

CONFORMATIONAL ANALYSIS OF 1,1,2,2-TETRAARYL-DISILANES

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Abstract—A study of the static stereochemistry of 1,1,2,2-tetraphenyldisilane (1) and 1,1,2,2-tetramesityldisilane (2) by empirical force field calculations, X-ray diffraction, and $^1\text{H NMR}$ reveals that the preference for the *anti* conformation, exhibited by the unclamped 1,1,2,2-tetraarylethanes, is sustained in the analogous disilanes, although in somewhat attenuated form. This *anti* preference stands in contrast to the *gauche* preference of 1,1,2,2-tetraalkyldisilanes. Examination of $^3J_{\text{HH}}$ coupling constants for $\text{R}_2\text{HSiSiHR}_2$ ($\text{R} = \text{phenyl, mesityl, 2,6-dimethylphenyl, t-butyl, cyclohexyl}$) suggests the existence of a Karplus relation for H-Si-Si-H systems.

CONFORMATIONAL equilibrium mixtures of 1,1,2,2-tetraalkylethanes contain a preponderance of the *gauche* conformer which increases with increasing steric bulk of the alkyl groups.¹⁻⁴ A similar though less pronounced trend has been noted for 1,1,2,2-tetraalkyldisilanes⁵ and for the analogous diphosphines.⁶⁻⁸ In sharp contrast, unclamped 1,1,2,2-tetraarylethanes show a marked preference for the *anti* conformation.⁹ Since differences in the static and dynamic stereochemistry of analogously substituted ethanes and disilanes are in the last analysis traceable to differences in the metrics (i.e., bond lengths,

etc.) of the two systems,^{2,5b} the question arises whether the deviant behavior of the tetraarylethanes is also shared by the metrically dissimilar 1,1,2,2-tetraaryldisilanes. To answer this question, we undertook a study of the static stereochemistry of 1,1,2,2-tetraphenyldisilane (1) and 1,1,2,2-tetramesityldisilane (2). These compounds were chosen because the phenyl and mesityl groups span a wide range of steric demand, and because comparable structural information is already available for the corresponding ethanes.⁹ The present paper reports the results of this study.

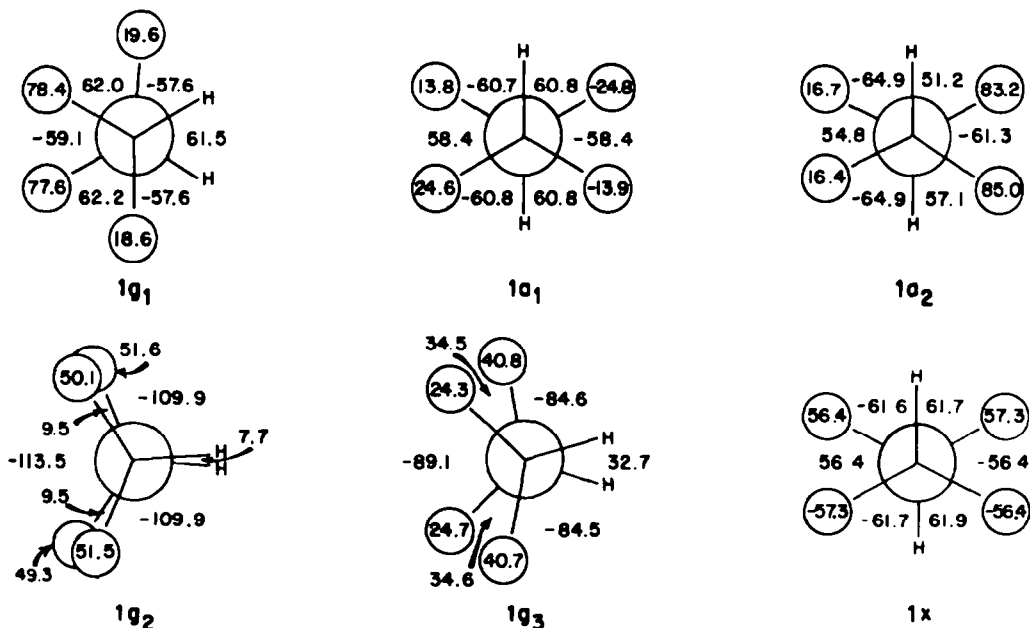


Fig. 1. Newman projections of some conformers of 1,1,2,2-tetraphenyldisilane (1). Top row, left to right: $1g_1$, $1a_1$ and $1a_2$. Bottom row, left to right: $1g_2$, $1g_3$ and $1x$. The numbers in the circles are the ring ϕ values and the other numbers represent the dihedral angles about the central bond.

1,1,2,2-Tetraphenyldisilane (1)

EFF calculations The empirical force field (EFF) method has proven successful in the calculation of ground state structures of strained as well as unstrained hydrocarbons^{10,11} and disilanes.^{5b} The energy hypersurface of 1 is expected to be very complex, and any exploration must therefore begin from a carefully selected set of input structures. In addition to *gauche-anti* isomerism about the central Si-Si bond, the phenyl rings are also free to rotate about the Si-C bonds; the degree of twist of each phenyl ring is described by the Si-Si-C-C dihedral angle (ϕ). As in the previous studies on the stereochemically correspondent¹² 1,1,2,2-tetraphenylethane,^{9a} all input structures were considered to have $|\phi| = 45^\circ$. Also, in keeping with the earlier observation^{5b} that ground state structures of the general type represented by 1 retain at least one element of symmetry, only non-asymmetric input structures were considered. Thus, four C_2 *gauche* and five *anti* (two C_{2v} and one each of C_s , C_1 , and C_2) input structures were used.

Minimization of the nine input structures by the previously described EFF,¹³ using the program BIGSTRN,¹⁴ yielded five minima, corresponding to two *anti* and three *gauche* conformers. Structural details are given in Figs 1 and 2. Table 1 lists the five conformers in order of increasing steric energy,^{13a} and shows that the lowest ($1g_1$) and highest ($1g_3$) energy minima are separated by only about 0.8 kcal/mol. Such a small energy difference is well within the error limits of the method,^{11a,15} and precludes reliable selection of one of the five minima as the ground state, i.e., as the global minimum. In short, the EFF calculations proved incapable of answering a central question of this study: does 1 adopt a *gauche* ($1g_1$, $1g_2$, $1g_3$) or an *anti* ($1a_1$, $1a_2$) conformation in the ground state? We therefore turned to X-ray analysis for further clarification of this point.

X-ray analysis Crystals of 1,¹⁶ obtained from benzene 60-70, were monoclinic, space group $P2_1/n$, with $a = 6.410(2)$, $b = 7.660(1)$, $c = 21.439(5)$ Å, $\beta = 90.37(2)^\circ$, and $d_{\text{calcd}} = 1.156 \text{ g cm}^{-3}$ for $Z = 2$ ($C_{24}H_{22}Si_2$, $M = 366.61$).

