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N-(Trimethylsilyl)methanesulfonamide

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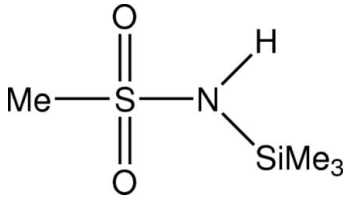
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(Si–C) = 0.003 Å; R factor = 0.039; wR factor = 0.094; data-to-parameter ratio = 28.8.

There are two molecules in the asymmetric unit of the title compound, $C_4H_{13}NO_2SSi$. In the crystal, molecules are linked *via* intermolecular N—H···O hydrogen bonds, forming chains along [001]. The crystal studied was an inversion twin, the refined ratio of twin domains being 0.61 (9):0.39 (9).

Related literature

For the original synthesis of the title compound, see: Roy (1993). For the synthetic application of the title compound, see: Roy *et al.* (1993). For related structures, see: Ni *et al.* (1995); Chunechom *et al.* (1998).



Experimental

Crystal data

$C_4H_{13}NO_2SSi$
M_r = 167.30
Monoclinic, $P2_1$
a = 8.2827 (4) Å
b = 10.9513 (5) Å
c = 9.6201 (3) Å
β = 92.536 (2)°
V = 871.75 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 150 K
0.32 × 0.25 × 0.24 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing 1995)
T_{min} = 0.830, T_{max} = 0.931
6920 measured reflections
4894 independent reflections
4195 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.094
S = 1.05
4894 reflections
170 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}
Absolute structure: Flack (1983), 1787 Friedel pairs
Flack parameter: 0.39 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1B—H1NB···O2A	0.81 (2)	2.11 (2)	2.917 (3)	173 (3)
N1A—H1NA···O2B^{i}	0.81 (2)	2.12 (2)	2.925 (3)	177 (3)

Symmetry code: (i) $x, y, z+1$.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL*.

AM would like to thank Ryerson University's Faculty of Engineering, Architecture and Science for funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2314).

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supporting information

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N-(Trimethylsilyl)methanesulfonamide

Andrew R. McWilliams, Sossina Gezahegna and Alan J. Lough

S1. Comment

N-trimethylsilylmethylsulfonamide, a key intermediate in the synthesis of polyoxothiazenes (Roy *et al.*, 1993) and polythionylphosphazenes (Chunechom *et al.*, 1998), was prepared *via* the reaction of methanesulfonyl chloride and hexamethyldisilazane (Roy, 1993). The asymmetric unit of the title compound, which contains two independent molecules, is shown in Fig. 1. The S—N bond distances in each molecule are intermediate between a typical S—N single bond (1.74 Å) and a typical S=N double bond (1.54 Å), (Ni *et al.*, 1995) suggesting the presence of some π-bonding between the sulfur and nitrogen atoms. The S—N—Si bond angles of 127.83 (14)° and 128.59 (14)Å are larger than might be expected, in terms of hybridization priciples, for either a tetrahedral or trigonal planar geometry about the nitrogen atom. In the crystal structure, molecules are linked *via* intermolecular N—H···O hydrogen bonds to form one-dimensional chains along [001] (Fig. 2).

S2. Experimental

The title compound was prepared *via* addition of methanesulfonyl chloride (7 ml, 102.5 mmol) to a three-necked round-bottom flask equipped with a magnetic stirring bar, gas inlet, reflux condenser and a rubber septa under an inert N_2 atmosphere. Hexamethyldisilazane (20 ml, 103.1 mmol) was added drop wise over 10 minutes with stirring at ambient temperatures. The flask was then placed into an oil bath and the reaction mixture heated to 363–373 K to initiate the reaction. The temperature of the oil bath was increased to between 388–393 K and the reaction mixture refluxed at this temperature for 2 h. The reaction mixture was allowed to cool to room temperature and the reaction by-product (Me_3SiCl) was removed *in vacuo*. The resulting crude white powder was recrystallized from a CH_2Cl_2/Hexane mixture producing colourless crystals. (Yield = 15.6 g, 91%).

