organic papers

(Chloromethyl)trimethylsilane at 160 K

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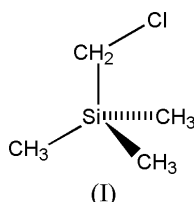
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Key indicators

Single-crystal X-ray study
$T = 160$ K
Mean σ(Si–C) = 0.004 Å
R factor = 0.040
wR factor = 0.115
Data-to-parameter ratio = 23.3

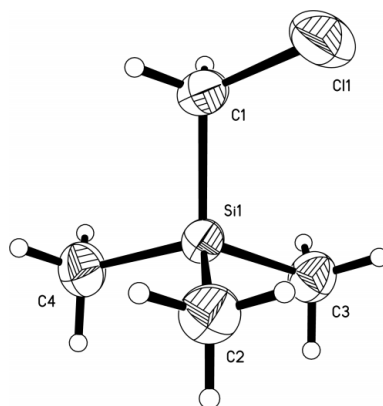For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

(Chloromethyl)trimethylsilane, Me_3SiCH_2Cl or $C_4H_{11}ClSi$, is a liquid at room temperature, and it was crystallized using *in situ* methods. The C—Si—C bond angles involving the chloromethyl group are somewhat smaller than those involving only methyl groups [105.5 (2)–109.47 (19)° *versus* 110.01 (19)–111.2 (2)°], which is ascribable to both the electronegative and the steric effects of the Cl atom.

Comment

(Chloromethyl)trimethylsilane, (I), is a liquid under ambient conditions, and a crystal was obtained by *in situ* crystallization of a sample held in a hand-drawn Pyrex capillary (Boese & Nussbaumer, 1994).

(I)

Molecules of (I) adopt the expected tetrahedral configuration at Si (Fig. 1). Si—C bond distances fall into the range 1.848 (4)–1.880 (4) Å, although to within experimental error the bond distances and angles have C_s symmetry, with a mirror plane passing through atoms Si1, C1, Cl1 and C4; the C4—Si1—C1—Cl1 torsion angle [175.3 (2)°] shows a somewhat more significant deviation from the symmetry. The bond angles at atom Si1 involving the more electronegative CH_2Cl group are smaller [105.5 (2)–109.47 (19)°] than those involving only methyl groups [110.01 (19)–111.2 (2)°]. The smaller

Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented as spheres of arbitrary radii.

magnitude of C1—Si1—C4 [105.5 (2)°] relative to C1—Si1—C2 [109.4 (2)°] and C1—Si1—C3 [109.47 (19)°] presumably reflects the steric influence of the Cl atom.

The only intermolecular interactions falling within the sum of the van der Waals radii (Bondi, 1964) of the participating atoms are weak Cl1···H33[i] [symmetry code (i): $-x$, $-y$, $z - \frac{1}{2}$] interactions (2.93 Å; the sum of the van der Waals radii of Cl and H is 2.95 Å). These result in chains that spiral about the 2_1 axis parallel to the c direction (Fig. 2).

Experimental

A sample of (I) was obtained from Aldrich and used as received. Compound (I) is a liquid under ambient conditions and it was crystallized *in situ* in a capillary (o.d. 0.34 mm) mounted on the diffractometer. A crystal was grown by first establishing a seed in a small volume of the liquid at 182.8 K, and then cooling at a rate of 10 K h⁻¹. The sample was then cooled to 160 K for data collection.

Crystal data

$C_4H_{11}ClSi$	Mo $K\alpha$ radiation
$M_r = 122.67$	Cell parameters from 72
Orthorhombic, $Pna2_1$	reflections
$a = 13.8776 (13)$ Å	$\theta = 15$–$16°$
$b = 6.3855 (9)$ Å	$\mu = 0.56$ mm⁻¹
$c = 8.4000 (10)$ Å	$T = 160$ K
$V = 744.37 (15)$ Å³	Cylinder, colourless
$Z = 4$	$0.50 \times 0.39 \times 0.39$ mm
$D_x = 1.095$ Mg m⁻³	0.50 mm length, 0.39 mm radius

Data collection

Stoe STADI-4 diffractometer equipped with an Oxford Cryo-systems low-temperature device (Cosier & Glazer, 1986)	1306 independent reflections
	1186 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.030$
	$\theta_{max} = 25.0°$
ω–θ scans	$h = -1 \rightarrow 16$
Absorption correction: ψ scan [azimuthal absorption correction (North *et al.*, 1968) applied using *XPREP* (Sheldrick, 1997)]	$k = -7 \rightarrow 7$
	$l = -9 \rightarrow 9$
	3 standard reflections
$T_{min} = 0.741$, $T_{max} = 0.804$	frequency: 60 min
3768 measured reflections	intensity decay: none

