Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

tert-Butylmethylsilanediol

Theresa I. Kückmann, Hans-Wolfram Lerner and Michael Bolte*

Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.033
wR factor = 0.097
Data-to-parameter ratio = 28.5

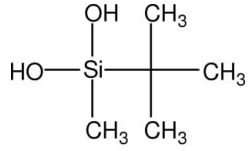
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 17 July 2006
Accepted 18 July 2006

The geometric parameters of the title compound, $C_5H_{14}O_2Si$, are unexceptional. The H atoms of both hydroxyl groups are disordered over two sites. In the crystal structure, zigzag chains are stabilized by O—H···O hydrogen bonds.

Comment

Previously, we reported on the properties and the syntheses of ^{t}Bu-substituted silicones and disiloxanes ^{t}Bu$_2$–Si*X*–O–Si*Y*–^{t}Bu$_2$ (*X* = *Y* = H and OH), which are accessible from the reaction of CF_3SO_2Cl with ^{t}Bu$_2$SiHOH or ^{t}Bu$_2$Si(OH)$_2$ (Lerner *et al.*, 2005). X-ray structure determination of ^{t}Bu$_2$Si(OH)$_2$, the starting material of these condensation reactions, shows a reversible phase transition at 211 K (Bats *et al.*, 2002). Surprisingly, we have obtained the silanediol ^{t}BuMeSi(OH)$_2$, (I), as a by-product of the reaction of $MeHSiCl_2$ and ^{t}BuLi. In an earlier report the hydrolysis reaction of ^{t}BuMeSiCl$_2$ was described, which produced the silanediol ^{t}BuMeSi(OH)$_2$ in a yield of 78% (Sommer & Tyler, 1954). We obtained single crystals of ^{t}BuMeSi(OH)$_2$ by slow evaporation of the reaction solution at ambient temperature.



(I)

The molecular structure of the title compound is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.27, November 2005 updated May 2006; Mogul, Version 1.1; Allen, 2002). Geometric parameters are unexceptional. The H atoms of both hydroxyl groups are disordered over two sites. In the crystal structure (Fig. 2), molecules are connected into zigzag chains stabilized by O—H···O hydrogen bonds.

Experimental

A solution of ^{t}BuLi in pentane (50 ml, 84 mmol) was added dropwise to a solution of $MeHSiCl_2$ (4.17 ml, 40 mmol) in pentane (10 ml) at 273 K. After complete addition of the lithium reagent, the mixture was allowed to warm to room temperature and stirred overnight. The reaction mixture was quenched with ammonium chloride in ice–water. The organic phase was washed with water (3 × 15 ml) and dried over $MgSO_4$. Slow evaporation of the solvent led to the deposition of a small quantity of colourless needles (10 mg, 0.08 mmol, 0.2%). ^{1}H NMR ($CDCl_3$): δ 0.970 (*s*, 9 H, ^{t}Bu), 0.166 (*s*, 3 H, Me), not observed (n.o.) (OH). ^{13}C NMR ($CDCl_3$): δ 25.6 [C(CH_3)$_3$], −5.3 ($SiCH_3$), n.o. [C(CH$_3$)$_3$]. ^{29}Si NMR (C_6D_6): δ −3.3.

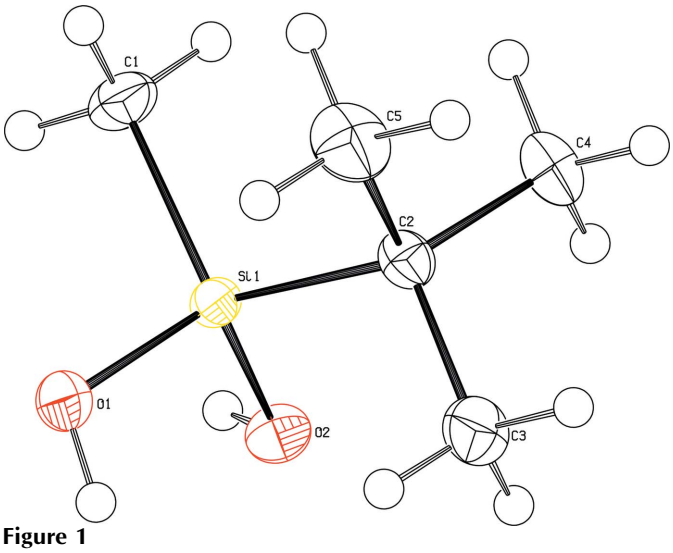


Figure 1
Molecular structure of the title compound with the atom numbering scheme; displacement ellipsoids are at the 50% probability level; H atoms are drawn as small spheres of arbitrary radii. Only one disorder component is shown for the hydroxyl groups.

