

Table 2. Bond angles involving the H atoms with *e.s.d.*'s in parentheses

C(1)–C(2)–H(C2)	126 (6) <sup>o</sup>	C(3)–C(2)–H(C2)	119 (6) <sup>o</sup>
C(2)–C(3)–H(C3)	115 (6)	C(4)–C(3)–H(C3)	122 (6)
C(3)–C(4)–H(C4)	110 (5)	C(5)–C(4)–H(C4)	128 (5)
C(4)–C(5)–H(C5)	133 (8)	C(6)–C(5)–H(C5)	110 (8)
N(1)–C(9)–H(C9)	103 (6)	C(10)–C(9)–H(C9)	97 (7)
C(12)–C(9)–H(C9)	123 (7)	O(4)–C(11)–H(C11)	90 (4)
O(5)–C(11)–H(C11)	107 (5)	C(12)–C(11)–H(C11)	105 (5)
C(9)–C(12)–H1(C12)	122 (5)	C(11)–C(12)–H1(C12)	105 (5)
C(9)–C(12)–H2(C12)	93 (9)	C(11)–C(12)–H2(C12)	105 (9)
O(5)–C(13)–H1(C13)	110 (5)	C(14)–C(13)–H1(C13)	99 (5)
O(5)–C(13)–H2(C13)	99 (8)	C(14)–C(13)–H2(C13)	101 (8)
C(13)–C(14)–H1(C14)	89 (6)	C(13)–C(14)–H2(C14)	109 (8)
C(13)–C(14)–H3(C14)	111 (6)		

The bond lengths and angles are within the expected values and agree well with those found in the above-mentioned 4-methylthio-2-phthalimido- $\gamma$ -butyrolactone and in  $\alpha$ -(3,5-dibromo-2-hydroxybenzylidene)- $\gamma$ -butyrolactone (Koenig, Chiu, Krebs & Walter, 1969). Because of the limited number of observed data, owing to the poor quality of the crystals, the estimated standard deviations of the interatomic distances and

angles are rather large and detailed discussion of the individual bonds and angles would be meaningless.

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## *N*-(*tert*-Butylphenylfluorosilyl)-*N*-(phenyldifluorosilyl)-2,4,6-trimethylaniline

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**Abstract.**  $C_{25}H_{30}F_3NSi_2$ ,  $M_r = 457.69$ , orthorhombic, *Pbcn*,  $a = 26.999$  (7),  $b = 9.209$  (3),  $c = 19.896$  (5) Å,  $U = 4947$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.229$  Mg m<sup>-3</sup>,  $\mu(Cu K\alpha) = 1.5$  mm<sup>-1</sup>; final  $R = 0.063$  for 2739 unique reflexions. The bonding geometry at the N atom is approximately planar.

**Introduction.** The title compound is one of a series of bis(fluorosilyl)anilines synthesized by Klingebiel, Neemann & Meller (1978). Its <sup>19</sup>F NMR spectrum shows an *AB* pattern for the SiF<sub>2</sub> group. The structure determination was undertaken to determine the bonding geometry at the N atom.

Crystals in the form of white blocks were obtained from petroleum spirit. A crystal 0.5 × 0.3 × 0.2 mm was used to collect data on a Syntex P2<sub>1</sub> diffractometer with monochromated Cu  $K\alpha$  radiation. Cell dimensions were obtained by least squares from 15 strong high-angle reflexions. 3958 reflexions were

collected in the range  $3^\circ < 2\theta < 116^\circ$ . After application of Lp corrections, averaging equivalent reflexions gave 2743 unique reflexions with  $F > 4\sigma(F)$ . Systematic absences  $0kl$ ,  $k$  odd;  $h0l$ ,  $l$  odd; and  $hk0$ ,  $h + k$  odd indicated space group *Pbcn*.