S3. Refinement

Hydrogen atoms were placed in calculated positions with C—H distances ranging from 0.98 Å and included in the refinement in a riding-model approximation with U_{iso}(H) = 1.5U_{eq}(C). The positional parameters of the H atoms bonded to N atoms were refined independently and with U_{iso}(H) = 1.5U_{eq}(N). The N—H distances were constrained to be the same in each molecule [0.81 (2) Å] using the SADI command in *SHELXL* (Sheldrick, 2008).

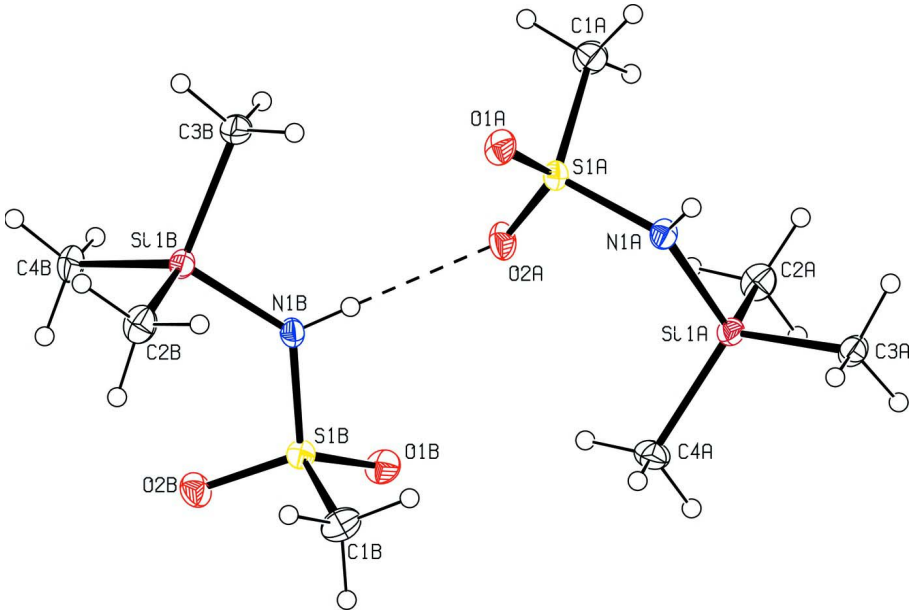

Figure 1

The asymmetric unit of title compound showing 30% probability ellipsoids. The dashed line indicates a hydrogen bond.

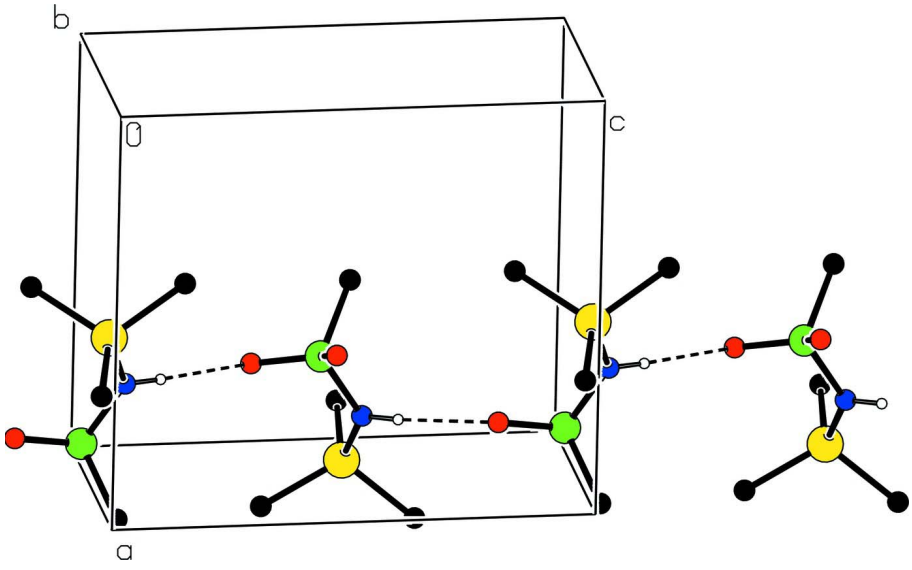

Figure 2

Part of the crystal structure showing hydrogen bonds as dashed lines.

