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Structures of the *cis* and *trans* Isomers of 2,4,6-Tri-*tert*-butyl-2,4,6-trifluorocyclotrisilazane, C₁₂H₃₀F₃N₃Si₃

BY WILLIAM CLEGG, GEORGE M. SHELDRICK AND DIETMAR STALKE

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, D-3400 Göttingen, Federal Republic of Germany

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Abstract. $M_r = 357.6$; *cis* isomer (1): trigonal, $\bar{P}3$, $a = 10.873$ (1), $c = 10.040$ (1) Å, $U = 1027.9$ Å³, $Z = 2$, $D_x = 1.155$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.25$ mm⁻¹, $F(000) = 384$, $T = 291$ K, $R = 0.054$ for 1232 observed reflections; *trans* isomer (2): monoclinic, $P2_1/n$, $a = 18.077$ (4), $b = 10.569$ (2), $c = 22.015$ (6) Å, $\beta = 104.67$ (3)°, $U = 4069.0$ Å³, $Z = 8$, $D_x = 1.167$ Mg m⁻³, $\mu = 0.25$ mm⁻¹, $F(000) = 1536$, $T = 291$ K, $R = 0.071$ for 4421 observed reflections. (1) has crystallographic threefold rotation symmetry and a virtually planar Si₃N₃ ring [r.m.s. deviation = 0.017 (2) Å]. The two crystallographically independent molecules of (2) lie in general positions and deviations from planarity of the ring are greater [r.m.s. $\Delta = 0.086$ (3) and 0.128 (3) Å].

Introduction. A number of chain and ring silazanes built up from –HNSiBuF– units have recently been synthesized by controlled condensation reactions of BuSiF₃ and LiNH₂ [Bu = *tert*-butyl, (CH₃)₃C] (Klingebiel & Vater, 1983). The cyclotrisilazane (BuSiFNH)₃ exists in two stable non-interconverting isomeric forms, the separation of which has been accomplished by fractional crystallization: the *cis* isomer is less soluble in non-polar solvents and can be selectively crystallized from a mixed solution in CH₂Cl₂ by addition of *n*-hexane.

We have investigated the structures of both isomers in order to assess the influence of the geometrical substituent distribution on the expected planarity of the Si₃N₃ ring.

Experimental. Crystals obtained from CH₂Cl₂/*n*-hexane (1) or *n*-hexane by cooling (2), 0.4 × 0.5 × 0.15 mm (1), 0.3 × 0.3 × 0.45 mm (2), mounted in capillaries, Stoe–Siemens AED diffractometer, unit-cell parameters from 2θ values of 36 (1) and 32 (2) reflections ($20 < 2\theta < 25^\circ$), 1826 reflections with $2\theta < 55^\circ$ and $h, k \geq 0$ (1) and 8179 reflections with $2\theta < 50^\circ$ and $k \geq 1$ (2), profile analysis (Clegg, 1981), no significant intensity variation for 3 standard reflections, no absorption corrections, $R_{int} = 0.027$ (1) and 0.037 (2), 1581 (1) and 7144 (2) unique reflections, 1232 (1) and 4421 (2) with $F > 4\sigma(F)$; automatic multiresolution direct methods, blocked-cascade refinement on F , $w^{-1} = \sigma^2(F) + gF^2$, $g = 0.00068$ (1), 0.00041 (2), Bu H atoms constrained to give C–H = 0.96 Å, H–C–H = 109.5°, $U(H) = 1.2U_{eq}(C)$, anisotropic thermal parameters for non-H atoms, no extinction corrections, scattering factors from *International Tables for X-ray Crystallography* (1974). (1): N–H refined freely, 77 parameters, $R = 0.054$, $wR = 0.068$, slope of normal probability plot = 1.51, max. $\Delta/\sigma = 0.013$, mean = 0.001, largest peak in final difference map = 0.25 e Å⁻³, largest hole = –0.33 e Å⁻³; (2): minor disorder component for one BuSiF group with site occupation factor 0.119 (2) for Si and F; disorder of Bu group not resolved; N–H constrained to lie on Si–N–Si external bisector with N–H = 0.62 Å [value obtained from free refinement of (1)], $U(H) = 1.2U_{eq}(N)$, 398 parameters, $R = 0.071$, $wR = 0.073$, slope = 1.61, max. $\Delta/\sigma = 0.029$, mean = 0.005, largest peak = 0.63 e Å⁻³, largest hole =