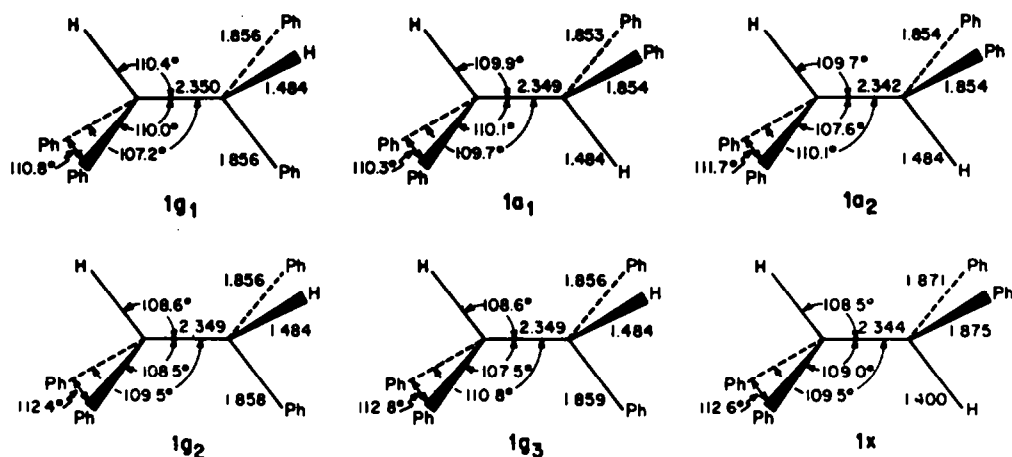


Fig. 2. Bond lengths (Å) and angles of some conformers of 1,1,2,2-tetraphenyldisilane (1), arranged as in Fig. 1. The numbers on the left of each structure are bond angles and those on the right are bond lengths. The two halves of each molecule are related by the symmetry given in Table 1, and the values shown represent averages based on idealized symmetry.

Table 1. Calculated steric energies of some conformers of 1,1,2,2-tetraphenyldisilane (1)

Structure	Symmetry	Steric Energy (kcal/mol)
$1g_1$	C_2	-7.12
$1a_1$	C_1	-6.68
$1a_2$	C_2	-6.55
$1g_2$	C_2	-6.35
$1g_3$	C_2	-6.31
$1x$	C_1	48.69

The intensity data were measured on a Hilger-Watts diffractometer (Ni filtered Cu K α radiation, $\theta - 2\theta$ scans, pulse height discrimination). A crystal measuring approximately $0.4 \times 0.5 \times 0.5$ mm was used for data collection; the data were corrected for absorption ($\mu = 15.0 \text{ cm}^{-1}$). A total of 1403 reflections were measured for $\theta < 57^\circ$, of which 1343 were considered to be observed ($I > 2.5\sigma(I)$). The structure was solved by a multiple solution procedure¹⁷ and was refined by full-matrix least squares. Three low theta reflections which were strongly affected by extinction were omitted from the final refinement. In the final refinement anisotropic thermal parameters were used for the heavier atoms and isotropic temperature factors were used for the H atoms. The H atoms were included in the structure factor calculations but their parameters were not refined. The final discrepancy indices are $R = 0.037$ and $wR = 0.051$ for the remaining 1340 observed reflections. The final difference map has no peaks greater than $\pm 0.1 \text{ e } \text{Å}^{-3}$.

The molecule sits on an inversion center in the crystal. Thus, it was necessary to locate only one silicon atom and the atoms of two phenyl rings. A stereoview of the final structure (1x) is given in Fig. 3, and the final

parameters in Tables 2 and 3. The C_i conformation of 1x is perfectly *anti*, and in this respect is comparable to 1a₁ and 1a₂. A comparison of bond lengths and bond angles (Fig. 2) of 1x with those of the five structured conformers shows good agreement; all six structures exhibit nearly standard values, reflecting the uncrowded nature of the molecule (the expected exceptions are the Si-H bond lengths, which were not refined in 1x). However, although the dihedral angles about the Si-Si bond in all three *anti* structures (1a₁, 1a₂, and 1x) are also quite similar and between 50 and 65°, corresponding to staggered conformations (Fig. 1), the angles of twist of the phenyl rings (ϕ) differ markedly. A similar discrepancy had previously been encountered and discussed for the case of pentaphenylethane,¹⁸ and it had then been noted that this discrepancy illustrates an inherent difficulty in calculating the structures of large molecules by the EFF method: the potential hypersurface being no doubt extremely complex, one can never be certain that all the minima, including the global minimum, have been found. When 1x was used as an input structure, minimization yielded 1a₂. Although no attempts were made to explore the hypersurface further, in view of the prohibitive costs

Table 2. Final atomic parameters for 1,1,2,2-tetraaryldisilane (I) with standard deviations in parentheses

Atom	X	Y	Z	B
Si	0.4789 (1)	0.1515 (1)	0.00423 (2)	*
C(11)	0.3134 (3)	0.2326 (3)	-0.06215 (9)	*
C(12)	0.1280 (3)	0.3223 (3)	-0.05267 (9)	*
C(13)	0.0087 (3)	0.3806 (3)	-0.10235 (11)	*
C(14)	0.0707 (4)	0.3490 (3)	-0.16254 (11)	*
C(15)	0.2529 (4)	0.2588 (3)	-0.17311 (10)	*
C(16)	0.3734 (3)	0.2022 (3)	-0.12367 (10)	*
C(21)	0.3647 (3)	0.2035 (2)	0.08217 (9)	*
C(22)	0.4730 (3)	0.3073 (3)	0.12473 (9)	*
C(23)	0.3972 (4)	0.3343 (3)	0.18409 (11)	*
C(24)	0.2148 (4)	0.2604 (3)	0.20236 (10)	*
C(25)	0.1022 (4)	0.1599 (3)	0.16161 (11)	*
C(26)	0.1768 (3)	0.1304 (3)	0.10172 (10)	*
HSi	0.675	0.230	0.0004	4.5
H(12)	0.081	0.345	-0.0080	5.0
H(13)	-0.127	0.447	-0.0946	6.0
H(14)	-0.015	0.391	-0.1990	6.0
H(15)	0.302	0.237	-0.2177	6.0
H(16)	0.506	0.134	-0.1312	5.5
H(22)	0.610	0.362	0.1123	5.0
H(23)	0.481	0.411	0.2133	6.0
H(24)	0.164	0.281	0.2467	7.0
H(25)	-0.038	0.107	0.1754	6.0
H(26)	0.094	0.053	0.0709	5.5

* Anisotropic thermal parameters are given in Table 3.

Table 3. Final anisotropic thermal parameters for 1,1,2,2-tetraaryldisilane (I) with standard deviations in parentheses

Atom	⁴ B ₁₁ × 10	⁴ B ₂₂ × 10	⁵ B ₃₃ × 10	⁴ B ₁₂ × 10	⁵ B ₁₃ × 10	⁵ B ₂₃ × 10
Si	234 (2)	202 (1)	217 (2)	32 (1)	11 (3)	-6 (3)
C(11)	274 (5)	169 (4)	234 (5)	15 (3)	0 (12)	29 (10)
C(12)	305 (6)	212 (4)	249 (5)	49 (4)	-19 (14)	-26 (11)
C(13)	330 (7)	239 (5)	321 (6)	77 (4)	-115 (16)	-35 (14)
C(14)	401 (8)	245 (5)	281 (6)	39 (5)	-255 (17)	42 (13)
C(15)	437 (8)	281 (6)	225 (5)	52 (5)	10 (16)	28 (13)
C(16)	330 (6)	252 (5)	243 (5)	71 (4)	77 (14)	46 (12)
C(21)	251 (5)	162 (3)	228 (5)	29 (3)	-15 (12)	27 (10)
C(22)	307 (6)	217 (4)	236 (5)	11 (4)	-62 (13)	2 (11)
C(23)	448 (8)	274 (6)	239 (6)	31 (5)	-179 (17)	-65 (13)
C(24)	471 (9)	298 (6)	220 (5)	75 (6)	143 (17)	98 (14)
C(25)	363 (7)	259 (5)	329 (7)	27 (5)	312 (18)	172 (14)
C(26)	323 (7)	203 (4)	296 (6)	2 (4)	75 (15)	-15 (12)