N-(Trimethylsilyl)methanesulfonamide

Crystal data

$C_4H_{13}NO_2SSi$
$M_r = 167.30$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 8.2827$ (4) Å
$b = 10.9513$ (5) Å
$c = 9.6201$ (3) Å
$\beta = 92.536$ (2)°
$V = 871.75$ (6) Å^3
$Z = 4$
$F(000) = 360$
$D_x = 1.275$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6920 reflections

θ = 2.8–32.0°
μ = 0.45 mm^{-1}
T = 150 K

Block, colourless
0.32 × 0.25 × 0.24 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing 1995)
T_{min} = 0.830, T_{max} = 0.931

6920 measured reflections
4894 independent reflections
4195 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 32.0°, θ_{min} = 2.8°
h = −12→12
k = −14→16
l = −12→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.094
S = 1.05
4894 reflections
170 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2 + 0.660P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}
Absolute structure: Flack (1983), 1787 Friedel pairs
Absolute structure parameter: 0.39 (9)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1A	0.84555 (7)	0.27915 (6)	0.95708 (6)	0.02134 (13)
Si1A	0.55749 (8)	0.11459 (7)	0.99573 (7)	0.02169 (14)
O1A	0.8724 (3)	0.40683 (18)	0.9815 (2)	0.0303 (4)
O2A	0.8188 (2)	0.2381 (2)	0.81562 (18)	0.0308 (4)
N1A	0.6945 (3)	0.2334 (2)	1.0411 (2)	0.0228 (4)
H1NA	0.693 (4)	0.265 (3)	1.117 (2)	0.027*
C1A	1.0156 (4)	0.2012 (3)	1.0271 (3)	0.0329 (6)
H1AA	1.1115	0.2264	0.9783	0.049*
H1AB	0.9994	0.1131	1.0158	0.049*
H1AC	1.0307	0.2207	1.1262	0.049*
C2A	0.6695 (4)	−0.0301 (3)	0.9698 (3)	0.0326 (6)

H2AA	0.7328	−0.0505	1.0552	0.049*
H2AB	0.7423	−0.0201	0.8930	0.049*
H2AC	0.5926	−0.0960	0.9476	0.049*
C3A	0.4308 (3)	0.1074 (3)	1.1496 (3)	0.0296 (6)
H3AA	0.3737	0.1851	1.1595	0.044*
H3AB	0.4997	0.0922	1.2332	0.044*
H3AC	0.3520	0.0411	1.1374	0.044*
C4A	0.4362 (4)	0.1526 (3)	0.8351 (3)	0.0322 (6)
H4AA	0.3771	0.2288	0.8490	0.048*
H4AB	0.3593	0.0866	0.8136	0.048*
H4AC	0.5082	0.1626	0.7577	0.048*
S1B	0.65985 (7)	0.31234 (6)	0.45178 (6)	0.02162 (13)
Si1B	0.94662 (8)	0.47963 (7)	0.51424 (7)	0.02164 (14)
O1B	0.6327 (3)	0.18445 (19)	0.4738 (2)	0.0310 (5)
O2B	0.6801 (2)	0.3547 (2)	0.31117 (19)	0.0307 (4)
N1B	0.8160 (3)	0.3552 (2)	0.5432 (2)	0.0223 (4)
H1NB	0.820 (3)	0.317 (3)	0.615 (2)	0.027*
C1B	0.4928 (4)	0.3904 (3)	0.5154 (3)	0.0340 (7)
H1BA	0.3947	0.3668	0.4613	0.051*
H1BB	0.5096	0.4787	0.5068	0.051*
H1BC	0.4811	0.3694	0.6134	0.051*
C2B	0.8316 (4)	0.6243 (3)	0.5137 (3)	0.0329 (6)
H2BA	0.7775	0.6327	0.6018	0.049*
H2BB	0.7506	0.6239	0.4363	0.049*
H2BC	0.9057	0.6930	0.5027	0.049*
C3B	1.0911 (3)	0.4706 (3)	0.6667 (3)	0.0309 (6)
H3BA	1.0326	0.4805	0.7523	0.046*
H3BB	1.1719	0.5355	0.6607	0.046*
H3BC	1.1451	0.3910	0.6677	0.046*
C4B	1.0488 (3)	0.4616 (3)	0.3477 (3)	0.0304 (6)
H4BA	0.9686	0.4671	0.2699	0.046*
H4BB	1.1023	0.3818	0.3460	0.046*
H4BC	1.1294	0.5264	0.3392	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0233 (3)	0.0193 (3)	0.0216 (3)	−0.0012 (2)	0.0028 (2)	0.0028 (2)
Si1A	0.0216 (3)	0.0221 (4)	0.0213 (3)	−0.0027 (3)	0.0008 (2)	−0.0005 (3)
O1A	0.0340 (11)	0.0163 (10)	0.0406 (11)	−0.0031 (8)	0.0019 (9)	0.0038 (9)
O2A	0.0417 (11)	0.0324 (11)	0.0187 (8)	−0.0058 (9)	0.0065 (7)	0.0011 (8)
N1A	0.0264 (10)	0.0239 (12)	0.0184 (10)	−0.0044 (9)	0.0032 (8)	−0.0033 (9)
C1A	0.0266 (13)	0.0273 (16)	0.0448 (17)	0.0039 (11)	0.0010 (12)	0.0066 (13)
C2A	0.0354 (15)	0.0216 (15)	0.0409 (15)	−0.0002 (11)	0.0039 (12)	−0.0032 (13)
C3A	0.0254 (12)	0.0378 (16)	0.0258 (12)	−0.0083 (11)	0.0038 (9)	−0.0004 (12)
C4A	0.0308 (14)	0.0392 (18)	0.0258 (13)	−0.0014 (11)	−0.0066 (10)	−0.0018 (12)
S1B	0.0240 (3)	0.0198 (3)	0.0211 (3)	−0.0010 (2)	0.0015 (2)	−0.0029 (2)
Si1B	0.0230 (3)	0.0216 (4)	0.0205 (3)	−0.0035 (3)	0.0030 (2)	0.0007 (3)

