Journal of Organometallic Chemistry, 480 (1994) 139–143

Crystal structure of the crowded silanol $(Me_3Si)_3CSiPh_2OH$

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(Received November 22, 1993)

Abstract

The unit cell in a crystal of $(Me_3Si)_3CSiPh_2OH$ contains two dimers, one of them centrosymmetric. There are no significant differences between the geometries of the molecules in the two types of dimers, in which the components appear to be held together by hydrogen bonding of the type Si—(H)O ···· H—O—Si.

Key words: Silicon; Crystal structure; Hydrogen bonding; silanol

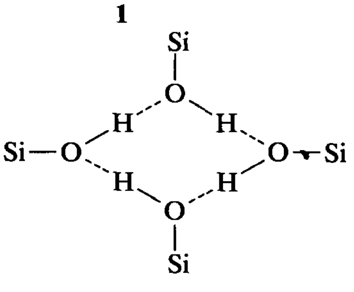
1. Introduction

There is much current interest in the structures of organosilanols, especially in hydrogen bonding aspects [1–8]. In several cases the presence of the bulky ligand $(Me_3Si)_3C$ or $(PhMe_2Si)_3C$ on a silicon atom bearing one or more OH groups has been shown to give rise to unusual structures [3–7]. Of particular interest are OH...π interactions between an aryl group and an Si–OH unit, either intramolecular, as in $HO(SiPh_2)_7OH$ [8] and $(PhMe_2Si)_3CSi(Me)(H)OH$ [5], or intermolecular, as in $(Me_3Si)_3CSi(Ph)(X)OH$ with X = I or OMe [4]. We thus thought it of interest to examine the structure of the diphenyl compound $(Me_3Si)_3CSiPh_2OH$, **1**; this might reasonably have been expected also to show OH...π bonding, but in the event it was found not to do so.

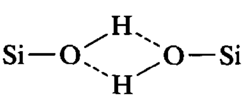
The crystal structure of the related silanol Ph_3SiOH is known [8]. It involves cyclic tetramers with the skeleton **I**.

$(Me_3Si)_3CSiPh_2OH$ **1**

Si—(H)O ···· H—O—Si **II**



I



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2. Results and discussion

The unit cell of **1** was found to contain two separate, though closely similar dimers, one centrosymmetric (see Fig. 1) and the other containing two distinct molecules (see Fig. 2). Unfortunately the hydrogen atoms could not be located, but the positions of the oxygen atoms ruled out the presence of any HO ··· π interactions, instead indicating that the molecules in the dimer are held together only by O—H ··· OH interactions, with an O ··· O separation of 2.85(2) Å. If the individual molecules in the centrosymmetric dimer are identical then the hydrogen bonding cannot be of the asymmetric type Si–(H)O ··· H–O–Si, **II**, and this raises the possibilty that there could be symmetrical bonding of type **III**; such bonding was at one time thought to be present in crystals of the diols $R_2Si(OH)_2$ with R = Et [9], but these were later shown to involve simple interactions of type **II** [10].) It is more likely, however, that the apparent centrosymmetry arises from the randomly distributed presence of some dimers with O(3) and others with O(3′) providing the H atom of the hydrogen bond.

Although the other dimer, in which the O ··· O distance is 2.90(1) Å, is non-centrosymmetric there are no significant differences in geometry between the two component molecules, or, indeed, between their geometries and that of the molecules in the other dimer (see Table 1), and there is no reason to suspect that the hydrogen bonding is in any way different in the two types of dimer.

Because of the similarity between the geometries of the various molecules in the dimers, only that in the centrosymmetric species will be discussed. The main