The anisotropic temperature factor has the form

$$\exp(-h^2 B_{11} + k^2 B_{22} + l^2 B_{33} + 2hkB_{12} + 2hlB_{13} + 2klB_{23})$$

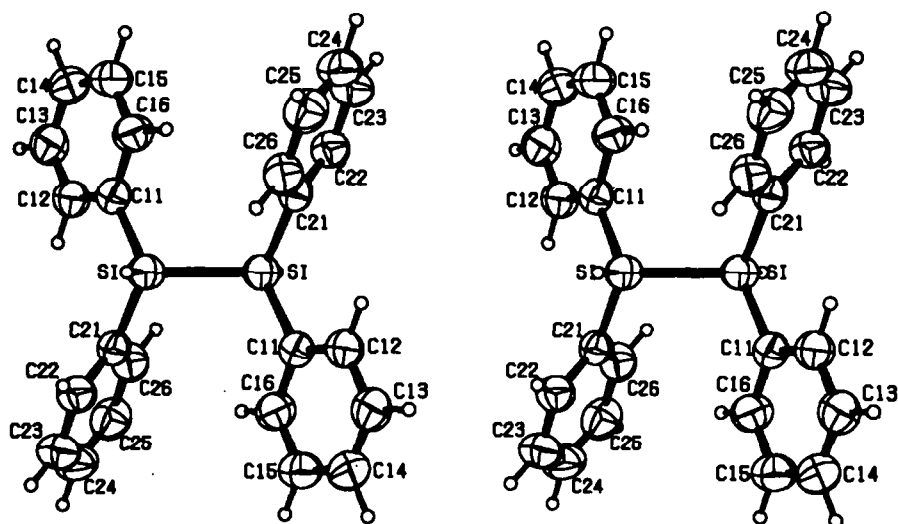


Fig. 3. Stereoview of the X-ray structure of 1,1,2,2-tetraphenyldisilane (1). Molecular C_1 symmetry.

that such a search would entail, it would appear that the X-ray structure does not correspond to a minimum on this surface.

1,1,2,2-Tetramesityldisilane (2)

EFF calculations Nine input structures for 2 were chosen on the same basis as those for 1. Minimization yielded seven minima, corresponding to three *gauche* and four *anti* conformers. The two lowest energy structures uncovered were *anti* ($2a_1$) and *gauche* ($2g_1$), with the former 2.5 kcal/mol more stable than the latter (Table 4). The steric energies of the remaining five minima were from 3.3 to 11.3 kcal/mol higher than that of $2a_1$, and the corresponding conformers are therefore not expected to contribute significantly to the conformational equilibrium mixture. The EFF calculations thus predict that 2 at room temperature should exist almost entirely in the *anti* conformation. Structural details for $2a_1$ and $2g_1$ are presented in Figs 4 and 5.

Both structures possess approximate C_2 symmetry. In each structure, two symmetry related rings (the A rings) are twisted so that one of the *ortho*-Me groups in each ring points toward the inside of the molecule. These rings have small values of $|\phi|$, and the Si-Si bond lies almost in the plane of the rings. In the other pair of symmetry related rings (the B rings), which have large $|\phi|$ values, the Si-Si bond axis is almost perpendicular to the planes of the rings. Also notable in each structure is the extreme expansion of one pair of Si-Si-C bond angles from tetrahedral geometry: in these angles, the C atoms belong to the A rings. Thus, in both $2a_1$ and $2g_1$, this pair of rings is bent back from the central bond (the other pair is slightly bent in), evidently as a result of nonbonded interactions arising from the inward projecting methyl groups on the A rings. The large magnitude of these distortions, in what is a relatively uncrowded system, is probably a reflection of the smaller bending constants about silicon, as compared to carbon.¹⁹

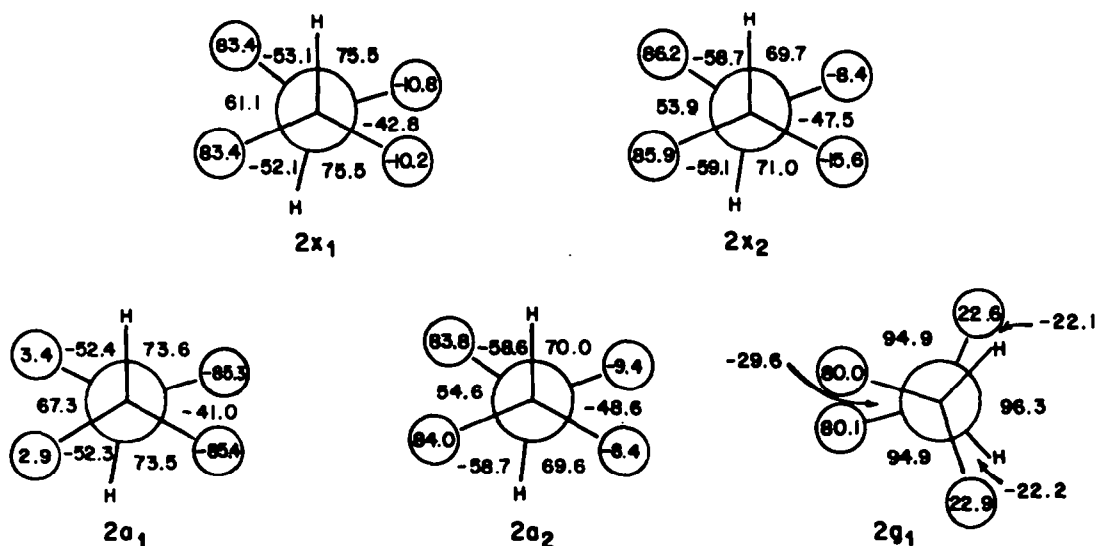


Fig. 4. Newman projections of some conformers of 1,1,2,2-tetramesityldisilane (2). Top row, left to right: $2x_1$ and $2x_2$. Bottom row, left to right: $2a_1$, $2a_2$ and $2g_1$. The numbers have the same meanings as those in Fig. 2.

Table 4. Calculated steric energies of some conformers of 1,1,2,2-tetraaryldisilane (2)

Structure	Symmetry	Steric Energy (kcal/mol)
$2a_1$	C_2	10.55
$2g_1$	C_2	13.08
$2x_1$	C_2	111.55
$2x_2$	C_2	106.88
$2a_2$	C_2	10.11

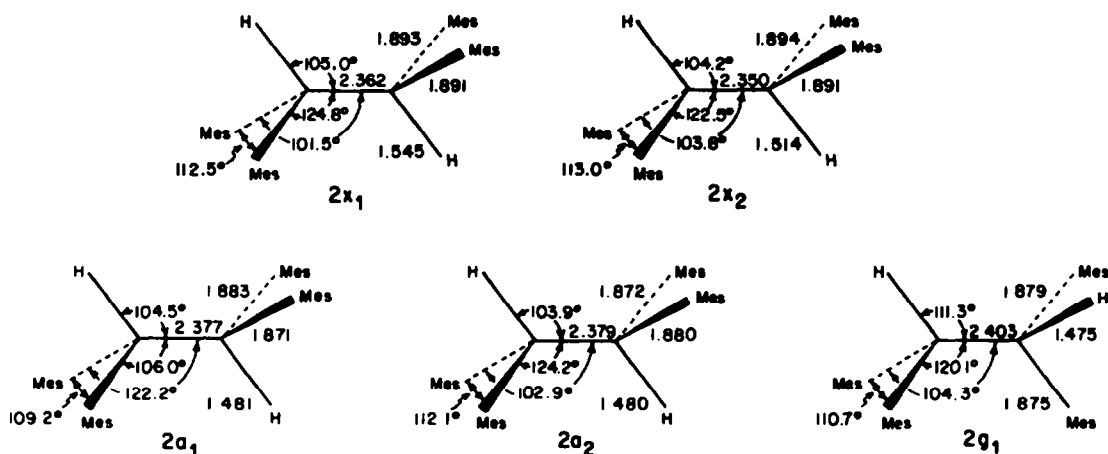


Fig. 5. Bond lengths (Å) and angles of some conformers of 1,1,2,2-tetraaryldisilane (2) arranged as in Fig. 4. The numbers have the same meanings as those in Fig. 2. The two halves of each molecule are related by idealized C_2 symmetry.