O1B	0.0349 (11)	0.0190 (11)	0.0387 (11)	−0.0052 (8)	−0.0021 (8)	−0.0036 (9)
O2B	0.0388 (11)	0.0333 (11)	0.0200 (8)	−0.0036 (8)	0.0005 (7)	−0.0021 (8)
N1B	0.0258 (10)	0.0209 (11)	0.0201 (10)	−0.0035 (8)	0.0007 (8)	0.0041 (9)
C1B	0.0244 (13)	0.0339 (18)	0.0443 (17)	0.0034 (11)	0.0060 (11)	−0.0082 (14)
C2B	0.0368 (15)	0.0209 (14)	0.0415 (16)	−0.0004 (12)	0.0057 (12)	0.0003 (13)
C3B	0.0277 (13)	0.0395 (17)	0.0253 (12)	−0.0071 (12)	−0.0015 (10)	0.0003 (13)
C4B	0.0321 (14)	0.0340 (17)	0.0260 (13)	−0.0037 (11)	0.0106 (10)	0.0026 (12)

Geometric parameters (Å, º)

S1A—O1A	1.434 (2)	S1B—O1B	1.436 (2)
S1A—O2A	1.4410 (19)	S1B—O2B	1.4469 (19)
S1A—N1A	1.600 (2)	S1B—N1B	1.602 (2)
S1A—C1A	1.755 (3)	S1B—C1B	1.759 (3)
Si1A—N1A	1.768 (2)	Si1B—N1B	1.769 (2)
Si1A—C4A	1.853 (3)	Si1B—C2B	1.849 (3)
Si1A—C3A	1.853 (3)	Si1B—C3B	1.854 (3)
Si1A—C2A	1.859 (3)	Si1B—C4B	1.855 (3)
N1A—H1NA	0.81 (2)	N1B—H1NB	0.81 (2)
C1A—H1AA	0.9800	C1B—H1BA	0.9800
C1A—H1AB	0.9800	C1B—H1BB	0.9800
C1A—H1AC	0.9800	C1B—H1BC	0.9800
C2A—H2AA	0.9800	C2B—H2BA	0.9800
C2A—H2AB	0.9800	C2B—H2BB	0.9800
C2A—H2AC	0.9800	C2B—H2BC	0.9800
C3A—H3AA	0.9800	C3B—H3BA	0.9800
C3A—H3AB	0.9800	C3B—H3BB	0.9800
C3A—H3AC	0.9800	C3B—H3BC	0.9800
C4A—H4AA	0.9800	C4B—H4BA	0.9800
C4A—H4AB	0.9800	C4B—H4BB	0.9800
C4A—H4AC	0.9800	C4B—H4BC	0.9800
O1A—S1A—O2A	118.36 (12)	O1B—S1B—O2B	118.44 (12)
O1A—S1A—N1A	110.00 (13)	O1B—S1B—N1B	109.44 (12)
O2A—S1A—N1A	106.77 (12)	O2B—S1B—N1B	107.15 (12)
O1A—S1A—C1A	107.19 (15)	O1B—S1B—C1B	106.99 (15)
O2A—S1A—C1A	107.34 (15)	O2B—S1B—C1B	107.15 (15)
N1A—S1A—C1A	106.61 (14)	N1B—S1B—C1B	107.15 (14)