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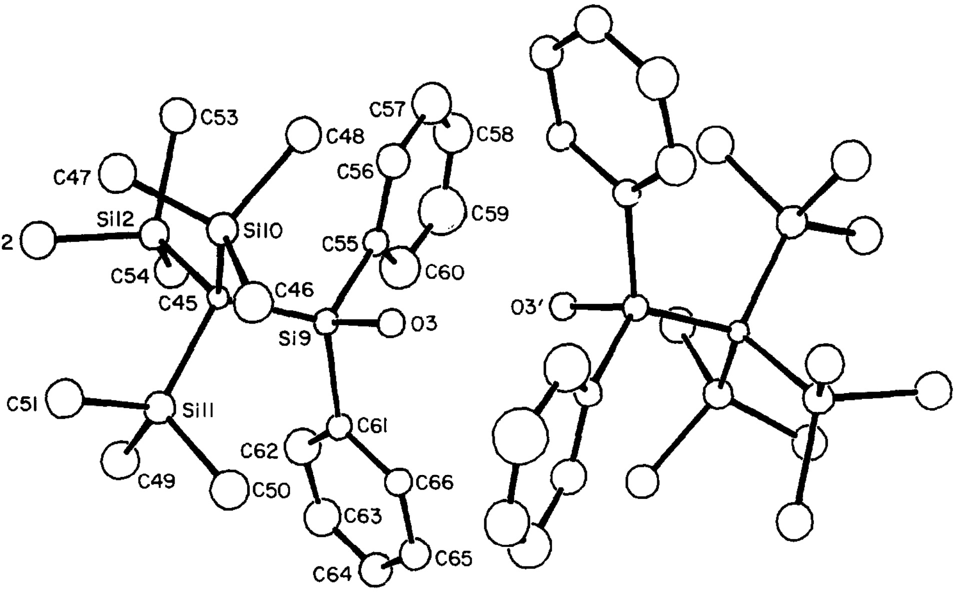

Fig. 1.

feature is that in order to minimize steric interactions the Ph groups move away from the $(Me_3Si)_3C$ group, to give values of 119.7(9)° and 117.0(9)° for the C(45)-Si-Ph angles and one of 104.8(9)° for the Ph-Si-Ph angle. The $(Me_3Si)_3C$-Si-O angle, 106.2(7)°, and the Ph-Si-O(3) angles, 104.7(7)° and 106.2(7)°, are correspondingly small. The Si(9)-O(3) bond distance of 1.678(11) Å is possibly slightly longer than is usual in silanols [1].

On the above interpretation only half of the hydrogen atoms of the OH groups are engaged in hydrogen bonding. Probably intermolecular repulsion between

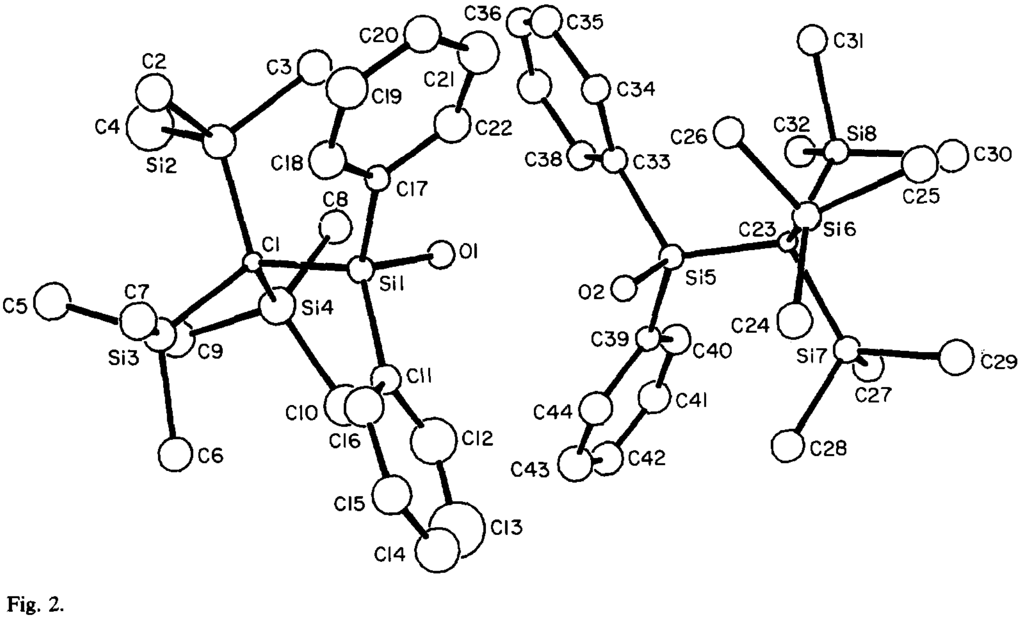

Fig. 2.

the substituents on the Si atom prevents formation of a tetramer such as that formed by Ph_3SiOH or of discrete molecules with intramolecular OH $\cdots$ π bond-

TABLE 1. Intramolecular distances(A) and angles(°) with estimated standard deviations in parentheses