To test the reliability of the EFF prediction, an X-ray analysis of 2 was also undertaken.

X-ray analysis Crystals of 2 (see Experimental Section), obtained from a benzene-ethanol mixture, were orthorhombic, space group $Pcab$, with $a = 8.195(2)$, $b = 39.037(6)$, $c = 40.673(6)$ Å, and $d_{\text{calcd}} = 1.092 \text{ g cm}^{-3}$ for $Z = 16$ ($C_{36}H_{46}Si_2$, $M = 534.94$). The intensity data were measured on a Hilger-Watts diffractometer (Ni filtered $\text{Cu K}\alpha$ radiation, θ - 2θ scans, pulse height discrimination). The size of the crystal used for data collection was approximately $0.10 \times 0.15 \times 0.7$ mm. The data were corrected for absorption ($\mu = 11.0 \text{ cm}^{-1}$). Of the 8766 independent reflections for $\theta < 57^\circ$, 5586 were considered to be observed ($I > 2.5\sigma(I)$). The crystal contained two symmetry nonequivalent molecules.

The structure was solved by a multiple solution procedure.¹⁷ The H atom positions were calculated from the molecular geometry. The orientations of the Me groups were based on peaks found on a difference map calculated after anisotropic refinement of the Si and C

atoms. For this purpose, the eight phenyl rings of the two independent molecules were split into two groups. Set A consists of the four rings for which the Si-Si-C-C torsion angles are about -11° (the even numbered rings comprising atoms C(21), ..., C(26), C(41), ..., C(46), etc.) and set B consists of the four rings for which the Si-Si-C-C torsion angles are about 90° (the odd numbered rings comprising atoms C(11), ..., C(16), C(31), ..., C(36) etc.). In set A ($n = 2, 4, 6, 8$), the methyl groups *ortho* to the silicon were oriented so that the torsion angles C(n1)-C(n2)-C(n7)-H and C(n1)-C(n6)-C(n9)-H were 160° for one H atom in each methyl group. In set B ($n = 1, 3, 5, 7$), the *ortho*-Me groups were rotated 20° about the $C_{\text{Me}}-C_{\text{aryl}}$ axis to bring these torsion angles to 180° . All eight *para*-Me groups were oriented so that the torsion angle C(n3)-C(n4)-C(n8)-H was 180° for one hydrogen of each Me group.

The final refinement was carried out by block diagonal least squares in which the matrix was partitioned into two blocks, one for each independent molecule. Aniso-

tropic thermal parameters were used for the Si and C atoms, and isotropic temperature factors were used for the H atoms. The four H atoms which are bonded to the Si atoms were refined, but all other H atoms were held fixed at their calculated positions. The final discrepancy indices are $R = 0.056$ and $wR = 0.053$ for the 5586 observed reflections. There were no peaks greater than $\pm 0.1 e \text{ \AA}^{-3}$ on the final difference map.

Structural details of both independent molecules ($2x_1$ and $2x_2$) are given in Figs 4 and 5, a stereoview of $2x_1$ is presented in Fig. 6, and the final parameters are listed in Tables 5 and 6. On inspection of Figs 4 and 5, it is immediately apparent that both $2x_1$ and $2x_2$ agree with the EFF prediction of an *anti* ground state for 2. Another feature shared among the three structures ($2x_1$, $2x_2$, and $2a_1$) is the essential C_2 symmetry, which pairwise relates mesityl rings with small and large angles of twist (*A* and *B* rings, respectively); furthermore, the large expansion and slight compression of the Si-Si-C(*A*) and Si-Si-C(*B*) bond angles, respectively, predicted by the EFF calculation and commented on above, is faithfully reproduced in the X-ray structures (Fig. 5). In one respect, however, the X-ray structures differ significantly from the calculated one: in $2x_1$ and $2x_2$, the *A* rings define C-Si-Si-C dihedral angles which are smaller than the corresponding angles defined by the *B* rings, whereas in $2a_1$ this relationship is exactly reversed. Furthermore, in the X-ray structures the H atoms are bent away from the *A* rings and toward the *B* rings, whereas the reverse is the case for $2a_1$; expressed in terms of the H-Si-Si-H dihedral angle which contains the *A* rings, $2x_1$, $2x_2$, and $2a_1$ have values of 193.8, 188.2, and 172.0°, respectively. Since in all three structures the rings which define the smaller C-Si-Si-C dihedral angles are contained by the larger H-Si-Si-H dihedral angle, it follows that these rings must also define the larger H-Si-Si-C dihedral angles. In particular, for $2x_1$ and $2x_2$ the average values of H-Si-Si-C(*A*) are 75.5 and 70.3°, respectively, and the average values of H-Si-Si-C(*B*) are -52.6 and -58.9°, respectively, whereas for $2a_1$ the average value of H-Si-Si-C(*A*) = -52.3° and of H-Si-Si-C(*B*) = 73.5°.

This discrepancy was resolved when $2x_1$ was used as an input structure in an EFF calculation. In this case (and in contrast to the case of 1, discussed above), minimization of $2x_1$ resulted in a new structure ($2a_2$) which is depicted in Figs 4 and 5, and which closely resembles the X-ray structures in all respects, including bond lengths, bond angles, central dihedral angles, ring twists and the relationships between these parameters. Furthermore, as shown in Table 4, the calculation also places $2a_2$ ca 0.5 kcal mol lower in energy than $2a_1$. Thus, within the error limits of the force field, $2a_1$ and $2a_2$ are of comparable energy. This finding dramatically illustrates the need for a thorough exploration of the hypersurface in EFF calculations of complex structures; as has been noted before,¹⁰ it is far from uncommon to get trapped in local (as opposed to global) minima, particularly with respect to torsional degrees of freedom.

Nuclear Magnetic Resonance Studies

The ¹H NMR spectrum of 1 at ambient temperature (32°) exhibits a singlet (δ 5.18 ppm) and a multiplet (δ 7.08-7.51 ppm) assigned to the Si-H and the Ar-H protons, respectively. Since the EFF calculations predict the existence of five conformers of 1 within a 0.8 kcal/mol range of stability, it was expected that a low temperature ¹H NMR study of 1 might exhibit splitting of the Si-H signal. However, at -100°, the Si-H signal remained a sharp singlet. There are three possible reasons for the absence of splitting: the calculations may have underestimated the energy differences between the five conformers, or splitting was unobserved due to accidental isochrony, or the temperature was not sufficiently low to prevent rapid interconversion of the conformers.