N1A—Si1A—C4A	111.04 (13)	N1B—Si1B—C2B	110.00 (13)
N1A—Si1A—C3A	102.36 (12)	N1B—Si1B—C3B	102.23 (12)
C4A—Si1A—C3A	111.74 (14)	C2B—Si1B—C3B	111.25 (15)
N1A—Si1A—C2A	109.97 (13)	N1B—Si1B—C4B	111.06 (13)
C4A—Si1A—C2A	109.58 (15)	C2B—Si1B—C4B	110.09 (15)
C3A—Si1A—C2A	111.98 (15)	C3B—Si1B—C4B	112.00 (13)
S1A—N1A—Si1A	127.83 (14)	S1B—N1B—Si1B	128.59 (14)
S1A—N1A—H1NA	112 (2)	S1B—N1B—H1NB	109 (2)
Si1A—N1A—H1NA	120 (2)	Si1B—N1B—H1NB	122 (2)
S1A—C1A—H1AA	109.5	S1B—C1B—H1BA	109.5

S1A—C1A—H1AB	109.5	S1B—C1B—H1BB	109.5
H1AA—C1A—H1AB	109.5	H1BA—C1B—H1BB	109.5
S1A—C1A—H1AC	109.5	S1B—C1B—H1BC	109.5
H1AA—C1A—H1AC	109.5	H1BA—C1B—H1BC	109.5
H1AB—C1A—H1AC	109.5	H1BB—C1B—H1BC	109.5
Si1A—C2A—H2AA	109.5	Si1B—C2B—H2BA	109.5
Si1A—C2A—H2AB	109.5	Si1B—C2B—H2BB	109.5
H2AA—C2A—H2AB	109.5	H2BA—C2B—H2BB	109.5
Si1A—C2A—H2AC	109.5	Si1B—C2B—H2BC	109.5
H2AA—C2A—H2AC	109.5	H2BA—C2B—H2BC	109.5
H2AB—C2A—H2AC	109.5	H2BB—C2B—H2BC	109.5
Si1A—C3A—H3AA	109.5	Si1B—C3B—H3BA	109.5
Si1A—C3A—H3AB	109.5	Si1B—C3B—H3BB	109.5
H3AA—C3A—H3AB	109.5	H3BA—C3B—H3BB	109.5
Si1A—C3A—H3AC	109.5	Si1B—C3B—H3BC	109.5
H3AA—C3A—H3AC	109.5	H3BA—C3B—H3BC	109.5
H3AB—C3A—H3AC	109.5	H3BB—C3B—H3BC	109.5
Si1A—C4A—H4AA	109.5	Si1B—C4B—H4BA	109.5
Si1A—C4A—H4AB	109.5	Si1B—C4B—H4BB	109.5
H4AA—C4A—H4AB	109.5	H4BA—C4B—H4BB	109.5
Si1A—C4A—H4AC	109.5	Si1B—C4B—H4BC	109.5
H4AA—C4A—H4AC	109.5	H4BA—C4B—H4BC	109.5
H4AB—C4A—H4AC	109.5	H4BB—C4B—H4BC	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*B*—H1*NB*···O2*A*	0.81 (2)	2.11 (2)	2.917 (3)	173 (3)
N1*A*—H1*NA*···O2*B*^{i}	0.81 (2)	2.12 (2)	2.925 (3)	177 (3)

Symmetry code: (i) *x*, *y*, *z*+1.