The structure was solved by multiresolution  $\sum_2$  sign expansion with  $323 E > 1.6$ . The best *E* map showed all but four non-hydrogen atoms. The missing atoms were located by a difference synthesis. Least-squares refinement gave  $R = 0.17$  (isotropic) and 0.10 (anisotropic). H atoms were then included at calculated positions and refined with C–H 1.08 Å, H–C–H 109.5°. The methyl H atoms of the mesitylene moiety could not be refined successfully, probably because of rotational disorder, and so were omitted. Refinement terminated at  $R = 0.063$ ,  $R' = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0.070$ . The weighting scheme was  $w = 1/[\sigma^2(F) + 0.001F^2]$ . Overall temperature factors for methyl and non-methyl H refined to 0.13 (1) and 0.10 (1) Å<sup>2</sup> respectively. F

atoms of the SiF<sub>2</sub> group showed high temperature factors; other temperature factors were normal.

Final atomic coordinates are given in Table 1,\* bond lengths and angles in Tables 2 and 3. Diagrams of the structure are given in Figs. 1 and 2.

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34383 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

	x	y	z
Si(1)	3976 (1)	1293 (1)	4061 (1)
F(1)	4509 (1)	2026 (2)	4180 (1)
C(11)	3872 (1)	93 (4)	4798 (2)
C(12)	3411 (2)	-491 (4)	4947 (2)
C(13)	3341 (2)	-1438 (5)	5478 (2)
C(14)	3736 (2)	-1823 (5)	5869 (2)
C(15)	4202 (2)	-1256 (5)	5740 (2)
C(16)	4265 (2)	-309 (4)	5212 (2)
C(21)	4054 (1)	209 (4)	3270 (2)
C(22)	4229 (2)	1146 (6)	2676 (2)
C(23)	3583 (2)	-621 (6)	3075 (3)
C(24)	4466 (2)	-907 (5)	3423 (2)
N(1)	3534 (1)	2656 (3)	4058 (1)
C(41)	3309 (1)	3178 (4)	3430 (2)
C(42)	3555 (1)	4197 (4)	3037 (2)
C(43)	3341 (2)	4640 (4)	2433 (2)
C(44)	2893 (2)	4106 (4)	2216 (2)
C(45)	2650 (1)	3119 (4)	2623 (2)
C(46)	2844 (1)	2657 (4)	3237 (2)
C(52)	4050 (2)	4858 (5)	3255 (3)
C(54)	2667 (2)	4603 (5)	1550 (2)
C(56)	2540 (2)	1622 (4)	3664 (2)
Si(2)	3377 (1)	3548 (1)	4783 (1)
F(2)	2909 (1)	2839 (5)	5138 (1)
F(3)	3202 (2)	5098 (4)	4584 (2)
C(31)	3867 (1)	3674 (4)	5414 (2)
C(32)	3889 (2)	2768 (5)	5972 (2)
C(33)	4275 (2)	2913 (5)	6428 (2)
C(34)	4624 (2)	3931 (6)	6360 (3)
C(35)	4611 (2)	4845 (6)	5830 (3)
C(36)	4237 (2)	4721 (5)	5361 (2)
H(121)	3097	-196	4639
H(131)	2977	-1871	5583
H(141)	3685	-2571	6282
H(151)	4513	-1557	6051
H(161)	4629	133	5116
H(221)	3942	1877	2508
H(222)	4346	471	2262
H(223)	4540	1766	2859
H(231)	3286	104	2937
H(232)	3472	-1260	3505
H(233)	3665	-1334	2658
H(241)	4309	-1706	3759
H(242)	4790	-431	3652
H(243)	4568	-1424	2956
H(431)	3532	5428	2127
H(451)	2298	2689	2459
H(321)	3607	1955	6051
H(331)	4293	2180	6850
H(341)	4916	4022	6729
H(351)	4892	5671	5773
H(361)	4232	5459	4939

Table 2. *Bond lengths* (Å)