(a) *Bonds*			
Si(1)–O(1)	1.702(13)	Si(1)–C(1)	1.92(2)
Si(1)–C(11)	1.86(2)	Si(1)–C(17)	1.83(2)
Si(2)–C(1)	1.90(2)	Si(2)–C(2)	1.87(3)
Si(2)–C(3)	1.89(2)	Si(2)–C(4)	1.88(3)
Si(3)–C(1)	1.92(2)	Si(3)–C(5)	1.88(2)
Si(3)–C(6)	1.86(2)	Si(3)–C(7)	1.88(3)
Si(4)–C(1)	1.96(2)	Si(4)–C(8)	1.92(2)
Si(4)–C(9)	1.95(2)	Si(4)–C(10)	1.92(2)
Si(5)–O(2)	1.653(14)	Si(5)–C(23)	1.89(2)
Si(5)–C(33)	1.87(2)	Si(5)–C(39)	1.87(2)
Si(6)–C(23)	1.93(2)	Si(6)–C(24)	1.85(2)
Si(6)–C(25)	1.88(2)	Si(6)–C(26)	1.86(2)
Si(7)–C(23)	1.95(2)	Si(7)–C(27)	1.87(2)
Si(7)–C(28)	1.85(2)	Si(7)–C(29)	1.88(2)
Si(8)–C(23)	1.93(2)	Si(8)–C(30)	1.87(2)
Si(8)–C(31)	1.88(2)	Si(8)–C(32)	1.84(2)
Si(9)–O(3)	1.678(11)	Si(9)–C(45)	1.89(2)
Si(9)–C(55)	1.87(2)	Si(9)–C(61)	1.89(2)
Si(10)–C(45)	1.89(2)	Si(10)–C(46)	1.90(2)
Si(10)–C(47)	1.85(2)	Si(10)–C(48)	1.86(3)
Si(11)–C(45)	1.93(2)	Si(11)–C(49)	1.83(2)
Si(11)–C(50)	1.85(2)	Si(11)–C(51)	1.88(2)
Si(12)–C(45)	1.95(2)	Si(12)–C(52)	1.89(2)
Si(12)–C(53)	1.88(3)	Si(12)–C(54)	1.89(2)
C(11)–C(12)	1.36(3)	C(11)–C(16)	1.37(3)
C(12)–C(13)	1.47(4)	C(13)–C(14)	1.31(5)
C(14)–C(15)	1.27(4)	C(15)–C(16)	1.43(3)
C(17)–C(18)	1.38(3)	C(17)–C(22)	1.37(3)
C(18)–C(19)	1.38(4)	C(19)–C(20)	1.32(3)
C(20)–C(21)	1.34(3)	C(21)–C(22)	1.40(4)
C(33)–C(34)	1.40(3)	C(33)–C(38)	1.40(3)
C(34)–C(35)	1.40(3)	C(35)–C(36)	1.30(3)
C(36)–C(37)	1.35(3)	C(37)–C(38)	1.44(3)
C(39)–C(40)	1.39(3)	C(39)–C(44)	1.45(3)
C(40)–C(41)	1.39(4)	C(41)–C(42)	1.34(3)
C(42)–C(43)	1.29(4)	C(43)–C(44)	1.43(4)
C(55)–C(56)	1.34(3)	C(55)–C(60)	1.38(3)
C(56)–C(57)	1.42(4)	C(57)–C(58)	1.31(4)
C(58)–C(59)	1.34(5)	C(59)–C(60)	1.47(5)
C(61)–C(62)	1.38(3)	C(61)–C(66)	1.40(3)
C(62)–C(63)	1.40(4)	C(63)–C(64)	1.33(3)
C(64)–C(65)	1.32(3)	C(65)–C(66)	1.40(3)
O(1)...O(2)	2.899(14)	O(3)...O(3)'	2.853(16)
(b) *Angles*			
O(1)–Si(1)–C(1)	107.1(7)	O(1)–Si(1)–C(11)	104.9(7)
O(1)–Si(1)–C(17)	106.0(8)	C(1)–Si(1)–C(11)	116.8(8)
C(1)–Si(1)–C(17)	114.8(7)	C(11)–Si(1)–C(17)	106.4(9)
C(1)–Si(2)–C(2)	113.8(9)	C(1)–Si(2)–C(3)	112.2(8)
C(1)–Si(2)–C(4)	113(1)	C(2)–Si(2)–C(3)	107(1)
C(2)–Si(2)–C(4)	105(1)	C(3)–Si(2)–C(4)	105(1)
C(1)–Si(3)–C(5)	111.2(9)	C(1)–Si(3)–C(6)	113.0(9)
C(1)–Si(3)–C(7)	114(1)	C(5)–Si(3)–C(6)	108(1)
C(5)–Si(3)–C(7)	102(1)	C(6)–Si(3)–C(7)	107(1)
C(1)–Si(4)–C(8)	114(1)	C(1)–Si(4)–C(9)	112.2(9)
C(1)–Si(4)–C(10)	115(1)	C(8)–Si(4)–C(9)	108(1)
C(8)–Si(4)–C(10)	104(1)	C(9)–Si(4)–C(10)	104(1)
O(2)–Si(5)–C(23)	105.7(7)	O(2)–Si(5)–C(33)	105.8(7)
O(2)–Si(5)–C(39)	108.0(8)	C(23)–Si(5)–C(33)	116.5(8)
C(23)–Si(5)–C(39)	115.6(8)	C(33)–Si(5)–C(39)	104.7(9)
C(23)–Si(6)–C(24)	113(1)	C(23)–Si(6)–C(25)	113.9(9)
C(23)–Si(6)–C(26)	115.4(8)	C(24)–Si(6)–C(25)	107(1)
C(24)–Si(6)–C(26)	104.1(9)	C(25)–Si(6)–C(26)	103(1)
C(23)–Si(7)–C(27)	115.0(8)	C(23)–Si(7)–C(28)	112.5(8)