Refinement

Refinement on F^2	where $P = 0.3333\max(0,F_o^2)$
$R[F^2 > 2\sigma(F^2)] = 0.040$	$+ 0.6667F_c^2$
$wR(F^2) = 0.115$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.43$ e Å⁻³
1305 reflections	$\Delta\rho_{min} = -0.32$ e Å⁻³
56 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	602 Friedel pairs
$w = 1/[\sigma^2(F)^2 + (0.0706P)^2 + 0.41P]$	Flack parameter = $-0.17 (17)$

Table 1
Selected geometric parameters (Å, °).

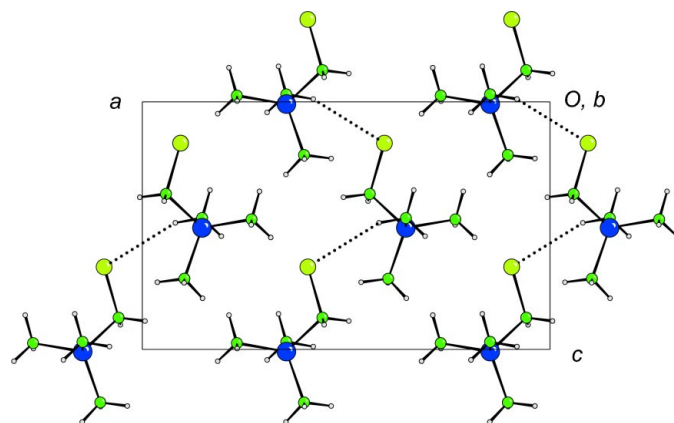|			
Cl1—C1	1.798 (5)	Si1—C3	1.862 (4)
Si1—C1	1.880 (4)	Si1—C4	1.867 (4)
Si1—C2	1.848 (4)		
C1—Si1—C2	109.4 (2)	C2—Si1—C4	111.2 (2)
C1—Si1—C3	109.47 (19)	C3—Si1—C4	111.18 (19)
C1—Si1—C4	105.5 (2)	Cl1—C1—Si1	111.9 (2)
C2—Si1—C3	110.01 (19)		

Figure 2
The molecular packing of (I), viewed along the *b* axis. Weak intermolecular Cl1···H33 interactions are shown as dotted lines.

The positions of the H atoms were recalculated geometrically after each refinement cycle, using a C—H distance of 1.00 Å, and they were assigned $U_{iso}(H) = 1.2U_{eq}(C)$. The 10,1,0 reflection was omitted as an outlier.

Data collection: *DIF*4 (Stoe & Cie, 1990); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1990); program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1996); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996) and *XP* (Sheldrick, 1997); software used to prepare material for publication: *CRYSTALS*, *enCIFer* (CCDC, 2003) and *PLATON* (Spek, 2003) used within *WinGX* (Farrugia, 1999).

References

Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, C. K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., García-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1996). *The DIRDIF96 Program System*. Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

Boese, R. & Nussbaumer, M. (1994). *Correlations, Transformations and Interactions in Organic Crystal Chemistry*, IUCr Crystallographic Symposia, Vol. 7, edited by D. W. Jones & A. Katrusiak, pp. 20–37. Oxford University Press.

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.

CCDC (2003). *enCIFer*. Test version. Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, England.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Sheldrick, G. M. (1997). *XP*, *XPREP* and *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Stoe & Cie (1990). *DIF*4 (Version 7.09/DOS) and *REDU*4 (Version 7.03/DOS). Stoe & Cie, Darmstadt, Germany.

Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.

supporting information

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(Chloromethyl)trimethylsilane

Crystal data

$C_4H_{11}ClSi$	$D_x = 1.095$ Mg m^{-3}
$M_r = 122.67$	Melting point: 182.8 K
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2c -2n	Cell parameters from 72 reflections
$a = 13.8776$ (13) Å	$\theta = 15–16°$
$b = 6.3855$ (9) Å	$\mu = 0.56$ mm^{-1}
$c = 8.400$ (1) Å	$T = 160$ K
$V = 744.37$ (15) Å^3	Cylinder, colourless
$Z = 4$	$0.50 \times 0.39 \times 0.39 \times 0.39$ (radius) mm
$F(000) = 264$	

