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X-ray Crystal and Molecular Structures of Bis(2-methylhydrazino)tetraphenyldisiloxane: Linear at Oxygen, Planar *vs* Pyramidal at Nitrogen

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Summary: Bis(2-methylhydrazino)tetraphenyldisiloxane, 1, is prepared by the controlled hydrolysis of bis(2-methylhydrazino)diphenylsilane, 2. Crystals of 1 are monoclinic, space group $P2_1/n$, with a = 10.3200(19) Å, b = 7.3343(20) Å, c = 16.658(3) Å, β = 94.013(15)°, and Z = 2. The molecule is centrosymmetric, with a linear SiOSi skeleton and with the bridging O atom at the center of symmetry. The coordinations at N(1) and N(2), are found to be planar and pyramidal, respectively, in 1. The solution NMR properties of 1 (^1H, ^{29}Si, ^{15}N) are also reported. The structural characteristics of 1 are compared with other hydrazinosilanes.

Introduction

Structural investigations of compounds containing Si–N and Si–O bonds have been the subject of long and continuing interest due to the anomalous geometries on nitrogen or oxygen when compared with their carbon analogues.[1-4] The replacement of the carbon atoms in dimethyl ether (∠C—O—C = 112°)[5] with silicons (∠Si—O—Si = 144°)[6] produces dramatic changes in bond angles, and linear SiOSi geometry was found in the compounds $O(SiPh_3)_2$,[7] $O[Si(CH_2Ph)_3]_2$,[8] and $O[Si(CH{=}CH_2)_3]_2$.[9] Planar geometry about nitrogen in aminosilane compounds has been established for nearly 40 years, since the original report on the structure of $N(SiH_3)_3$,[10] although recent findings indicate that some aminosilanes, R_3SiNH_2, are pyramidal at nitrogen.[3] For several decades, the rationalization of these observations in terms of the role of silicon 3d orbitals was the subject of much debate.[2,3,11] Most recent computations show that d orbitals do not contribute significantly to bonding in organosilicon compounds.[4,12]

As part of a wider investigation of the synthesis and structures of hydrazinosilanes, we have prepared and characterized bis(2-methylhydrazino)tetraphenyldisiloxane, 1. In the present paper we discuss the structural characteristics of this compound and compare them to other hydrazinosilanes whose structures have been determined.

Results and Discussion

Compound 1 was prepared in good yield by controlled hydrolysis of bis(2-methylhydrazino)diphenylsilane, 2, which was synthesized by the catalytic dehydrocoupling reaction of diphenylsilane with methylhydrazine,[13] shown in (1).

$$\mathrm{Ph_2SiH_2} + 2\mathrm{MeNHNH_2} \xrightarrow[-2\mathrm{H_2}]{\mathrm{DMT/80\ ^\circ C}} \underset{\mathbf{2}}{\mathrm{Ph_2Si(NHNHMe)_2}} \xrightarrow[-\mathrm{MeHNNH_2}]{{}^1/_2\mathrm{H_2O}} \underset{\mathbf{1}}{{}^1/_2\mathrm{O[SiPh_2(NHNHMe)]_2}} \qquad (1)$$

DMT = dimethyltitanocene

Crystal Structure. The compounds $O[SiPh_2(NHNMe_2)]_2$ and $O[SiPh_2(NHNHPh)]_2$ were prepared 30 years ago by Wannagat and Niederprüm,[14] but no structural studies have been reported for this class of compounds. An ORTEP plot showing the molecular structure of 1 is presented in Figure 1. Important bond lengths and angles for 1 are given in Table 1.

The structure is centrosymmetric, with a linear SiOSi skeleton and with the bridging O atom at the center of symmetry. The Si–O distance 1.6131(13) Å is the same, within experimental uncertainty, as that in the compounds $O(SiPh_3)_2$ 1.616(1),[7] $O[Si(CH_2Ph)_3]_2$ 1.613(4),[8] and $O[Si(CH{=}CH_2)_3]_2$ 1.614(1).[9] The Si–C distances, mean value 1.870(5) Å, lie very close to those of $O(SiPh_3)_2$, *i.e.*, 1.866(2),[7] and Ph_4Si, 1.872(7).[15] The bond angles about the silicon atoms are all close to tetrahedral. The Si–N distance, 1.695(4) Å, and the N–N distance, 1.430(5) Å, are identical to those in compound 2,[13] but slightly shorter than those in other hydrazinosilanes (see Table 2).