The ¹H NMR spectrum of 2 was measured at ambient temperature (33°) and also at the slow exchange limit (-80°). To assist in the assignment of signals in the Me region, the ¹H NMR spectra at 33° and at the slow exchange limit (-71°) were also recorded for 1,1,2,2-tetrakis(2,6-dimethylphenyl)disilane (3). The chemical shift data for 2 and 3 are collected in Table 7. The room

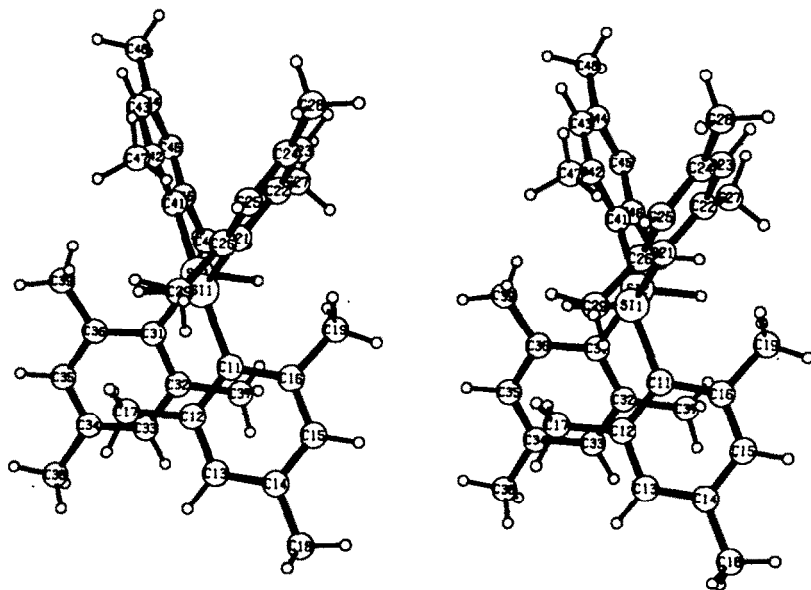


Fig. 6. Stereoview of the X-ray structure of 1,1,2,2-tetramesityl-disilane ($2x_1$). The view of the molecule (approximate C_2 symmetry) is along the Si-Si bond in a direction perpendicular to the C_2 axis. The *A* rings are at the top, the *B* rings at the bottom.

Table 5. Final atomic parameters for 1,1,2,2-tetramesityldisilane (2) with standard deviations in parentheses

Atom	X	Y	Z	B
Si(1)	0.3606(2)	0.34312(3)	0.18876(3)	*
Si(2)	0.5882(2)	0.37881(3)	0.18088(3)	*
Si(3)	0.6582(2)	0.41145(3)	0.44525(3)	*
Si(4)	0.6362(2)	0.35628(3)	0.42185(3)	*
C(11)	0.3637(6)	0.31287(10)	0.15248(10)	*
C(12)	0.2789(6)	0.32019(11)	0.12313(11)	*
C(13)	0.2862(6)	0.29738(13)	0.09673(11)	*
C(14)	0.3707(7)	0.26685(13)	0.09853(12)	*
C(15)	0.4575(6)	0.26002(11)	0.12692(12)	*
C(16)	0.4570(6)	0.28260(11)	0.15338(10)	*
C(17)	0.1807(6)	0.35245(12)	0.11801(12)	*
C(18)	0.3688(8)	0.24142(13)	0.07024(12)	*
C(19)	0.5669(6)	0.27417(11)	0.18202(11)	*
C(21)	0.3173(6)	0.32058(10)	0.22891(11)	*
C(22)	0.4287(7)	0.31594(11)	0.25481(12)	*
C(23)	0.3791(8)	0.30105(13)	0.28422(12)	*
C(24)	0.2218(9)	0.28946(13)	0.28882(13)	*
C(25)	0.1125(7)	0.29280(13)	0.26338(15)	*
C(26)	0.1539(7)	0.30859(11)	0.23381(12)	*
C(27)	0.6045(7)	0.32631(13)	0.25271(12)	*
C(28)	0.1707(9)	0.27382(17)	0.32149(14)	*
C(29)	0.0235(6)	0.31300(12)	0.20830(13)	*
C(31)	0.5556(5)	0.39603(11)	0.13774(10)	*
C(32)	0.6156(6)	0.37887(11)	0.10978(11)	*
C(33)	0.5816(6)	0.39086(13)	0.07830(11)	*
C(34)	0.4863(6)	0.41968(14)	0.07317(12)	*
C(35)	0.4290(6)	0.43676(11)	0.10011(12)	*
C(36)	0.4596(6)	0.42535(11)	0.13214(11)	*
C(37)	0.7184(6)	0.34683(12)	0.11191(11)	*
C(38)	0.4493(7)	0.43133(15)	0.03847(12)	*
C(39)	0.3808(6)	0.44534(11)	0.15972(11)	*
C(41)	0.6464(6)	0.41313(10)	0.21160(10)	*
C(42)	0.5531(6)	0.42363(11)	0.23903(11)	*
C(43)	0.6120(7)	0.44798(12)	0.26049(10)	*
C(44)	0.7636(7)	0.46280(12)	0.25672(11)	*
C(45)	0.8546(6)	0.45316(11)	0.22979(11)	*
C(46)	0.8006(6)	0.42875(11)	0.20741(11)	*
C(47)	0.3822(6)	0.41001(12)	0.24583(11)	*
C(48)	0.8271(7)	0.48865(13)	0.28129(12)	*
C(49)	0.9113(6)	0.42002(12)	0.17884(12)	*
C(51)	0.5358(6)	0.43741(10)	0.41466(10)	*
C(52)	0.6078(6)	0.45285(11)	0.38687(11)	*
C(53)	0.5106(8)	0.46766(12)	0.36263(11)	*
C(54)	0.3439(8)	0.46819(13)	0.36469(13)	*
C(55)	0.2736(6)	0.45423(13)	0.39235(13)	*
C(56)	0.3642(7)	0.43912(11)	0.41714(11)	*
C(57)	0.7908(6)	0.45429(12)	0.38224(11)	*
C(58)	0.2409(8)	0.48358(15)	0.33781(14)	*
C(59)	0.2723(6)	0.42439(13)	0.44601(12)	*
C(61)	0.6073(6)	0.42209(10)	0.48948(9)	*
C(62)	0.5291(6)	0.40064(10)	0.51201(11)	*
C(63)	0.5094(6)	0.41112(12)	0.54443(11)	*
C(64)	0.5616(7)	0.44218(13)	0.55568(11)	*
C(65)	0.6336(7)	0.46405(11)	0.53355(12)	*
C(66)	0.6584(6)	0.45492(10)	0.50070(11)	*
C(67)	0.4592(7)	0.36612(11)	0.50325(11)	*
C(68)	0.5460(8)	0.45212(14)	0.59160(11)	*
C(69)	0.7409(7)	0.48068(11)	0.47845(12)	*
C(71)	0.7359(6)	0.36221(10)	0.38025(10)	*
C(72)	0.6474(6)	0.37121(10)	0.35205(11)	*
C(73)	0.7273(7)	0.37866(11)	0.32260(11)	*
C(74)	0.8952(7)	0.37664(12)	0.32006(12)	*
C(75)	0.9830(6)	0.36678(12)	0.34714(13)	*
C(76)	0.9082(7)	0.36014(11)	0.37721(12)	*
C(77)	0.4635(6)	0.37323(12)	0.35145(11)	*
C(78)	0.9808(7)	0.38426(14)	0.28770(12)	*
C(79)	1.0165(6)	0.35059(13)	0.40578(12)	*
C(81)	0.7028(6)	0.31521(10)	0.44241(10)	*
C(82)	0.7957(6)	0.31265(11)	0.47123(11)	*
C(83)	0.8306(6)	0.28050(13)	0.48488(11)	*
C(84)	0.7750(7)	0.25068(13)	0.47051(13)	*
C(85)	0.6862(7)	0.25298(12)	0.44190(13)	*
C(86)	0.6480(6)	0.28447(12)	0.42782(11)	*
C(87)	0.8678(7)	0.34292(12)	0.48884(11)	*
C(88)	0.8150(7)	0.21616(12)	0.48598(14)	*
C(89)	0.5511(7)	0.28386(12)	0.39624(11)	*
HSi(1)	0.222(4)	0.3670(7)	0.1843(7)	2.2(7)
HSi(2)	0.738(4)	0.3547(8)	0.1800(8)	4.7(9)
HSi(3)	0.845(4)	0.4212(8)	0.4426(7)	3.7(8)
HSi(4)	0.455(4)	0.3512(8)	0.4168(8)	3.9(8)
H(13)	0.227	0.3035	0.0750	6.0
H(15)	0.521	0.2373	0.1283	6.5
H(17)A	0.129	0.3532	0.0951	7.5
H(17)B	0.250	0.3737	0.1202	7.5
H(17)C	0.088	0.3542	0.1344	7.5