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$) for (1)
$$U = \frac{1}{3} (\text{trace of the orthogonalized } U_{ij} \text{ matrix}).$$

| | x | y | z | U |
|------|-----------|-----------|-----------|-----------|
| Si | 6802 (1) | 5046 (1) | 6339 (1) | 417 (3) |
| N | 5283 (2) | 3450 (2) | 6374 (3) | 482 (11) |
| H(1) | 4753 (30) | 3515 (32) | 6374 (29) | 491 (97) |
| F | 6862 (2) | 5818 (2) | 4963 (2) | 622 (9) |
| C(1) | 6907 (3) | 6310 (3) | 7646 (3) | 587 (14) |
| C(2) | 5599 (4) | 6492 (4) | 7591 (4) | 893 (26) |
| C(3) | 6986 (5) | 5766 (4) | 9023 (3) | 946 (27) |
| C(4) | 8243 (6) | 7746 (4) | 7408 (6) | 1341 (29) |

Table 2. Bond lengths (\AA) and angles ($^\circ$) for (1)

| | | | |
|-------------------------|------------|--------------------|------------|
| Si-F | 1.602 (2) | Si-C(1) | 1.862 (3) |
| N-Si | 1.695 (2) | N-Si ⁱⁱ | 1.692 (3) |
| N-H(1) | 0.615 (38) | C(1)-C(2) | 1.532 (7) |
| C(1)-C(3) | 1.523 (5) | C(1)-C(4) | 1.529 (4) |
| N-Si-N ⁱⁱ | 107.0 (2) | F-Si-N | 108.5 (1) |
| F-Si-N ⁱⁱ | 108.6 (1) | C(1)-Si-N | 114.2 (1) |
| C(1)-Si-N ⁱⁱ | 113.9 (1) | F-Si-C(1) | 104.5 (1) |
| Si-N-Si ⁱ | 133.0 (2) | Si-N-H(1) | 111.9 (28) |
| Si ⁱ -N-H(1) | 115.1 (28) | Si-C(1)-C(2) | 110.6 (2) |
| Si-C(1)-C(3) | 110.3 (3) | Si-C(1)-C(4) | 108.7 (3) |
| C(2)-C(1)-C(3) | 108.7 (3) | C(2)-C(1)-C(4) | 109.4 (4) |
| C(3)-C(1)-C(4) | 109.2 (3) | | |

Symmetry operators: (i) 1-y, x-y, z; (ii) 1-x+y, 1-x, z.

$-0.32 e \text{\AA}^{-3}$.* Programs: *SHELXTL* (Sheldrick, 1978), diffractometer control program by WC.

Discussion. Atomic coordinates, bond lengths and angles are given in Tables 1-4. The molecular structures are shown in Figs. 1-3. The more symmetrical *cis* isomer (1) forms much better quality crystals in a higher-symmetry space group; it was possible to refine freely the H atom attached to N. Thermal motion in (2) is rather greater (although the crystal density is slightly higher), and free refinement of six independent N-H groups proved unsuccessful. There is also disorder of one of the BuSiF groups in the second molecule (Fig. 3); derived geometry within and around this group is unreliable. It was not possible to resolve the disorder of the Bu group or of the neighbouring NH groups.