C(23)–Si(7)–C(29)	113.2(9)	C(27)–Si(7)–C(28)	106(1)
C(27)–Si(7)–C(29)	104(1)	C(28)–Si(7)–C(29)	105(1)
C(23)–Si(8)–C(30)	113.5(8)	C(23)–Si(8)–C(31)	112.7(8)
C(23)–Si(8)–C(32)	115.2(9)	C(30)–Si(8)–C(31)	105.4(9)
C(30)–Si(8)–C(32)	105(1)	C(31)–Si(8)–C(32)	104.3(9)
O(3)–Si(9)–C(45)	104.7(7)	O(3)–Si(9)–C(55)	102.8(7)
O(3)–Si(9)–C(61)	106.2(7)	C(45)–Si(9)–C(55)	119.7(9)
C(45)–Si(9)–C(61)	117.0(9)	C(55)–Si(9)–C(61)	104.8(9)
C(45)–Si(10)–C(46)	112(1)	C(45)–Si(10)–C(47)	114.4(9)
C(45)–Si(10)–C(48)	116.7(9)	C(46)–Si(10)–C(47)	105(1)
C(46)–Si(10)–C(48)	103(1)	C(47)–Si(10)–C(48)	104(1)
C(45)–Si(11)–C(49)	115(1)	C(45)–Si(11)–C(50)	112(1)
C(45)–Si(11)–C(51)	114(1)	C(49)–Si(11)–C(50)	105(1)
C(49)–Si(11)–C(51)	107(1)	C(50)–Si(11)–C(51)	103(1)
C(45)–Si(12)–C(52)	113.8(9)	C(45)–Si(12)–C(53)	113.8(9)
C(45)–Si(12)–C(54)	114.9(9)	C(52)–Si(12)–C(53)	104(1)
C(52)–Si(12)–C(54)	105(1)	C(53)–Si(12)–C(54)	104(1)
Si(1)–C(1)–Si(2)	110.3(9)	Si(1)–C(1)–Si(3)	112.8(9)
Si(1)–C(1)–Si(4)	107.1(8)	Si(2)–C(1)–Si(3)	111.7(8)
Si(2)–C(1)–Si(4)	107.1(9)	Si(3)–C(1)–Si(4)	107.6(9)
Si(1)–C(11)–C(12)	120(2)	Si(1)–C(11)–C(16)	126(2)
C(12)–C(11)–C(16)	114(2)	C(11)–C(12)–C(13)	122(3)
C(12)–C(13)–C(14)	119(3)	C(13)–C(14)–C(15)	122(3)
C(14)–C(15)–C(16)	121(2)	C(11)–C(16)–C(15)	123(2)
Si(1)–C(17)–C(18)	129(1)	Si(1)–C(17)–C(22)	119(2)
C(18)–C(17)–C(22)	112(2)	C(17)–C(18)–C(19)	125(2)
C(18)–C(19)–C(20)	118(2)	C(19)–C(20)–C(21)	122(2)
C(20)–C(21)–C(22)	118(2)	C(17)–C(22)–C(21)	125(2)
Si(5)–C(23)–Si(6)	110.6(8)	Si(5)–C(23)–Si(7)	109.5(9)
Si(5)–C(23)–Si(8)	111.9(8)	Si(6)–C(23)–Si(7)	105.5(8)
Si(6)–C(23)–Si(8)	109.9(9)	Si(7)–C(23)–Si(8)	109.3(8)
Si(5)–C(33)–C(34)	123(2)	Si(5)–C(33)–C(38)	123(2)
C(34)–C(33)–C(38)	114(2)	C(33)–C(34)–C(35)	124(2)
C(34)–C(35)–C(36)	119(2)	C(35)–C(36)–C(37)	124(2)
C(36)–C(37)–C(38)	118(2)	C(33)–C(38)–C(37)	121(2)
Si(5)–C(39)–C(40)	128(1)	Si(5)–C(39)–C(44)	118(2)
C(40)–C(39)–C(44)	113(2)	C(39)–C(40)–C(41)	124(2)
C(40)–C(41)–C(42)	120(2)	C(41)–C(42)–C(43)	120(3)
C(42)–C(43)–C(44)	123(2)	C(39)–C(44)–C(43)	119(2)
Si(9)–C(45)–Si(10)	111.8(9)	Si(9)–C(45)–Si(11)	109(1)
Si(9)–C(45)–Si(12)	110.7(8)	Si(10)–C(45)–Si(11)	107.9(8)
Si(10)–C(45)–Si(12)	109(1)	Si(11)–C(45)–Si(12)	108.3(8)
Si(9)–C(55)–C(56)	124(1)	Si(9)–C(55)–C(60)	120(2)
C(56)–C(55)–C(60)	116(2)	C(55)–C(56)–C(57)	126(2)
C(56)–C(57)–C(58)	116(3)	C(57)–C(58)–C(59)	124(3)
C(58)–C(59)–C(60)	118(3)	C(55)–C(60)–C(59)	120(3)
Si(9)–C(61)–C(62)	125(2)	Si(9)–C(61)–C(66)	118(1)
C(62)–C(61)–C(66)	117(2)	C(61)–C(62)–C(63)	121(2)
C(62)–C(63)–C(64)	120(2)	C(63)–C(64)–C(65)	120(2)
C(64)–C(65)–C(66)	123(2)	C(61)–C(66)–C(65)	119(2)
Si(1)–O(1)...O(2)	126.7(7)	Si(5)–O(2)...O(1)	110.1(6)
Si(9)–O(3)...O(3) [a]	113.6(6)		