[⊗] Abstract published in *Advance ACS Abstracts*, April 15, 1994.

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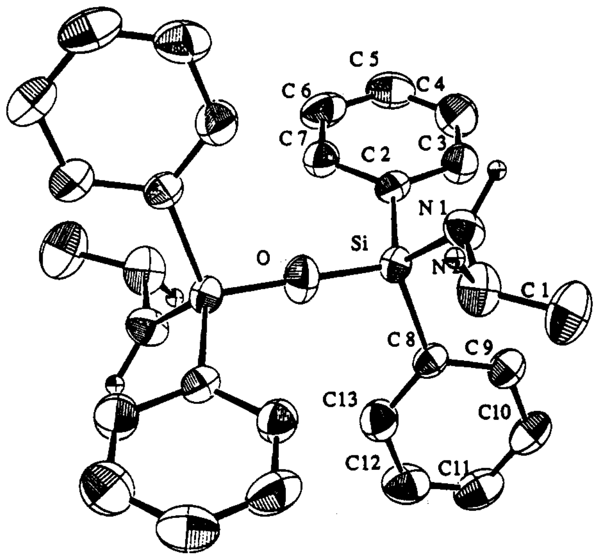

Figure 1. ORTEP plot of 1 (50% ellipsoids). H atoms have been omitted for clarity, except those on nitrogen.

Table 1. Important Bond Lengths (Å) and Angles (deg) of 1

Si–O	1.6131(13)	O–Sia	1.6131(13)
Si–N1	1.695(4)	N1–N2	1.430(5)
Si–C2	1.876(5)	N2–C1	1.432(7)
O–Si–N1	113.02(15)	Si–N1–N2	114.3(3)
O–Si–C2	109.54(15)	Si–N1–H	127.0(3)
O–Si–C8	107.93(16)	N2–N1–H	118.7(3)
N1–Si–C2	106.75(20)	N1–N2–C1	111.7(4)
N1–Si–C8	110.37(19)	N1–N2–H	107.1(3)
C2–Si–C8	109.18(19)	C1–N2–H	111.9(4)
Si–O–Sia	180.0	Σ∠N1[a]	360.0(3)
		Σ∠N2[a]	330.7(4)

[a] Sum of angles at N atoms.

The longer Si–N and N–N distances in the hydrazinosilanes 3–6 (Table 2) could be explained in terms of steric repulsion due to the presence of silyl substituents on both nitrogen atoms. It is interesting to note that the Si–N bond lengths in hydrazinosilanes (Table 2) lie in the range 1.70–1.76 Å, which is also the range for most neutral aminosilanes.[1] It seems that the Si–N distances are not significantly affected by the formal oxidation state of the nitrogen atoms.[17,18] In compound 1, planar coordination at N1 and pyramidal coordination at N2 is evident from the sum of the angles about the nitrogen atoms. Similar geometries about the nitrogens are adopted in 2. Further scrutiny of Table 2 leads to the conclusion that the geometry about nitrogen in hydrazinosilanes falls into two categories. The first is characterized by one nitrogen which is planar and another which is pyramidal, such as is found in the compounds containing a SiNN fragment (1, 2, 7–9).[21] In the second, both nitrogens are planar, as in the compounds containing a Si_2NN fragment (3–6).

A projection of an ORTEP model of a fragment of 1 along the N–N bond is shown in Figure 2a. This conformation, in which the Si–N1–H plane bisects the H–N2–Me angle, conforms to the predictions of theory for this type of molecule.[22] A similar conformational relationship exists in the cyclic compound 7 (Figure 2b).[13]

The crystal structure of 1 shows no evidence for either intermolecular or intramolecular hydrogen bonding.