Corresponding bond lengths and angles for the two isomers are insignificantly different; for (1) and for (2) respectively, mean values for the most important parameters (ignoring disorder) are: Si-N 1.695 (2), 1.696 (7); Si-F 1.602 (2), 1.600 (5); Si-C 1.862 (3), 1.851 (9) \AA ; N-Si-N 107.0 (2), 106.5 (8); Si-N-Si 133.0 (2), 131.5 (22) $^\circ$. They are also similar to those of the simple cyclotrisilazane *cis*-(PhSiFNMe)₃ (Clegg, Noltemeyer, Sheldrick & Vater, 1980). As in this case,

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39132 (66 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$) for (2)
$$U = \frac{1}{3} (\text{trace of the orthogonalized } U_{ij} \text{ matrix}).$$

| | x | y | z | U |
|-----------------|-----------|-----------|-----------|-----------|
| Si(1) | 2385 (1) | -870 (1) | -459 (1) | 587 (5) |
| F(1) | 2186 (2) | -679 (3) | -1203 (1) | 771 (11) |
| C(11) | 2362 (3) | -2610 (5) | -333 (2) | 707 (21) |
| C(12) | 2921 (4) | -3277 (6) | -619 (4) | 1317 (38) |
| C(13) | 1566 (3) | -3106 (6) | -627 (4) | 1276 (36) |
| C(14) | 2558 (5) | -2890 (7) | 353 (3) | 1636 (47) |
| N(1) | 1739 (2) | -35 (4) | -188 (2) | 728 (16) |
| Si(2) | 1829 (1) | 1012 (1) | 407 (1) | 591 (5) |
| F(2) | 1676 (2) | 323 (3) | 1007 (1) | 915 (13) |
| C(21) | 1117 (2) | 2302 (4) | 234 (2) | 651 (19) |
| C(22) | 309 (3) | 1788 (6) | 69 (3) | 956 (27) |
| C(23) | 1225 (4) | 3053 (6) | -348 (3) | 1260 (35) |
| C(24) | 1230 (4) | 3189 (6) | 803 (3) | 1476 (39) |
| N(2) | 2751 (2) | 1544 (4) | 577 (2) | 674 (16) |
| Si(3) | 3517 (1) | 1079 (1) | 321 (1) | 644 (5) |
| F(3) | 4182 (1) | 680 (3) | 922 (1) | 807 (11) |
| C(31) | 3944 (3) | 2339 (5) | -72 (2) | 708 (20) |
| C(32) | 4074 (3) | 3547 (5) | 327 (3) | 1011 (27) |
| C(33) | 4712 (3) | 1897 (7) | -170 (3) | 1137 (32) |
| C(34) | 3404 (3) | 2650 (6) | -712 (3) | 1059 (28) |
| N(3) | 3252 (2) | -221 (4) | -124 (2) | 824 (18) |
| Si(4) | 77 (1) | -82 (1) | 7896 (1) | 539 (5) |
| F(4) | 330 (1) | -559 (3) | 8606 (1) | 761 (11) |
| C(41) | 8 (3) | -1507 (5) | 7393 (3) | 715 (22) |
| C(42) | 808 (3) | -2012 (6) | 7401 (3) | 1087 (31) |
| C(43) | -407 (4) | -1174 (7) | 6705 (3) | 1210 (33) |
| C(44) | -430 (3) | -2550 (6) | 7623 (3) | 1158 (34) |
| N(4) | -764 (2) | 713 (3) | 7797 (2) | 610 (15) |
| Si(5) | -1068 (1) | 2054 (1) | 7395 (1) | 543 (5) |
| F(5) | -1726 (1) | 1724 (3) | 6774 (1) | 709 (11) |
| C(51) | -1516 (2) | 3175 (4) | 7834 (2) | 620 (19) |
| C(52) | -1732 (4) | 4408 (5) | 7475 (3) | 1153 (33) |
| C(53) | -2251 (3) | 2612 (6) | 7949 (3) | 997 (29) |
| C(54) | -963 (3) | 3461 (6) | 8466 (3) | 1180 (31) |
| N(5) | -321 (2) | 2621 (4) | 7143 (2) | 725 (17) |
| Si(6) | 608 (1) | 2163 (2) | 7243 (1) | 586 (6) |
| F(6) | 737 (2) | 1668 (4) | 6588 (1) | 871 (15) |
| C(61) | 1320 (3) | 3439 (5) | 7436 (2) | 770 (22) |
| C(62) | 1202 (3) | 4389 (6) | 6919 (3) | 906 (29) |
| C(63) | 2124 (3) | 2908 (7) | 7583 (3) | 975 (30) |
| C(64) | 1222 (4) | 4167 (7) | 8035 (3) | 1181 (38) |
| N(6) | 738 (2) | 967 (4) | 7781 (2) | 704 (16) |
| Si(6 \times) | 634 (5) | 2675 (9) | 7746 (4) | 411 (35) |
| F(6 \times) | 778 (12) | 3526 (21) | 8335 (10) | 684 (90) |