F(1)-Si(1)	1.606 (3)	C(11)-Si(1)	1.858 (5)
C(21)-Si(1)	1.874 (5)	N(1)-Si(1)	1.732 (4)
C(11)-C(12)	1.388 (6)	C(11)-C(16)	1.394 (4)
C(12)-C(13)	1.383 (7)	C(13)-C(14)	1.367 (8)
C(14)-C(15)	1.386 (8)	C(15)-C(16)	1.376 (7)
C(21)-C(22)	1.538 (7)	C(21)-C(23)	1.534 (7)
C(21)-C(24)	1.545 (7)	N(1)-C(41)	1.470 (6)
N(1)-Si(2)	1.712 (4)	C(41)-C(42)	1.391 (6)
C(41)-C(46)	1.398 (6)	C(42)-C(43)	1.395 (7)
C(42)-C(52)	1.531 (7)	C(43)-C(44)	1.374 (7)
C(44)-C(45)	1.383 (7)	C(44)-C(54)	1.528 (7)
C(45)-C(46)	1.395 (6)	C(46)-C(56)	1.518 (6)
F(2)-Si(2)	1.590 (4)	F(3)-Si(2)	1.554 (5)
C(31)-Si(2)	1.828 (5)	C(31)-C(32)	1.391 (7)
C(31)-C(36)	1.392 (7)	C(32)-C(33)	1.386 (8)
C(33)-C(34)	1.336 (8)	C(34)-C(35)	1.350 (9)
C(35)-C(36)	1.378 (8)		

Table 3. *Bond angles* (°)

C(11)-Si(1)-F(1)	105.6 (2)	C(43)-C(42)-C(41)	118.9 (5)
C(21)-Si(1)-F(1)	104.3 (3)	C(52)-C(42)-C(41)	121.7 (5)
C(21)-Si(1)-C(11)	111.3 (3)	C(52)-C(42)-C(43)	119.4 (5)
N(1)-Si(1)-F(1)	108.2 (2)	C(44)-C(43)-C(42)	122.1 (5)
N(1)-Si(1)-C(11)	109.2 (3)	C(45)-C(44)-C(43)	117.9 (5)
N(1)-Si(1)-C(21)	117.4 (3)	C(54)-C(44)-C(43)	121.1 (5)
C(12)-C(11)-Si(1)	122.3 (4)	C(54)-C(44)-C(45)	121.0 (5)
C(16)-C(11)-Si(1)	120.7 (4)	C(46)-C(45)-C(44)	122.4 (5)
C(16)-C(11)-C(12)	117.0 (4)	C(45)-C(46)-C(41)	118.2 (4)
C(13)-C(12)-C(11)	122.0 (5)	C(56)-C(46)-C(41)	123.2 (4)
C(14)-C(13)-C(12)	119.5 (5)	C(56)-C(46)-C(45)	118.6 (4)
C(15)-C(14)-C(13)	120.3 (5)	F(2)-Si(2)-N(1)	112.0 (3)
C(15)-C(16)-C(11)	121.7 (5)	F(3)-Si(2)-N(1)	107.6 (3)
C(16)-C(15)-C(14)	119.6 (5)	F(3)-Si(2)-F(2)	104.4 (3)
C(22)-C(21)-Si(1)	112.4 (4)	C(31)-Si(2)-N(1)	115.5 (3)
C(23)-C(21)-Si(1)	112.7 (4)	C(31)-Si(2)-F(2)	107.3 (3)
C(23)-C(21)-C(22)	109.8 (5)	C(31)-Si(2)-F(3)	109.6 (3)
C(24)-C(21)-Si(1)	105.7 (4)	C(32)-C(31)-Si(2)	122.8 (4)
C(24)-C(21)-C(22)	107.7 (5)	C(36)-C(31)-Si(2)	120.8 (4)
C(24)-C(21)-C(23)	108.3 (5)	C(36)-C(31)-C(32)	116.4 (5)
C(41)-N(1)-Si(1)	121.6 (3)	C(33)-C(32)-C(31)	119.8 (5)
Si(2)-N(1)-Si(1)	121.0 (3)	C(34)-C(33)-C(32)	122.1 (6)
Si(2)-N(1)-C(41)	117.2 (3)	C(35)-C(34)-C(33)	119.9 (6)
C(42)-C(41)-N(1)	120.1 (4)	C(36)-C(35)-C(34)	119.7 (6)
C(46)-C(41)-N(1)	119.5 (4)	C(35)-C(36)-C(31)	122.1 (5)
C(46)-C(41)-C(42)	120.4 (4)		