[a] The symmetry element is: $-x, 2-y, -z$.

ing, such as those formed by $(Me_3Si)_3SCi(Ph)(X)OH$ with X = I or OMe, in all of which all the available hydroxyl hydrogen atoms are used.

TABLE 2. Fractional atomic coordinates and equivalent isotropic thermal parameters

Atom	x	y	z	U_{eq}
Si(1)	−0.1084(5)	0.7684(4)	0.6804(2)	0.040(2)
Si(2)	−0.1866(6)	0.9227(5)	0.5676(3)	0.066(2)
Si(3)	−0.3702(6)	0.8904(5)	0.6703(3)	0.062(2)
Si(4)	−0.2809(7)	0.7333(5)	0.5988(3)	0.085(3)
Si(5)	0.2032(5)	0.4467(4)	0.6658(2)	0.038(2)
Si(6)	0.4048(5)	0.4480(4)	0.7439(3)	0.047(2)
Si(7)	0.3301(5)	0.2564(4)	0.7474(2)	0.044(2)
Si(8)	0.4640(5)	0.3529(4)	0.6366(2)	0.040(2)
Si(9)	0.1468(5)	0.8368(4)	0.0043(2)	0.040(2)
Si(10)	0.3369(6)	0.9056(5)	−0.0850(3)	0.057(2)
Si(11)	0.2436(6)	0.7263(5)	−0.0919(3)	0.061(2)
Si(12)	0.4057(6)	0.7162(4)	0.0088(3)	0.056(2)
O(1)	−0.0326(10)	0.6744(8)	0.6531(5)	0.043(4)
O(2)	0.1450(10)	0.5165(8)	0.7103(5)	0.039(4)
O(3)	0.0831(11)	0.9461(8)	−0.0331(5)	0.045(4)
C(1)	−0.2359(15)	0.8321(11)	0.6305(7)	0.022(5)
C(2)	−0.1882(20)	1.0419(16)	0.5842(10)	0.076(8)
C(3)	−0.0335(19)	0.8775(15)	0.5412(9)	0.070(8)
C(4)	−0.2797(28)	0.9508(21)	0.5030(13)	0.143(13)
C(5)	−0.4752(20)	0.9793(16)	0.6187(10)	0.078(8)
C(6)	−0.4500(19)	0.8010(15)	0.7154(9)	0.067(7)
C(7)	−0.3373(20)	0.9670(15)	0.7178(9)	0.071(8)
C(8)	−0.1777(20)	0.6953(15)	0.5385(9)	0.071(8)
C(9)	−0.4384(21)	0.7745(16)	0.5690(10)	0.084(9)
C(10)	−0.2581(24)	0.6137(18)	0.6530(11)	0.106(10)
C(11)	−0.1465(17)	0.7133(13)	0.7560(8)	0.050(7)
C(12)	−0.1547(25)	0.6188(19)	0.7712(12)	0.122(11)
C(13)	−0.1838(32)	0.5725(25)	0.8303(15)	0.174(14)
C(14)	−0.1909(23)	0.6210(18)	0.8707(11)	0.103(10)
C(15)	−0.1834(20)	0.7089(16)	0.8590(10)	0.079(8)
C(16)	−0.1614(21)	0.7575(16)	0.8012(10)	0.081(8)
C(17)	−0.0032(16)	0.8429(12)	0.6843(7)	0.034(6)
C(18)	−0.0204(19)	0.9258(15)	0.7050(9)	0.072(8)
C(19)	0.0649(21)	0.9758(16)	0.7093(10)	0.086(9)
C(20)	0.1726(19)	0.9410(15)	0.6929(9)	0.067(7)
C(21)	0.2011(21)	0.8573(17)	0.6757(10)	0.089(9)
C(22)	0.1116(20)	0.8104(15)	0.6714(9)	0.073(8)
C(23)	0.3490(15)	0.3784(11)	0.6964(7)	0.023(5)
