

méthylpyridin-2-yl)(furan-2-yl)carboxamide (Rodier, Cense, Robert & Le Baut, 1991).

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## Redetermination of the Structure of *N*-(Trimethylsilyl)dimesylamine [(H<sub>3</sub>C)<sub>3</sub>SiN(SO<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>] at 178 K

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**Abstract.** C<sub>5</sub>H<sub>15</sub>NO<sub>4</sub>S<sub>2</sub>Si, *M*<sub>r</sub> = 245.4, monoclinic, *P*2<sub>1</sub>, *a* = 7.746 (3), *b* = 11.965 (5), *c* = 12.538 (4) Å, β = 91.86 (3)°, *V* = 1161.4 (8) Å<sup>3</sup>, *Z* = 4, *D*<sub>x</sub> = 1.403 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 0.528 mm<sup>-1</sup>, *F*(000) = 520, *T* = 178 K, *R* = 0.031 for 4405 reflections. The two independent molecules in the asymmetric unit display no major differences. The N—Si bonds are long [1.850 (2), 1.853 (2) Å]. The geometry at nitrogen is essentially planar (the N atoms lie 0.03, 0.01 Å out of the plane of their three substituents).

**Experimental.** A colourless tablet 0.5 × 0.5 × 0.2 mm was mounted in inert oil and rapidly transferred to the cold gas stream of the diffractometer (Siemens R3 with LT-2 low-temperature attachment). Intensities were registered to 2θ<sub>max</sub> = 55° using highly oriented graphite-crystal-monochromated Mo *K*α radiation. ω scan technique, scan speed 4.9° min<sup>-1</sup>, range 1.00° (in ω). Of 5120 measured reflections (hemisphere + *h* ± *k* ± *l* except for inaccessible reflections), 4706 were unique (Friedel opposites not merged, *R*<sub>int</sub> 0.021, index ranges *h* 0 to 10, *k* -15 to 9, *l* -16 to 16) and 4405 [*F* > 4σ(*F*)] considered observed. The orientation matrix was refined from setting angles of 47 reflections in the 2θ range 20–23°. Three check reflections showed no significant intensity variation. No absorption correction was applied.

The structure was solved by direct methods and subjected to anisotropic full-matrix least-squares refinement on *F* [Σ*w*(*F*<sub>o</sub> - *F*<sub>c</sub>)<sup>2</sup> minimized]. Program system: *SHELXTL-Plus* (Sheldrick, 1989) on a MicroVAX II. H atoms were included using a riding model. The absolute structure was determined by an η refinement [η = 1.32 (13)]. The weighting scheme was *w*<sup>-1</sup> = σ<sup>2</sup>(*F*) + 0.0005*F*<sup>2</sup>; the final *R* was 0.031, with *wR* 0.039 (for all data *R* was 0.036, *wR* 0.043). 261 parameters; data to parameter ratio 16.9:1; *S* 1.3; maximum Δ/σ 0.005; maximum Δρ 0.68, -0.45 e Å<sup>-3</sup>. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atom coordinates are given in Table 1, with derived bond lengths and angles in Table 2.\* Fig. 1 shows the two independent molecules and the numbering scheme.

**Related literature.** The structure was determined at room temperature by Schomburg, Blaschette & Wieland (1986); their comments on the structure, in particular concerning the long Si—N bonds, are still valid but they appear to have given the wrong space

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54837 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0087]

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^4$ )

$U_{eq}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$
Si	4032.1 (8)	2766	3659.3 (5)	272 (2)
Si(1)	1042.6 (9)	2377.5 (9)	5107.1 (5)	391 (2)
S(2)	157.5 (7)	2766.0 (8)	2911.2 (4)	286 (2)
C(1)	4398 (3)	2982 (3)	2226 (2)	446 (10)
C(2)	4782 (4)	3990 (3)	4434 (3)	448 (10)
C(3)	5074 (4)	1431 (3)	4063 (3)	443 (10)
C(13)	116 (5)	3615 (3)	5561 (2)	530 (11)
C(23)	-75 (4)	1413 (3)	2392 (3)	466 (10)
O(11)	2621 (3)	2201 (2)	5718 (2)	531 (8)
O(12)	-216 (3)	1507 (2)	5056 (2)	579 (8)
O(21)	812 (2)	3477 (2)	2102 (1)	375 (6)
O(22)	-1424 (2)	3087 (2)	3382 (2)	416 (6)
N	1682 (2)	2663 (2)	3872 (1)	293 (6)
Si'	4014.8 (7)	7682.3 (8)	1473.6 (5)	254 (2)
S(1')	7843.9 (7)	8523.1 (8)	1490.9 (4)	264 (2)
S(2')	7077.5 (7)	6270.2 (8)	894.2 (5)	296 (2)
C(1')	3546 (3)	9100 (3)	1962 (3)	409 (9)
C(2')	3397 (4)	6638 (3)	2481 (3)	414 (9)
C(3')	2907 (3)	7522 (3)	147 (2)	382 (9)
C(13')	8034 (4)	9143 (3)	231 (2)	381 (9)
O(23')	8072 (4)	5642 (3)	2029 (3)	430 (9)
O(11')	9457 (2)	7997 (2)	1783 (1)	360 (6)
O(12')	7169 (2)	9324 (2)	2209 (2)	387 (6)
O(21')	8323 (2)	6427 (2)	92 (2)	405 (6)
O(22')	5542 (2)	5648 (2)	626 (2)	416 (6)
N'	6365 (2)	7518 (2)	1303 (2)	259 (6)