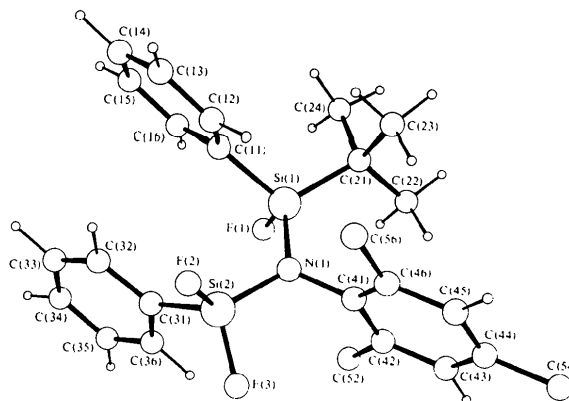


Fig. 1. A molecule of the title compound, showing the atom labelling.

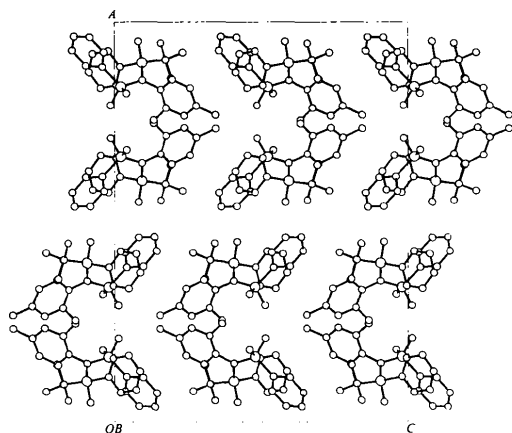


Fig. 2. Packing diagram projected down **b**. H atoms are omitted for clarity.

**Discussion.** The geometry at the N atom is approximately planar, with angles at N of 121.6, 121.0 and 117.2° and deviations Si(1) 0.01, N(1) -0.03, Si(2) 0.01, C(41) 0.01 Å from the mean plane of the four atoms. This may be attributed to the familiar  $d\pi-p\pi$  bonding in Si-N compounds, typified by trisilylamine (Beagley & Conrad, 1970). The N-Si distances are significantly different [1.732 (4) and 1.712 (4) Å respectively for N(1)-Si(1) and N(1)-Si(2)]. This may be due to the greater electronegativity of the SiF<sub>2</sub> group inducing more  $d\pi-p\pi$  bonding between N(1) and Si(2). An alternative explanation, however, is a change in

covalent radii on changing the electronegativities of the substituents; thus the Si-H length (determined by microwave spectroscopy) changes from  $1.485 \pm 0.01$  Å in SiH<sub>3</sub>F (Kewley, McKinney & Robiette, 1970) to  $1.455 \pm 0.01$  Å in SiHF<sub>3</sub> (Heath, Thomas & Sheridan, 1954), and there are clearly no possible  $\pi$  interactions for the Si-H bond. The mesitylene ring cannot be involved in  $\pi$  interactions with the Si-N-Si system as it is approximately perpendicular to it [torsion angles Si(1)-N(1)-C(41)-C(42) -82.1°, Si(2)-N(1)-C(41)-C(42) 92.9°].

The only short non-bonded contact is C(56)⋯F(3) 3.06 Å (second atom at  $\frac{1}{2} - x, -\frac{1}{2} + y, z$ ).

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## Melitracene Hydrobromide: 9-(3-Dimethylaminopropylidene)-10,10-dimethyl-9,10-dihydroanthracene Hydrobromide

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**Abstract.** C<sub>21</sub>H<sub>26</sub>N<sup>+</sup>.Br<sup>-</sup>, *M<sub>r</sub>* = 372.36, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 17.680 (2), *b* = 10.3084 (4), *c* = 22.618 (2) Å,  $\beta$  = 107.92 (1)°, *U* = 3922 (1) Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.25 Mg m<sup>-3</sup>,  $\mu$ (Mo *K*α) = 2.577 mm<sup>-1</sup>. Melitracene is a tricyclic antidepressant. Its structure was refined to *R* = 0.064 for 2813 independent reflexions. The molecules are held together by salt bridges and van der

Waals forces. The molecular conformation and other data of pharmacological interest are given.

**Introduction.** From this study of the crystal structure of melitracene and studies of other antidepressant drugs in progress, we have accumulated information about their conformations and charge distributions from which