the *cis* isomer (1) contains a virtually planar Si₃N₃ ring, with a very slight puckering towards a chair conformation, and the electron-withdrawing fluorine substituents cause a reduction in Si-N bond length compared with other simple cyclotrisilazanes, such as (Bu₂SiNH)₃ [1.727 (2) \AA] (Clegg, Sheldrick & Stalke, 1984).

There is no significant difference between the two crystallographically independent molecules of the *trans* isomer (2) (ignoring the disorder). Deviations from planarity of the Si₃N₃ rings are greater than in (1), but are still very small compared with those observed when bulky substituents are attached to N, as in (Me₂SiNSiMe₃)₃, which has a boat conformation (Adamson & Daly, 1970).

We may conclude that a planar Si₃N₃ ring is normal for cyclotrisilazanes, except when the steric bulk of the N substituents or ring fusion render this impossible (Clegg, Sheldrick & Stalke, 1984). Bulky Si substituents do not greatly affect this planar conformation, and crystal packing produces only very minor perturbations.

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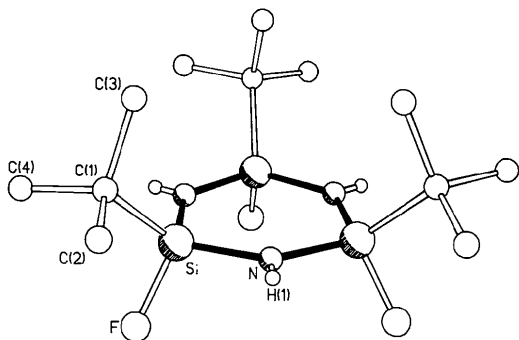


Fig. 1. Molecular structure of the *cis* isomer (1). H atoms are omitted from the *tert*-butyl groups.

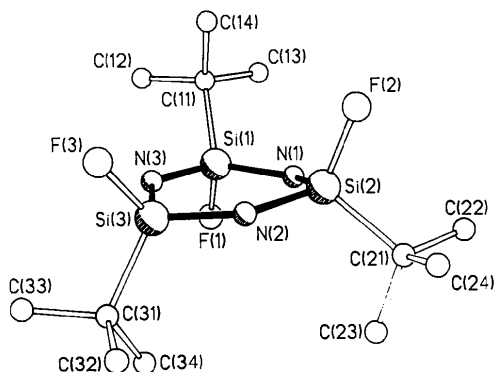


Fig. 2. Molecule 1 of the *trans* isomer (2). H atoms are omitted.

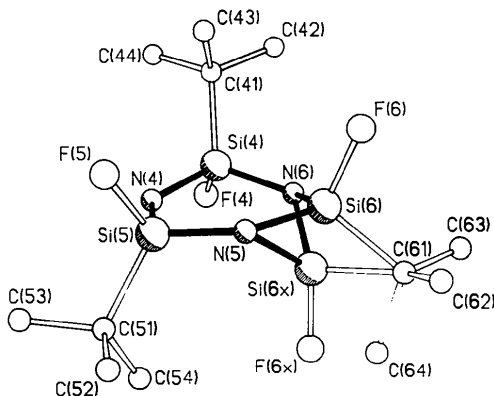


Fig. 3. Molecule 2 of the *trans* isomer (2), showing both disorder components. H atoms are omitted.