C(24)	0.3265(19)	0.4401(15)	0.8147(9)	0.068(8)
C(25)	0.5652(20)	0.4076(15)	0.7580(9)	0.073(8)
C(26)	0.3892(18)	0.5796(14)	0.7139(8)	0.051(7)
C(27)	0.3249(19)	0.1580(15)	0.7104(9)	0.064(7)
C(28)	0.1964(19)	0.2694(15)	0.7932(9)	0.065(7)
C(29)	0.4528(20)	0.2019(15)	0.7985(9)	0.073(8)
C(30)	0.5917(19)	0.2550(15)	0.6629(9)	0.065(7)
C(31)	0.5272(18)	0.4609(14)	0.6014(8)	0.053(7)
C(32)	0.4101(19)	0.3171(15)	0.5764(9)	0.064(7)
C(33)	0.2069(17)	0.5295(13)	0.5933(8)	0.044(6)
C(34)	0.2503(18)	0.6140(14)	0.5837(8)	0.052(7)
C(35)	0.2422(19)	0.6824(15)	0.5314(9)	0.065(7)
C(36)	0.1989(18)	0.6644(14)	0.4879(8)	0.057(7)
C(37)	0.1533(20)	0.5859(16)	0.4909(9)	0.078(8)
C(38)	0.1568(18)	0.5167(14)	0.5452(8)	0.053(7)
C(39)	0.0977(16)	0.3709(12)	0.6595(8)	0.039(6)
C(40)	0.1110(18)	0.2993(14)	0.6286(9)	0.061(7)
C(41)	0.0256(20)	0.2477(15)	0.6259(9)	0.074(8)
C(42)	−0.0757(20)	0.2642(15)	0.6558(9)	0.075(8)
C(43)	−0.0973(21)	0.3315(16)	0.6841(10)	0.083(8)
C(44)	−0.0130(19)	0.3849(15)	0.6908(9)	0.071(8)
C(45)	0.2814(15)	0.7985(12)	−0.0402(7)	0.029(5)
C(46)	0.2457(21)	0.9670(16)	−0.1505(10)	0.084(8)
C(47)	0.4879(21)	0.8764(16)	−0.1136(10)	0.081(8)
C(48)	0.3383(19)	1.0057(15)	−0.0494(9)	0.071(8)
C(49)	0.2338(22)	0.6014(17)	−0.0590(10)	0.091(9)
C(50)	0.1014(22)	0.7825(17)	−0.1254(10)	0.088(9)
C(51)	0.3474(21)	0.7228(16)	−0.1558(10)	0.084(8)
C(52)	0.5207(21)	0.6400(16)	−0.0300(10)	0.081(8)
C(53)	0.4892(20)	0.7846(16)	0.0424(9)	0.078(8)
C(54)	0.3559(20)	0.6277(16)	0.0718(10)	0.080(8)
C(55)	0.1629(17)	0.8595(13)	0.0769(8)	0.043(6)
C(56)	0.2205(20)	0.9240(15)	0.0858(9)	0.072(8)
C(57)	0.2318(23)	0.9453(18)	0.1398(11)	0.106(10)
C(58)	0.1775(24)	0.8992(19)	0.1839(11)	0.107(10)
C(59)	0.1138(30)	0.8350(23)	0.1805(14)	0.162(14)
C(60)	0.1089(24)	0.8112(19)	0.1245(11)	0.116(11)
C(61)	0.0324(16)	0.7603(13)	0.0157(8)	0.042(6)
C(62)	0.0467(21)	0.6666(17)	0.0469(10)	0.088(9)
C(63)	−0.0472(21)	0.6190(17)	0.0589(10)	0.092(9)
C(64)	−0.1518(20)	0.6629(15)	0.0385(9)	0.073(8)
C(65)	−0.1693(19)	0.7534(15)	0.0092(9)	0.063(7)
C(66)	−0.0798(17)	0.8054(13)	−0.0039(8)	0.048(6)