NMR Study in Solution. Experimental Si–O (Si–N) bond lengths are always shorter (ca. 0.05–0.15 Å) than the sum of the respective covalent radii based on the Schomaker–Stevenson equation.[1,23] This is true of all of the compounds discussed in the present paper. The most frequently used rationalization of these differences and the widening of the bond angle about O (or planar N) is $(p-d)\pi$ bonding with the donation of lone pair electrons from O (N) into the vacant 3d orbitals of silicon.[24] Over the years this bonding model has been questioned, particularly from the theoretical point of view.[12,25] Several interpretations of these structural features in terms of σ/π hyperconjugative effects,[26] steric and electrostatic effects,[12a–c,f] and second-order Jahn–Teller effects[27] have been proposed. However, an alternative explanation that considers the details of frontier orbital interactions that involve only p and s orbitals accounts for the structural and chemical observations in a comprehensive and relatively straightforward manner.[4]

In an attempt to obtain more information about the nature of Si–O (Si–N) bonding, an NMR study of 1 and 2 was carried out. The NMR data for compounds 1 and 2 (^1H, ^{15}N, and ^{29}Si) are summarized in Table 3.

A significant shielding (6.7 ppm) of ^{29}Si in 1 relative to 2 may be attributed to the delocalization of the lone pairs (n_π and/or n_σ) on oxygen into unoccupied π^* orbitals on silicon, which favors a linear geometry at oxygen.[4] There may also be a contribution from the fact that the $\equiv$Si—O group is less electron withdrawing than the MeNHNH group. The ^1H NMR data show that the proton on N1 in 1 is shifted downfield about 0.7 ppm and the proton on N2 couples very weakly with the methyl group compared to 2. This suggests a lower electron density on N1. The strong downfield shift of the N1 resonance relative to N2 in the ^{15}N NMR spectra of both 1 and 2 indicates that the $SiPh_2$ group is an electron acceptor compared to a Me group, which is in agreement with the ^{29}Si and ^1H data.

Experimental Section

All reactions were carried out under an atmosphere of dry argon. The solvents used were dried over Na/benzophenone and

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Table 2. Comparison of Si–N and N–N Bond Lengths and Sum of Angles about Nitrogen in Some Silylhydrazine Compounds

compd	Si–N (Å)	N–N (Å)	$\Sigma\angle N$[a]	ref
bis(2-methylhydrazino)diphenylsilane (**2**)	1.695(4)	1.430(5)	*b*	13
bis(2-methylhydrazino)bis(diphenyl)disiloxane (**1**)	1.695(4)	1.430(5)	360 N1, 330.7 N2	this work
tetrasilylhydrazine (**3**)	1.731(4)	1.457(16)	360	16
tetrakis(phenylsilyl)hydrazine (**4**)	1.728(2)		360	17
1,6-diaza-2,5,7,10-tetrasila[4.4.0]bicyclodecane (**5**)	1.722(2)	1.497(2)	360	18
bis(1-aza-2,6-disila-1-cyclohexyl) (**6**)	1.730(2)	1.474(3)	360	18
1,2,4,5-tetraaza-3,3,6,6-tetraphenyl-3,6-disilacyclohexane (**7**)	1.731(4)	1.455(6)	360 N1, 344.8 N2, 359.1 N4, 336.0 N5	13
1,2,4,5-tetraaza-1,4-dimethyl-3,3,6,6-tetraphenyl-3,6-disilacyclohexane (**8**)	1.719(4)	1.436(6)	360 N1, 353.5 N2, 359.9 N4, 350.5 N5	19
1,2,4,5-tetraaza-3,3,6,6-tetrafluoro-1,2,4,5-tetra(*tert*-1,2,4,5-tetra(*tert*-butyldimethylsilyl)-3,6-disilacyclohexane (**9**)	1.748(4)[c]	1.491(8)	357.6	20

[a] Sum of angles at N. [b] Hydrogens on nitrogens were not located; however, the angle value for Si–N1–N2 of 119.3(3)° is suggestive of planar geometry at N1, and for N1–N2–C of 110.2(3), pyramidal at N2. [c] Endocyclic Si–N distance 1.705(4) Å, exocyclic Si–N distance 1.795(5) Å, average 1.748(4) Å.