Table 4. Bond lengths (Å) and angles (°) for (2)

| | | | |
|-------------------|------------|--------------------|------------|
| Si(1)–N(1) | 1.689 (4) | Si(4)–N(4) | 1.702 (4) |
| Si(1)–N(3) | 1.698 (4) | Si(4)–N(6) | 1.695 (4) |
| Si(2)–N(1) | 1.691 (4) | Si(5)–N(4) | 1.688 (4) |
| Si(2)–N(2) | 1.707 (4) | Si(5)–N(5) | 1.693 (4) |
| Si(3)–N(2) | 1.695 (4) | Si(6)–N(5) | 1.706 (4) |
| Si(3)–N(3) | 1.685 (4) | Si(6)–N(6) | 1.708 (4) |
| Si(1)–F(1) | 1.598 (3) | Si(4)–F(4) | 1.596 (3) |
| Si(2)–F(2) | 1.591 (3) | Si(5)–F(5) | 1.607 (2) |
| Si(3)–F(3) | 1.602 (3) | Si(6)–F(6) | 1.604 (4) |
| Si(1)–C(11) | 1.862 (5) | Si(4)–C(41) | 1.855 (5) |
| Si(2)–C(21) | 1.848 (5) | Si(5)–C(51) | 1.842 (5) |
| Si(3)–C(31) | 1.859 (6) | Si(6)–C(61) | 1.839 (5) |
| C(11)–C(12) | 1.496 (9) | C(41)–C(42) | 1.537 (8) |
| C(11)–C(13) | 1.515 (7) | C(41)–C(43) | 1.550 (7) |
| C(11)–C(14) | 1.492 (8) | C(41)–C(44) | 1.517 (9) |
| C(21)–C(22) | 1.512 (6) | C(51)–C(52) | 1.523 (7) |
| C(21)–C(23) | 1.561 (9) | C(51)–C(53) | 1.534 (8) |
| C(21)–C(24) | 1.537 (8) | C(51)–C(54) | 1.524 (7) |
| C(31)–C(32) | 1.533 (8) | C(61)–C(62) | 1.493 (8) |
| C(31)–C(33) | 1.531 (8) | C(61)–C(63) | 1.515 (7) |
| C(31)–C(34) | 1.532 (7) | C(61)–C(64) | 1.575 (9) |
| Si(6x)–N(5) | 1.893 (9) | Si(6x)–N(6) | 1.815 (10) |
| Si(6x)–F(6x) | 1.546 (23) | Si(6x)–C(61) | 1.756 (11) |
| F(1)–Si(1)–C(11) | 105.6 (2) | F(4)–Si(4)–C(41) | 106.7 (2) |
| F(1)–Si(1)–N(1) | 107.5 (2) | F(4)–Si(4)–N(4) | 107.9 (2) |
| C(11)–Si(1)–N(1) | 114.9 (2) | C(41)–Si(4)–N(4) | 113.7 (2) |
| F(1)–Si(1)–N(3) | 109.7 (2) | F(4)–Si(4)–N(6) | 108.4 (2) |
| C(11)–Si(1)–N(3) | 112.7 (2) | C(41)–Si(4)–N(6) | 112.6 (2) |
| N(1)–Si(1)–N(3) | 106.3 (2) | N(4)–Si(4)–N(6) | 107.4 (2) |
| N(1)–Si(2)–F(2) | 109.9 (2) | N(4)–Si(5)–F(5) | 109.6 (2) |
| N(1)–Si(2)–C(21) | 113.4 (2) | N(4)–Si(5)–C(51) | 113.1 (2) |
| F(2)–Si(2)–C(21) | 104.5 (2) | F(5)–Si(5)–C(51) | 104.9 (2) |
| N(1)–Si(2)–N(2) | 106.4 (2) | N(4)–Si(5)–N(5) | 106.8 (2) |
| F(2)–Si(2)–N(2) | 109.6 (2) | F(5)–Si(5)–N(5) | 106.2 (2) |
| C(21)–Si(2)–N(2) | 113.1 (2) | C(51)–Si(5)–N(5) | 116.0 (2) |