Table 5. (Contd)

Atom	X	Y	Z	B
H(18)A	0.438	0.2207	0.0757	10.0
H(18)B	0.416	0.2520	0.0495	10.0
H(18)C	0.255	0.2335	0.0653	10.0
H(19)A	0.624	0.2522	0.1800	7.5
H(19)B	0.499	0.2732	0.2039	7.5
H(19)C	0.651	0.2931	0.1862	7.5
H(23)	0.464	0.2987	0.3034	6.5
H(25)	-0.009	0.2833	0.2662	7.0
H(27)A	0.657	0.3289	0.2753	8.0
H(27)B	0.619	0.3493	0.2411	8.0
H(27)C	0.673	0.3090	0.2401	8.0
H(28)A	0.049	0.2663	0.3212	13.0
H(28)B	0.180	0.2908	0.3403	13.0
H(28)C	0.236	0.2529	0.3276	13.0
H(29)A	-0.071	0.2966	0.2112	8.0
H(29)B	0.068	0.3092	0.1849	8.0
H(29)C	-0.025	0.3372	0.2083	8.0
H(33)	0.629	0.3776	0.0586	6.0
H(35)	0.359	0.4586	0.0970	6.5
H(37)A	0.750	0.3383	0.0887	7.5
H(37)B	0.658	0.3274	0.1227	7.5
H(37)C	0.824	0.3505	0.1241	7.5
H(38)A	0.378	0.4527	0.0385	11.0
H(38)B	0.390	0.4131	0.0255	11.0
H(38)C	0.552	0.4372	0.0261	11.0
H(39)A	0.314	0.4654	0.1520	7.5
H(39)B	0.465	0.4546	0.1761	7.5
H(39)C	0.303	0.4303	0.1737	7.5
H(43)	0.541	0.4553	0.2803	6.5
H(45)	0.965	0.4654	0.2257	7.0
H(47)A	0.347	0.4137	0.2693	8.0
H(47)B	0.372	0.3848	0.2407	8.0
H(47)C	0.296	0.4222	0.2314	8.0
H(48)A	0.940	0.4971	0.2751	10.0
H(48)B	0.834	0.4789	0.3042	10.0
H(48)C	0.753	0.5096	0.2822	10.0
H(49)A	0.994	0.4387	0.1744	8.0
H(49)B	0.845	0.4169	0.1578	8.0
H(49)C	0.972	0.3980	0.1828	8.0
H(53)	0.567	0.4782	0.3426	6.0
H(55)	0.146	0.4548	0.3948	6.5
H(57)A	0.825	0.4661	0.3611	7.5
H(57)B	0.842	0.4301	0.3812	7.5
H(57)C	0.850	0.4665	0.4009	7.5
H(58)A	0.119	0.4824	0.3427	11.0
H(58)B	0.258	0.4719	0.3157	11.0
H(58)C	0.266	0.5090	0.3341	11.0
H(59)A	0.150	0.4272	0.4439	7.5
H(59)B	0.306	0.4358	0.4673	7.5
H(59)C	0.294	0.3988	0.4487	7.5
H(63)	0.450	0.3950	0.5608	6.5
H(65)	0.676	0.4881	0.5413	7.0
H(67)A	0.444	0.3508	0.5231	8.0
H(67)B	0.535	0.3533	0.4874	8.0
H(67)C	0.350	0.3682	0.4919	8.0
H(68)A	0.595	0.4759	0.5960	11.0
H(68)B	0.609	0.4356	0.6063	11.0
H(68)C	0.430	0.4524	0.5989	11.0
H(69)A	0.735	0.5045	0.4879	8.0
H(69)B	0.693	0.4806	0.4560	8.0
H(69)C	0.863	0.4749	0.4761	8.0
H(73)	0.657	0.3858	0.3023	6.0
H(75)	1.108	0.3645	0.3456	6.5
H(77)A	0.420	0.3795	0.3290	7.5
H(77)B	0.420	0.3905	0.3678	7.5
H(77)C	0.412	0.3501	0.3575	7.5
H(78)A	1.099	0.3814	0.2896	10.0
H(78)B	0.954	0.4081	0.2795	10.0
H(78)C	0.940	0.3676	0.2695	10.0
H(79)A	1.136	0.3500	0.3994	7.5
H(79)B	0.988	0.3273	0.4152	7.5
H(79)C	1.006	0.3678	0.4246	7.5
H(83)	0.904	0.2795	0.5065	6.5
H(85)	0.653	0.2309	0.4306	7.0
H(87)A	0.896	0.3386	0.5120	8.0
H(87)B	0.789	0.3634	0.4879	8.0
H(87)C	0.972	0.3514	0.4769	8.0
H(88)A	0.761	0.1970	0.4726	10.0
H(88)B	0.768	0.2142	0.5092	10.0
H(88)C	0.934	0.2115	0.4870	10.0
H(89)A	0.552	0.2609	0.3848	8.0
H(89)B	0.591	0.3016	0.3796	8.0
H(89)C	0.429	0.2898	0.4002	8.0

* Anisotropic thermal parameters are given in Table 6.

Table 6. Final anisotropic thermal parameters for 1,1,2,2-tetraesityldisilane (2) with standard deviations in parentheses