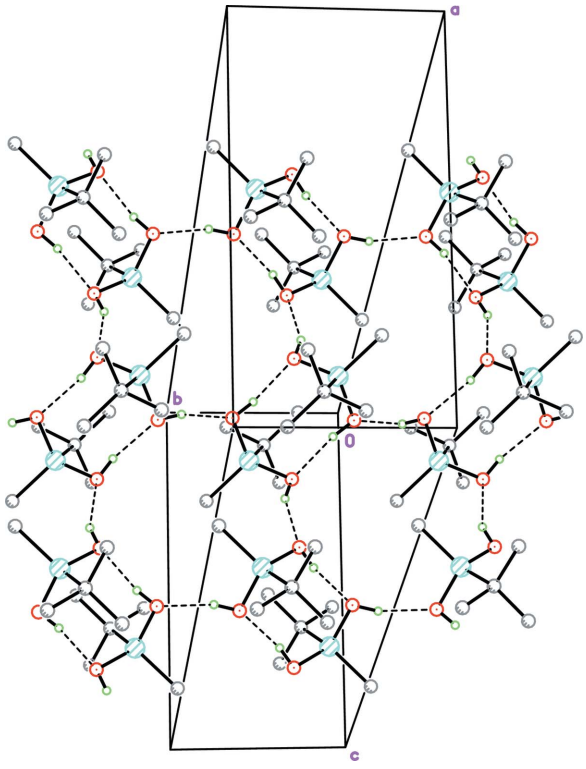


Figure 2
Packing diagram of the title compound. Only one disorder component is shown for the hydroxyl groups. Hydrogen bonds are shown as dashed lines, and H atoms not involved in these interactions have been omitted.

Crystal data

$C_5H_{14}O_2Si$
M_r = 134.25
Monoclinic, $C2/c$
a = 22.765 (2) Å
b = 6.1372 (4) Å
c = 11.8688 (12) Å
β = 109.805 (7)°
V = 1560.1 (2) Å^3
Z = 8
D_x = 1.143 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 173 (2) K
Needle, colourless
0.48 × 0.23 × 0.13 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.890, T_{max} = 0.971
19678 measured reflections
2169 independent reflections
1990 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 29.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.097
S = 1.07
2169 reflections
76 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 1.3702P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0090 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.84	1.96	2.7903 (13)	168
O1—H1′···O1^{ii}	0.84	1.91	2.7304 (18)	164
O2—H2···O2^{iii}	0.84	1.97	2.7444 (18)	152
O2—H2′···O1^{i}	0.84	1.96	2.7903 (13)	168

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y+2, -z+1$; (iii) $-x+1, y, -z+\frac{3}{2}$.

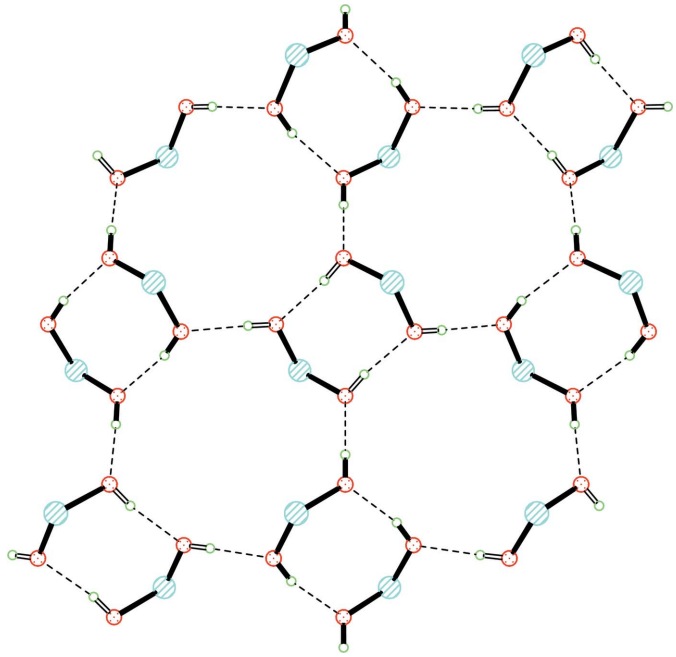


Figure 3
Partial packing diagram viewed approximately perpendicular to (100). C atoms and H atoms bonded to C atoms have been omitted for clarity. The unprimed H atoms are shown with full bonds and the primed H atoms are connected by open bonds. The figure shows two zigzag chains of hydrogen bonds (dashed lines) in which two primed H atoms and two unprimed H atoms alternate. The directional sense of the H bonds was chosen arbitrarily. Since none of the chains is in contact with any other chain, the sense of each chain is independent.

H atoms were located in a difference map, but were subsequently refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(O) or U_{iso}(H) = 1.5U_{eq}(C)] in the riding-model approximation with O—H = 0.84 Å and C—H = 0.98 Å. The hydroxyl groups were allowed to rotate but not tip. The disorder of the hydroxyl H atoms is correlated, because they would be too close to their symmetry equivalents. Therefore, all occupancies were set to 0.5.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Bats, J. W., Scholz, S. & Lerner, H.-W. (2002). *Acta Cryst.* C**58**, o439–o441.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Lerner, H.-W., Scholz, S., Wiberg, N., Polborn, K., Bolte, M. & Wagner, M. (2005). *Z. Anorg. Allg. Chem.* **76**, 1863–1870.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sommer, L. H. & Tyler, L. J. (1954). *J. Am. Chem. Soc.* **76**, 1030–1033.

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

Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.