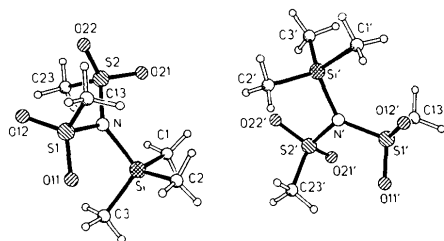


Fig. 1. The two independent molecules of the title compound, showing the atom-numbering scheme. Radii are arbitrary.

group and *Z* value ( $P2_1/m$ ,  $Z = 8$  instead of  $P2_1$ ,  $Z = 4$ ). The redetermination is appreciably more precise (4405 unique observed reflections, 2635 if Friedel opposites merged, *cf.* 1753 in the older determina-

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

Si—C(1)	1.847 (3)	Si—C(2)	1.841 (3)
Si—C(3)	1.853 (3)	Si—N	1.853 (2)
S(1)—C(13)	1.748 (4)	S(1)—O(11)	1.437 (2)
S(1)—O(12)	1.427 (3)	S(1)—N	1.676 (2)
S(2)—C(23)	1.752 (4)	S(2)—O(21)	1.430 (2)
S(2)—O(22)	1.429 (2)	S(2)—N	1.664 (2)
Si'—C(1')	1.844 (3)	Si'—C(2')	1.850 (3)
Si'—C(3')	1.857 (3)	Si'—N'	1.850 (2)
S(1')—C(13')	1.756 (3)	S(1')—O(11')	1.436 (2)
S(1')—O(12')	1.426 (2)	S(1')—N'	1.672 (2)
S(2')—C(23')	1.764 (3)	S(2')—O(21')	1.429 (2)
S(2')—O(22')	1.434 (2)	S(2')—N'	1.678 (2)
C(1)—Si—C(2)	110.3 (2)	C(1)—Si—C(3)	107.9 (2)
C(2)—Si—C(3)	114.5 (1)	C(1)—Si—N	109.4 (1)
C(2)—Si—N	105.8 (1)	C(3)—Si—N	108.9 (1)
C(13)—S(1)—O(11)	107.6 (2)	C(13)—S(1)—O(12)	110.2 (2)
O(11)—S(1)—O(12)	119.0 (2)	C(13)—S(1)—N	105.3 (1)
O(11)—S(1)—N	104.5 (1)	O(12)—S(1)—N	109.2 (1)
C(23)—S(2)—O(21)	108.7 (1)	C(23)—S(2)—O(22)	108.8 (1)
O(21)—S(2)—O(22)	117.5 (1)	C(23)—S(2)—N	105.2 (1)
O(21)—S(2)—N	107.4 (1)	O(22)—S(2)—N	108.6 (1)
Si—N—S(1)	117.9 (1)	Si—N—S(2)	124.6 (1)
S(1)—N—S(2)	117.4 (1)	C(1')—Si'—C(2')	109.7 (1)
C(1')—Si'—C(3')	107.6 (1)	C(2')—Si'—C(3')	114.8 (1)
C(1')—Si'—N'	109.9 (1)	C(2')—Si'—N'	106.5 (1)
C(13')—Si'—N'	108.2 (1)	C(13')—S(1')—O(11')	108.5 (1)
C(13')—S(1')—O(12')	109.1 (1)	O(11')—S(1')—O(12')	117.9 (1)
C(13')—S(1')—N'	104.7 (1)	O(11')—S(1')—N'	107.9 (1)
O(12')—S(1')—N'	108.0 (1)	C(23')—S(2')—O(21')	109.7 (1)
C(23')—S(2')—O(22')	107.8 (1)	O(21')—S(2')—O(22')	118.5 (1)
C(23')—S(2')—N'	105.8 (1)	O(21')—S(2')—N'	109.5 (1)
O(22')—S(2')—N'	104.8 (1)	Si'—N'—S(1')	125.4 (1)
Si'—N'—S(2')	117.7 (1)	Si(1')—N'—S(2')	116.9 (1)

tion; typical standard deviations of bond lengths are reduced by a factor of 3–4).

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