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

3. Experimental details

The silanol 1 was made as described previously [11], but was purified by recrystallization from heptane rather than by sublimation; the yield was 87%. A crystal of dimensions $0.15 \times 0.15 \times 0.1$ mm was used for the data collection.

Crystal data: $C_{22}H_{38}OSi_4$, $M = 430.9$, triclinic, space group $P\bar{1}$, $a = 11.753(3)$ Å, $b = 14.577(6)$ Å, $c = 23.948(8)$ Å, $\alpha = 76.4(3)°$, $\beta = 85.47(2)°$, $\gamma = 77.41(3)°$, $U = 3890.3$ Å^3, $Z = 6$, $D_c = 1.10$ g cm^{-3}, $F(000) = 1.404$, Mo-Kα radiation, $\lambda = 0.71069$ Å, $\mu = 2.3$ cm^{-1}.

Data were collected at room temperature on an Enraf-Nonius CAD4 diffractometer. Intensities of 7217 unique reflections with $+h \pm k \pm l$ and $2 < \theta < 25°$ were measured by a 2θ scan. Corrections were made for Lorentz and polarization effects and for decay of 12% shown by standard reflections, but not for absorption; 2435 unique refections with $|F^2| < 2\sigma(F^2)$ were used in the refinement, where $\sigma(F^2) = [\sigma^2(I) + (0.04I)^2]^{1/2}/L_P$.

Non-hydrogen atoms were located by direct methods and refined by full-matrix least squares but, because of the limited data set, with isotropic temperature factors. Except for those of the OH groups, which could not be located and were omitted, the H atoms were placed in calculated positions with $U_{iso} = 1.3U_{eq}$ for the atoms to which they are attached. Three independent molecules were found to be present in the unit cell. Refinement converged at $R = 0.094$, $R' = 0.089$, $w = \sigma^{-2}(F)$; $s = 2.0$, $(\Delta p)_{max} = 0.63$, $(\Delta p)_{min} = -0.14$ e Å^{-3}.

The atomic coordinates of non-hydrogen atoms are listed in Table 2. Tables of H atom coordinates have been deposited at the Cambridge Crystallographic Data Centre.

Acknowledgments

We thank the SERC for support, The King Saud University, Riyadh, for granting sabbatical leave to A. Al-M., and the Ministry of Higher Education of the Kingdom of Saudi Arabia for the award of a studentship to S.S. Al-J.

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