / \sum wF_o^2)^{1/2} = 0.073$ . An analysis of variance showed no trends with

indices,  $\sin \theta$  or  $|F_o|$ , and there were no significant features in a final difference synthesis. Atomic coordinates are given in Table 1.\* The numbering scheme is shown in Fig. 1.

**Discussion.** The compound, one of a series of 1,2-diaza-3-sila-5-cyclopentenes, was prepared by a cyclization reaction from a fluorosilylhydrazone (Klingebiel & Werner, 1979). The crystallographically determined structure is in agreement with that proposed. Bond lengths and angles are given in Table 2.

Bond lengths are unexceptional. The two phenyl rings show typical librational effects, observed bond lengths decreasing away from the centre of the molecule. The two rings are essentially planar, r.m.s.

\* Lists of structure factors, thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34429 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ( $\times 10^4$ )

	<i>x</i>	<i>y</i>	<i>z</i>
N(1)	2258 (2)	7868 (1)	1380 (1)
N(2)	2563 (2)	8975 (1)	1677 (1)
Si(3)	4136 (1)	9722 (1)	1304 (1)
C(4)	4088 (2)	8627 (2)	581 (1)
C(5)	3022 (2)	7692 (1)	838 (1)
C(20)	1918 (2)	9156 (2)	2324 (1)
C(21)	1868 (3)	10278 (2)	2589 (1)
C(22)	1263 (3)	10476 (2)	3231 (1)
C(23)	692 (3)	9582 (2)	3610 (1)
C(24)	708 (2)	8480 (2)	3345 (1)
C(25)	1315 (2)	8263 (2)	2706 (1)
C(30)	3413 (2)	11160 (1)	946 (1)
C(31)	4249 (3)	12194 (2)	1083 (1)
C(32)	3707 (4)	13205 (2)	745 (1)
C(33)	2353 (4)	13198 (2)	251 (1)
C(34)	1485 (3)	12202 (2)	104 (1)
C(35)	1998 (3)	11193 (2)	455 (1)
C(36)	6163 (2)	9702 (2)	1886 (1)
C(37)	6178 (3)	10411 (2)	2568 (1)
C(38)	7543 (2)	10132 (2)	1461 (1)
C(39)	6505 (3)	8434 (2)	2096 (1)
C(50)	2849 (3)	6550 (2)	473 (1)

Table 2. Bond lengths (Å) and angles (°)

N(1)—N(2)	1.414 (2)	C(20)—C(21)	1.399 (2)
N(2)—Si(3)	1.762 (2)	C(21)—C(22)	1.387 (3)
N(2)—C(20)	1.405 (2)	C(22)—C(23)	1.374 (3)
Si(3)—C(4)	1.869 (2)	C(23)—C(24)	1.376 (3)
Si(3)—C(30)	1.871 (2)	C(24)—C(25)	1.384 (3)
Si(3)—C(36)	1.876 (2)	C(25)—C(20)	1.389 (2)
C(4)—C(5)	1.507 (2)	C(30)—C(31)	1.390 (3)
C(5)—C(50)	1.496 (3)	C(31)—C(32)	1.385 (3)
C(5)—N(1)	1.279 (2)	C(32)—C(33)	1.361 (4)
C(36)—C(37)	1.533 (3)	C(33)—C(34)	1.368 (4)
C(36)—C(38)	1.540 (3)	C(34)—C(35)	1.387 (3)
C(36)—C(39)	1.542 (3)	C(35)—C(30)	1.396 (2)
C(5)—N(1)—N(2)	112.9 (1)	Si(3)—C(4)—C(5)	102.4 (1)
N(1)—N(2)—Si(3)	112.8 (1)	C(4)—C(5)—N(1)	119.8 (1)
N(1)—N(2)—C(20)	114.8 (1)	C(4)—C(5)—C(50)	121.0 (2)
Si(3)—N(2)—C(20)	129.8 (1)	N(1)—C(5)—C(50)	119.2 (2)
N(2)—Si(3)—C(4)	90.4 (1)	Si(3)—C(36)—C(37)	114.3 (1)
N(2)—Si(3)—C(30)	111.9 (1)	Si(3)—C(36)—C(38)	109.3 (1)
N(2)—Si(3)—C(36)	112.7 (1)	Si(3)—C(36)—C(39)	106.5 (1)
C(4)—Si(3)—C(30)	111.2 (1)	C(37)—C(36)—C(38)	109.5 (2)
C(4)—Si(3)—C(36)	111.5 (1)	C(37)—C(36)—C(39)	108.1 (2)
C(30)—Si(3)—C(36)	116.3 (1)	C(38)—C(36)—C(39)	108.9 (2)
Si(3)—C(30)—C(31)	125.2 (1)	N(2)—C(20)—C(21)	118.9 (1)
Si(3)—C(30)—C(35)	117.9 (1)	N(2)—C(20)—C(25)	122.4 (2)
C(35)—C(30)—C(31)	116.7 (2)	C(25)—C(20)—C(21)	118.7 (2)
C(30)—C(31)—C(32)	121.5 (2)	C(20)—C(21)—C(22)	120.0 (2)
C(31)—C(32)—C(33)	120.2 (2)	C(21)—C(22)—C(23)	120.8 (2)
C(32)—C(33)—C(34)	120.2 (2)	C(22)—C(23)—C(24)	119.5 (2)
C(33)—C(34)—C(35)	119.8 (2)	C(23)—C(24)—C(25)	120.7 (2)
C(34)—C(35)—C(30)	121.5 (2)	C(24)—C(25)—C(20)	120.4 (2)

deviations from calculated mean planes being 0.007 and 0.010 Å.

The coordination of N(2) is almost planar, N(2) lying 0.13 Å out of the plane of N(1), Si(3) and C(20). This slight non-planarity is probably caused by steric interaction of the *tert*-butyl and two phenyl groups, which also produces a Si(3)—N(2)—C(20) angle of 129.8°.

The central SiN<sub>2</sub>C<sub>2</sub> ring has an envelope conformation. The N and C atoms lie in a plane (r.m.s. deviation 0.003 Å), and the N—Si—C flap is inclined at 11.8° to it.

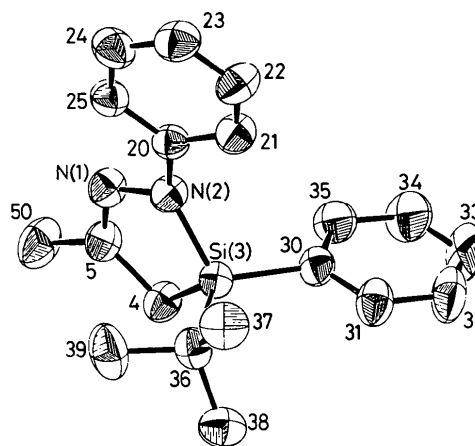


Fig. 1. ORTEP (Johnson, 1965) drawing of the molecule, displaying thermal motion as 50% probability ellipsoids.

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