Atom	4	5	5	5	5	5
	B11x10	B22x10	B33x10	B12x10	B13x10	B23x10
Si (1)	151(3)	59(1)	73(1)	-3(4)	-16(5)	-1(1)
Si (2)	150(3)	62(1)	65(1)	-6(4)	10(4)	-6(1)
Si (3)	166(3)	60(1)	58(1)	-6(4)	0(4)	2(1)
Si (4)	174(3)	64(1)	56(1)	-2(5)	6(5)	-3(1)
C(11)	153(9)	51(3)	69(3)	-14(15)	-15(16)	4(3)
C(12)	155(10)	66(4)	80(4)	-58(17)	-25(17)	1(3)
C(13)	194(11)	89(4)	73(4)	-44(19)	-76(18)	5(3)
C(14)	242(13)	84(4)	75(4)	-77(20)	20(20)	-11(3)
C(15)	219(12)	69(4)	84(4)	-8(18)	3(19)	-10(3)
C(16)	177(10)	66(4)	70(4)	-37(17)	-12(17)	6(3)
C(17)	207(12)	97(5)	98(4)	57(19)	-125(19)	4(3)
C(18)	375(16)	127(6)	95(4)	7(25)	-78(24)	-39(4)
C(19)	265(12)	80(4)	87(4)	127(19)	-86(20)	-2(3)
C(21)	171(11)	62(4)	71(4)	7(17)	11(17)	-3(3)
C(22)	223(12)	72(4)	70(4)	-33(19)	-14(20)	2(3)
C(23)	254(14)	96(5)	76(4)	16(21)	-28(21)	9(3)
C(24)	334(17)	100(5)	80(5)	-56(25)	75(25)	13(4)
C(25)	226(13)	96(5)	108(5)	-95(20)	77(23)	17(4)
C(26)	185(12)	69(4)	86(4)	-18(18)	10(20)	-1(3)
C(27)	201(12)	126(5)	90(4)	-72(21)	-52(20)	1(4)
C(28)	486(21)	193(7)	109(5)	-210(32)	135(29)	54(5)
C(29)	169(11)	98(5)	128(5)	-66(19)	-17(21)	14(4)
C(31)	133(9)	64(3)	61(3)	-39(15)	8(15)	-9(3)
C(32)	144(10)	73(4)	75(4)	-7(16)	18(17)	-9(3)
C(33)	183(11)	106(5)	65(4)	-28(19)	33(18)	-10(3)
C(34)	173(11)	110(5)	73(4)	-48(20)	-11(19)	14(4)
C(35)	202(11)	77(4)	81(4)	-19(17)	30(19)	7(3)
C(36)	155(10)	68(4)	74(4)	-52(17)	35(17)	1(3)
C(37)	216(12)	106(5)	84(4)	96(20)	53(18)	-18(3)
C(38)	299(15)	182(7)	75(4)	1(26)	-6(21)	36(4)
C(39)	231(12)	77(4)	87(4)	85(18)	54(19)	-8(3)
C(41)	157(10)	66(3)	57(3)	-43(16)	6(16)	-1(3)
C(42)	200(11)	62(4)	62(3)	-16(17)	-22(17)	3(3)
C(43)	243(13)	75(4)	61(3)	-64(19)	4(18)	-8(3)
C(44)	270(13)	86(4)	58(4)	-94(21)	-28(20)	-4(3)
C(45)	206(11)	83(4)	71(4)	-123(19)	-37(19)	9(3)
C(46)	170(11)	72(4)	63(3)	-13(17)	-4(17)	2(3)
C(47)	207(11)	96(4)	87(4)	-64(19)	133(19)	-19(3)
C(48)	393(17)	120(5)	94(4)	-257(25)	-69(24)	-27(4)
C(49)	195(11)	107(5)	93(4)	-64(19)	43(19)	-3(4)
C(51)	155(10)	55(3)	61(3)	9(15)	-10(16)	2(3)
C(52)	193(11)	61(4)	64(3)	-11(17)	-14(18)	4(3)
C(53)	235(13)	80(4)	66(4)	29(20)	6(20)	11(3)
C(54)	236(14)	97(5)	80(4)	101(22)	-81(22)	13(4)
C(55)	165(11)	97(5)	97(4)	60(19)	-2(20)	6(4)
C(56)	193(11)	76(4)	63(4)	-4(18)	-5(19)	0(3)
C(57)	182(11)	93(4)	84(4)	-30(18)	38(18)	26(3)
C(58)	318(16)	179(7)	122(5)	155(28)	-143(25)	54(5)
C(59)	163(10)	132(5)	89(4)	-11(19)	51(19)	9(4)
C(61)	187(10)	54(3)	58(3)	4(15)	-34(16)	-3(3)
C(62)	202(11)	58(4)	62(3)	-21(16)	22(17)	-3(3)
C(63)	227(12)	80(4)	64(3)	20(19)	37(18)	8(3)
C(64)	236(12)	87(4)	66(4)	85(20)	-55(19)	-15(3)
C(65)	241(12)	71(4)	76(4)	9(19)	-44(20)	-17(3)
C(66)	187(10)	58(3)	77(4)	12(17)	-38(18)	5(3)
C(67)	321(14)	74(4)	72(4)	-89(20)	101(19)	0(3)
C(68)	384(17)	151(6)	70(4)	52(26)	6(22)	-39(4)
C(69)	292(13)	67(4)	104(4)	-100(19)	-44(21)	-1(3)
C(71)	167(10)	67(4)	56(3)	-3(16)	34(16)	0(3)
C(72)	186(11)	67(4)	53(3)	36(17)	-3(18)	-2(3)
C(73)	224(12)	88(4)	53(4)	43(19)	-25(18)	-2(3)
C(74)	239(13)	89(4)	67(4)	25(20)	60(21)	7(3)
C(75)	169(11)	98(5)	83(4)	48(18)	73(19)	10(4)
C(76)	201(12)	79(4)	70(4)	30(18)	-33(19)	3(3)
C(77)	178(11)	118(5)	71(4)	53(19)	-68(17)	-8(3)
C(78)	329(15)	150(6)	87(4)	57(25)	177(23)	28(4)
C(79)	167(11)	147(6)	91(4)	39(21)	-28(19)	16(4)
C(81)	206(11)	64(4)	60(3)	-2(16)	32(17)	-3(3)
C(82)	218(11)	68(4)	68(4)	47(17)	59(18)	5(3)
C(83)	238(12)	86(4)	77(4)	71(20)	39(18)	15(3)
C(84)	270(14)	67(4)	104(4)	69(21)	182(22)	14(4)
C(85)	269(14)	62(4)	96(4)	-25(19)	148(22)	-9(4)
C(86)	226(11)	69(4)	74(4)	16(19)	95(19)	-5(3)
C(87)	298(13)	92(4)	80(4)	43(21)	-115(20)	0(3)
C(88)	339(16)	81(4)	155(6)	155(22)	163(25)	38(4)
C(89)	341(15)	88(4)	78(4)	-66(21)	12(21)	-23(3)

The anisotropic temperature factor has the form

$$\exp(-h^2 B_{11} + k^2 B_{22} + l^2 B_{33} + 2hkl B_{12} + 2hl B_{13} + 2kl B_{23})$$

Table 7. ^1H Chemical shift data for 1,1,2,2-tetramesityldisilane (2) and 1,1,2,2-tetrakis(2,6-dimethylphenyl)disilane (3)^{a,b}

Proton	2		3	
	33°C	-80°C	33°C	-71°C
<u>ortho</u> -methyl	2.14	1.71, 2.21, 2.24, 2.44	2.20	1.76, 2.23 2.28, 2.55
<u>para</u> -methyl	2.21	2.16	-	-
Si-H	5.48	5.43	5.56	5.52
Ar-H	6.70	6.54, 6.69, 6.81, 6.83	6.75-7.28(m)	6.59-7.41(m)

^aUnless otherwise specified, all signals are singlets; δ -values in ppm (± 0.01 ppm); internal standard TMS ($\delta_{\text{TMS}} = 0$). ^bIn CD_2Cl_2 at 100 MHz.

temperature spectra of the two compounds are very similar, except for the absence of a *para*-Me signal in 3 and the expected differences in the signals of the aromatic protons, which show up as a singlet in 2 and as a multiplet in 3.

The low temperature spectrum of 2 shows five singlets in the Me region in the ratio 1:1:2:1:1, while the low temperature spectrum of 3 shows four singlets in a 1:1:1:1 ratio. Evidently, at the slow exchange limit there are four diastereotopic *ortho*-Me groups in both 2 and 3, and the signal of greater intensity in the Me region of 2 (δ 2.16 ppm) arises from the *para*-Me group.