Data collection

Stoe STADI-4 diffractometer equipped with an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986)	3768 measured reflections
	1306 independent reflections
	1186 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.030$
$\omega–\theta$ scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
Absorption correction: ψ scan	$h = -1 \rightarrow 16$
Azimuthal absorption correction (North et al., 1968) applied using XPREP (Sheldrick, 1997)	$k = -7 \rightarrow 7$
	$l = -9 \rightarrow 9$
	3 standard reflections every 0 reflections
$T_{min} = 0.741$, $T_{max} = 0.804$	intensity decay: 0.0%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F^*) + (0.0706P)^2 + 0.41P]$ where $P = 0.333*\max(F_o^2,0) + (1-0.333)F_c^2$ (SHELXL97; Sheldrick, 1997)
$wR(F^2) = 0.115$	
$S = 1.02$	
1305 reflections	$(\Delta/\sigma)_{max} = 0.001$
56 parameters	$\Delta\rho_{max} = 0.43$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.32$ e Å^{-3}
Primary atom site location: Patterson	Absolute structure: Flack (1983), 602 Friedel pairs
Secondary atom site location: difference Fourier map	Absolute structure parameter: −0.17 (17)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
Cl1	0.09384 (8)	0.28224 (18)	−0.33332 (15)	0.0602
Si1	0.14648 (6)	0.17201 (14)	0.00888 (17)	0.0327
C1	0.0582 (3)	0.3037 (6)	−0.1281 (5)	0.0430
C2	0.2681 (3)	0.2813 (6)	−0.0252 (5)	0.0492
C3	0.1468 (3)	−0.1149 (6)	−0.0303 (5)	0.0464
C4	0.1032 (3)	0.2305 (7)	0.2146 (5)	0.0522
H11	0.0538	0.4553	−0.0993	0.0516*
H12	−0.0064	0.2366	−0.1147	0.0516*
H21	0.3151	0.2113	0.0476	0.0591*
H22	0.2673	0.4351	−0.0034	0.0591*
H23	0.2874	0.2561	−0.1382	0.0591*
H31	0.1937	−0.1849	0.0426	0.0557*
H32	0.1659	−0.1411	−0.1433	0.0557*
H33	0.0808	−0.1727	−0.0112	0.0557*
H41	0.1477	0.1644	0.2936	0.0629*
H42	0.1024	0.3857	0.2311	0.0629*
H43	0.0368	0.1733	0.2286	0.0629*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0550 (6)	0.0864 (8)	0.0393 (6)	0.0021 (6)	−0.0026 (6)	0.0070 (7)
Si1	0.0317 (4)	0.0313 (4)	0.0350 (5)	0.0017 (4)	0.0013 (5)	−0.0014 (5)
C1	0.041 (2)	0.044 (2)	0.044 (2)	0.0084 (17)	−0.0020 (17)	0.0021 (19)
C2	0.039 (2)	0.050 (2)	0.059 (3)	−0.0074 (17)	0.001 (2)	−0.001 (2)
C3	0.048 (2)	0.0382 (18)	0.053 (3)	0.0006 (17)	0.0011 (19)	−0.0025 (17)
C4	0.060 (3)	0.058 (3)	0.039 (2)	0.006 (2)	0.0051 (19)	−0.0053 (18)

Geometric parameters (Å, º)

Cl1—C1	1.798 (5)	C2—H22	0.999
Si1—C1	1.880 (4)	C2—H21	1.000
Si1—C2	1.848 (4)	C3—H33	1.000
Si1—C3	1.862 (4)	C3—H32	1.000
Si1—C4	1.867 (4)	C3—H31	1.000
C1—H12	1.000	C4—H43	0.998
C1—H11	1.000	C4—H42	1.000
C2—H23	1.000	C4—H41	0.999
C1—Si1—C2	109.4 (2)	H23—C2—Si1	109.391
C1—Si1—C3	109.47 (19)	H22—C2—Si1	109.438
C1—Si1—C4	105.5 (2)	H21—C2—Si1	109.407
C2—Si1—C3	110.01 (19)	H33—C3—H32	109.483
C2—Si1—C4	111.2 (2)	H33—C3—H31	109.471
C3—Si1—C4	111.18 (19)	H32—C3—H31	109.508

H12—C1—H11	109.474	H33—C3—Si1	109.441
H12—C1—Cl1	108.829	H32—C3—Si1	109.458
H11—C1—Cl1	108.829	H31—C3—Si1	109.467
H12—C1—Si1	108.878	H43—C4—H42	109.610
H11—C1—Si1	108.891	H43—C4—H41	109.690
Cl1—C1—Si1	111.9 (2)	H42—C4—H41	109.489
H23—C2—H22	109.553	H43—C4—Si1	109.412
H23—C2—H21	109.488	H42—C4—Si1	109.281
H22—C2—H21	109.551	H41—C4—Si1	109.344