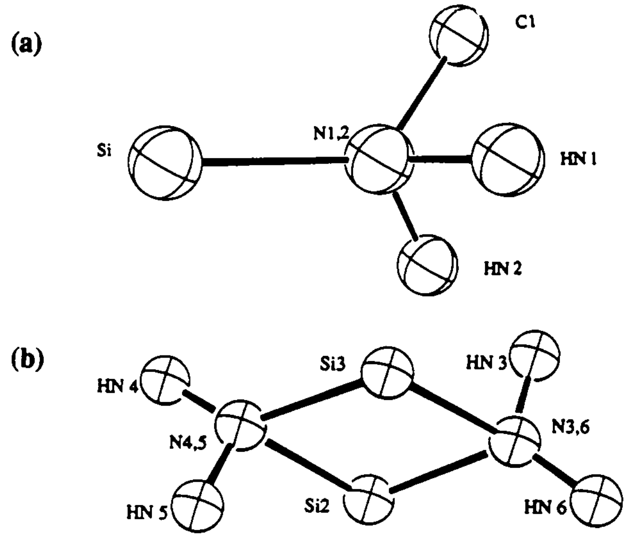

Figure 2. ORTEP fragments of compounds (a) **1** and (b) **7**, projected along the N–N bonds to show the common conformational relationship between the planar and pyramidal nitrogens.

Table 3. Comparison of NMR Data for Compounds 1 and 2

compd	^1H (δ)[a]	^{29}Si (δ)	^{15}N (δ)
$O[SiPh_2(N^1HN^2HMe)]_2$ (**1**)	2.24 (CH_3), 2.62 (N2H), 3.08 (N1H)	−36.21	−299.7 (N1), −327.8 (N2)
$Ph_2Si(N^1HN^2HMe)_2$ (**2**)	2.34 (CH_3), 2.59 (N2H), 2.91 (N1H)	−29.51	−305.4 (N1), −327.3 (N2)

[a] Phenyl group resonances in both compounds show multiple peaks in the region 7.20–7.80 ppm.

Table 4. Atomic Coordinates *x*, *y*, *z* and Thermal Parameters B_{eq}[a] for 1[b]

	x	*y*	*z*	B_{eq}
Si	0.65554(12)	0.52349(17)	0.01229(7)	2.52(5)
O	1/2	1/2	0	3.71(22)
N1	0.7327(4)	0.3293(5)	0.04342(22)	3.41(19)
N2	0.7156(4)	0.1804(5)	−0.01172(24)	4.06(22)
C1	0.8375(6)	0.1104(7)	−0.0341(3)	5.7(3)
C2	0.6962(5)	0.7002(6)	−0.0915(3)	2.78(21)
C3	0.8246(5)	0.7381(7)	0.1162(3)	3.69(24)
C4	0.8563(5)	0.8755(8)	0.1710(3)	4.8(3)
C5	0.7605(6)	0.9767(8)	0.2023(3)	4.6(3)
C6	0.6325(6)	0.9412(7)	0.1789(3)	4.5(3)
C7	0.5996(4)	0.8039(6)	0.1239(3)	3.36(22)
C8	0.7150(4)	0.6016(6)	−0.0850(3)	2.72(20)
C9	0.8477(5)	0.6013(6)	−0.095(3)	3.43(23)
C10	0.8975(5)	0.6733(7)	−0.1645(3)	4.2(3)
C11	0.8133(6)	0.7491(8)	−0.2233(3)	4.8(3)
C12	0.6836(6)	0.7499(8)	−0.2141(3)	4.7(3)
C13	0.6342(5)	0.6782(7)	−0.1452(3)	3.93(24)

[a] B_{eq} is the mean of the principal axes of the thermal ellipsoid. [b] ESDs refer to the last digit printed.

distilled prior to use. $Ph_2Si(NHNHMe)_2$, **2**, was prepared by the catalytic dehydrocoupling reaction of diphenylsilane with methylhydrazine, as described elsewhere.[13] All glassware was heated to at least 120 °C, evacuated, and purged with dry argon several times. ^1H (^{29}Si, ^{15}N) NMR spectra were recorded on a Varian XL-200 (300, 500) FT-NMR spectrometer at 20 °C with internal TMS (^1H, ^{29}Si) and external CH_3NO_2 (^{15}N) as standards. Benzene-d_6 was used as solvent. Low resolution mass spectra were obtained on a DuPont 21-492b spectrometer with an ionizing energy of 70 eV. IR spectra were recorded on a Bruker IFS-88 with a resolution of 4 cm^{-1} under a dry N_2 purge, using a mercury cadmium telluride detector. Elemental analyses were performed by Galbraith Laboratories, Inc.