| N(2)–Si(3)–F(3) | 107.9 (2) | N(5)–Si(6)–F(6) | 109.8 (2) |
| N(2)–Si(3)–C(31) | 114.2 (2) | N(5)–Si(6)–C(61) | 115.5 (2) |
| F(3)–Si(3)–C(31) | 105.1 (2) | F(6)–Si(6)–C(61) | 101.2 (2) |
| N(2)–Si(3)–N(3) | 107.0 (2) | N(5)–Si(6)–N(6) | 104.9 (2) |
| F(3)–Si(3)–N(3) | 108.0 (2) | F(6)–Si(6)–N(6) | 110.8 (2) |
| C(31)–Si(3)–N(3) | 113.9 (2) | C(61)–Si(6)–N(6) | 114.6 (2) |
| Si(1)–N(1)–Si(2) | 132.5 (2) | Si(4)–N(4)–Si(5) | 130.0 (2) |
| Si(2)–N(2)–Si(3) | 131.8 (2) | Si(5)–N(5)–Si(6) | 134.3 (2) |
| Si(1)–N(3)–Si(3) | 132.6 (3) | Si(4)–N(6)–Si(6) | 127.5 (2) |
| Si(1)–C(11)–C(12) | 111.1 (4) | Si(4)–C(41)–C(42) | 110.7 (3) |
| Si(1)–C(11)–C(13) | 109.4 (3) | Si(4)–C(41)–C(43) | 109.9 (4) |
| C(12)–C(11)–C(13) | 109.1 (5) | C(42)–C(41)–C(43) | 108.2 (5) |
| Si(1)–C(11)–C(14) | 109.7 (4) | Si(4)–C(41)–C(44) | 110.6 (4) |
| C(12)–C(11)–C(14) | 108.7 (5) | C(42)–C(41)–C(44) | 108.3 (4) |
| C(13)–C(11)–C(14) | 108.7 (6) | C(43)–C(41)–C(44) | 109.1 (4) |
| Si(2)–C(21)–C(22) | 111.4 (3) | Si(5)–C(51)–C(52) | 111.5 (4) |
| Si(2)–C(21)–C(23) | 108.6 (4) | Si(5)–C(51)–C(53) | 110.6 (3) |
| C(22)–C(21)–C(23) | 107.4 (4) | C(52)–C(51)–C(53) | 107.3 (4) |
| Si(2)–C(21)–C(24) | 109.9 (3) | Si(5)–C(51)–C(54) | 109.5 (3) |
| C(22)–C(21)–C(24) | 109.6 (5) | C(52)–C(51)–C(54) | 109.1 (4) |
| C(23)–C(21)–C(24) | 109.9 (5) | C(53)–C(51)–C(54) | 108.7 (5) |
| Si(3)–C(31)–C(32) | 110.6 (4) | Si(6)–C(61)–C(62) | 111.1 (3) |
| Si(3)–C(31)–C(33) | 110.2 (4) | Si(6)–C(61)–C(63) | 110.8 (4) |
| C(32)–C(31)–C(33) | 108.7 (4) | C(62)–C(61)–C(63) | 110.8 (5) |
| Si(3)–C(31)–C(34) | 109.8 (4) | Si(6)–C(61)–C(64) | 109.3 (4) |
| C(32)–C(31)–C(34) | 108.5 (4) | C(62)–C(61)–C(64) | 106.6 (5) |
| C(33)–C(31)–C(34) | 109.0 (5) | C(63)–C(61)–C(64) | 107.9 (4) |
| N(5)–Si(6x)–N(6) | 93.8 (4) | C(61)–Si(6x)–F(6x) | 94.2 (10) |
| N(5)–Si(6x)–F(6x) | 122.4 (10) | N(6)–Si(6x)–F(6x) | 123.3 (10) |
| N(5)–Si(6x)–C(61) | 110.5 (5) | N(6)–Si(6x)–C(61) | 113.5 (6) |
| Si(5)–N(5)–Si(6x) | 116.3 (4) | Si(4)–N(6)–Si(6x) | 126.0 (4) |

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