Preparation of Compound 1. The reaction was carried out at ambient temperature in a 25-mL Schlenk flask, equipped with stirring bar and a rubber septum. After being purged with argon, the flask was charged with 0.40 g (1.47 mmol) of **2** and 5 mL of toluene. A solution of H_2O (0.0126 mL) in toluene/THF (10:1) (8 mL) was added dropwise to the above solution through a cannula. The mixture was then stirred for 20 h, and the solvents were removed under reduced pressure. The residue was recrystallized from toluene/hexane (1:1) by cooling a saturated solution to 4 °C for 2 weeks. Yield: 0.42 g (62%). ^1H NMR (200 MHz): δ 2.24 (s, 3H, N^2CH), 2.62 (br, 1H, N2H), 3.07 (s, 1H, N1H), 7.20 (m, 6H, Ph_2Si), 8.00 (m, 4H, Ph_2Si). ^{29}Si NMR (59.6 MHz): δ −36.21. $^{15}N\{^1H\}$ NMR (HMQC, 499.84 MHz): δ −299.72 (N1), −327.81 (N2). EI-MS (*m/e*, abundance, fragmentation): 424 (100) [$M^{\bullet+}$ − H_2NNHMe], 379 (6) [$M^{\bullet+}$ − H_2NNHMe −HNNHMe], 347 (7) [$M^{\bullet+}$ − H_2NNHMe − Ph], 318 (11) [$M^{\bullet+}$ − H_2NNHMe − PhH − Si], 303 (13) [$M^{\bullet+}$ − H_2NNHMe − PhH − Si − Me]. IR: 3337 (s), 3302 (s) (N–H); 1098 (s, Si–O–Si); 996 (s), 974 (s) (Si-N). Mp: 130–132 °C.

Anal. Calcd for $C_{26}H_{30}Si_2N_4O$: C, 66.34; H, 6.42; N, 11.90. Found: C, 66.61; H, 6.41; N, 11.95.

Crystal Structure of Compound 1. A crystal of dimensions 0.40 × 0.25 × 0.10 mm was sealed in a thin-walled capillary. The intensity data were collected on a Rigaku diffractometer, controlled by TEXRAY software,[28] using the $\theta/2\theta$ scan mode. The last least squares cycle was calculated with 32 atoms, 152 parameters, and 1017 out of 1646 reflections. Weights based on counting statics were used. The weight modifier *K* in KF_o^2 is 0.000 050. Standard intensities showed no change over the course of collection (average variation 0.17%). Merging *R* for 105 pairs of symmetry related reflections was 1.2%. The structure was

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(28) TEXRAY-Diffractometer Control Program. Molecular Structure Corp., College Station, Texas, 1985.

solved by direct methods.[29] Hydrogens were included in calculated positions, except those on nitrogen which were located in a difference map and then constrained to an idealized bond length. All non-hydrogens were refined anisotropically. All computations were performed using the NRCVAX system[30] of crystallographic software. Crystal data: $C_{26}H_{30}N_4Si_2O$, monoclinic, $P2_1/n$, $a = 10.3200(19)$ Å, $b = 7.3343(20)$ Å, $c = 16.658(3)$ Å, $\beta = 94.013(15)°$, $V = 1257.7(5)$ Å^3, $M = 470.72$, $D_c = 1.243$ mg m^{-3}, $Z = 2$, λ(Mo Kα) = 0.709 30 Å, $R_F = 0.047$, $R_w = 0.044$, GoF = 1.38. The atomic coordinates and thermal parameters are listed in Table 4.

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Supplementary Material Available: Complete tables of bond distances and bond angles, calculated hydrogen atom parameters, and anisotropic thermal parameters for 1 (3 pages). Ordering information is given on any current masthead page.

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