The presence of four *ortho*-Me proton and four aromatic proton signals in 2 at the slow exchange limit (Table 7) rules out structures with C_{2h} and C_1 symmetry (which, in the absence of accidental isochrony, should give rise to two and eight signals, respectively) and is consistent only with C_n , C_2 , and C_1 point group symmetry.^{9a} The NMR data are thus consistent with the C_2 structure of 2 obtained by EFF calculations and by X-ray diffraction. The existence of two diastereotopic aryl groups (the *A* and *B* rings), with edge nonequivalence in each ring arising from slow rotation about the Si-C bond, also implies nonequivalence of the *para*-Me groups, and one should therefore in principle expect two signals for these protons at the slow exchange limit. The absence of splitting of the signal at δ 2.16 ppm must therefore be ascribed to accidental isochrony.

Of particular interest are the vicinal $^3J_{\text{HH}}$ coupling constants involving the central atoms (H-Si-Si-H) in 1-3, and related disilanes. Previous reports^{20,21} of vicinal coupling constants in disilanes have dealt only with molecules that give average values (e.g., coupling between H_3Si and H_2Si groups), ranging from 2.3 Hz for $(\text{SiH}_3\text{SiH}_2)_2\text{NCH}_3$ to 4.0 Hz for disilane^{20a} (though anions may have higher values).²¹ In the course of the present work we recorded the $^3J_{\text{HH}}$ values for 1, 2, 3, 1,1,2,2-tetra-*tert*-butyldisilane (4),^{5b} and 1,1,2,2-tetracyclohexyldisilane (5).^{5b} It should be noted that the

coupling constants in these compounds arise from a single vicinal interaction. The coupling constants were obtained by observing the ^{29}Si satellites of the Si-H signal. Ideally, such an ABX spectrum should exhibit eight signals in the AB portion of the spectrum; however, only the protons directly bonded to ^{29}Si were observed, because the $^2J_{\text{SiH}}$ is not sufficiently large to prevent the ^{29}Si -Si-H signals from being swamped by the intense Si-H signal. The $^3J_{\text{HH}}$ and $^1J_{\text{SiH}}$ values are collected in Table 8.

All five compounds exhibit $^1J_{\text{SiH}}$ couplings of the expected order of magnitude.²² The $^3J_{\text{HH}}$ couplings show considerable variation, with the smallest (<1.0 Hz for 4) and largest (9.8 Hz for 2) extending beyond both the lower and upper limits of previously reported frequencies. This large range of coupling constant values could in principle arise from a variety of sources, including electronic effects of the substituents and variations in Si-Si bond lengths, H-Si-Si bond angles, and H-Si-Si-H dihedral angles. However, the small effect of monosubstitution by ligands of differing electronic nature (H_3SiSiH_3 , $^3J_{\text{HH}} = 4.0$ Hz; $\text{H}_3\text{SiSiH}_2\text{F}$, $^3J_{\text{HH}} = 2.7$ Hz; $\text{H}_3\text{SiSiH}_2\text{CH}_3$, $^3J_{\text{HH}} = 2.5$ Hz)^{20a} makes it unlikely that electronic effects are responsible for this large spread. The role of variations in Si-Si bond lengths and H-Si-Si bond angles is also assessed as small, because 2 and 4, which exhibit the largest and smallest $^3J_{\text{HH}}$ values, respectively, possess bond lengths (2.379 Å and 2.391 Å^{2b}) and bond angles (103.9° and 103.8°^{5b}) which are more similar to each other than to the corresponding values for 1 (2.350 Å and ca 109°), whose coupling constant represents an intermediary value. On the other hand, it seems reasonable that a relationship of the Karplus type should exist between H-Si-Si-H dihedral angles and $^3J_{\text{HH}}$ values.²³ The molecules with the smallest and largest $^3J_{\text{HH}}$ values are calculated to exist exclusively as *gauche* (H-Si-Si-H = 94.7° for 4)^{5b} and *anti* (H-Si-Si-H = 171.9° for 2a₂) conformers, respectively. Compound 5 is found to exist

Table 8. $^1J_{SiH}$ and $^3J_{HH}$ values^a for some tetrasubstituted disilanes ($R_2HSiSiHR_2$)

Compound	R	$^1J_{SiH}$	$^3J_{HH}$
<u>1</u>	phenyl	187.0	2.5
<u>2</u>	mesityl	186.5	9.8
<u>3</u>	2,6-dimethylphenyl	184.6	9.1
<u>4</u>	<u>tert</u> -butyl	164.7	< 1
<u>5</u>	cyclohexyl	158.5	< 3

^aRefers to coupling between H-Si-Si-H vicinal protons. ^bIn Hz.

as a *gauche* conformer by EFF calculations ($H-Si-Si-H = 108.2^\circ$)^{5b} and by X-ray analysis ($H-Si-Si-H = 115.7^\circ$)^{5b} and the small $^3J_{HH}$ value is in accord with these findings. In the case of **1** the EFF calculations predict a set of *gauche* and *anti* conformers all very close in energy (Table 1) and the molecular structure in the crystal is an *anti* conformer which is not a minimum on the EFF hypersurface. Thus it is possible that at normal temperatures **1** exists as a mixture of *anti* and *gauche* conformers in solution, a notion that is supported by the intermediary value of the observed vicinal coupling constant.

CONCLUSION

The question which motivated the present study, whether the preference of unclamped 1,1,2,2-tetraarylethanes for the *anti* conformation is shared by the analogously substituted disilanes, has been answered in the affirmative: the preference is preserved, although in attenuated form. Both molecules adopt the *anti* conformation in the solid state. However, while tetraphenyl- and tetramesitylethane are both found to exist effectively exclusively in the *anti* conformation at normal temperatures,⁹ tetraphenyldisilane is calculated to exist as a mixture of *gauche* and *anti* conformers, and the more crowded tetramesityldisilane is calculated to prefer the *anti* ground state by only 3 kcal/mol. The decrease in the *anti-gauche* energy gap is readily understood as a consequence of the longer Si-Si and Si-C bonds (as compared to C-C bonds), which lessen intramolecular nonbonded interactions and, consequently, decrease the energy difference between the rival conformations. Whether this trend will also be observed in the metrically comparable tetraaryldiphosphines is hard to predict, since electronic effects (i.e., conjugation of the lone pair on phosphorus with the aryl π -system) may have a significant effect on conformational stability.

The present study has also uncovered the possible existence of a Karplus relationship²³ between $^3J_{HH}$ and the H-Si-Si-H dihedral angle. There appears to be evidence for a similar relationship in H-Si-C-H systems.^{19,24}

EXPERIMENTAL

¹H NMR Measurements. The ¹H NMR spectra of **1** were

recorded at 90 MHz on a JEOL FX90Q spectrometer operating in the Fourier transform mode. The solvent was CFC₃, and C₆D₆ (10%) was added to obtain a lock (²H). The internal standard was TMS, and the temps, considered to be accurate to $\pm 2^\circ$, were measured with a Wilmad low temp NMR thermometer. The ¹H NMR spectra of **2** and **3** were recorded at 100 MHz on a Varian XL-100 spectrometer operating in the Fourier transform mode. The spectrometer was locked on ²H in the solvent, CD₂Cl₂. Temps, considered to be accurate to $\pm 2^\circ$, were measured with a copper-constantan thermocouple, and the internal standard was TMS. The $^3J_{HH}$ studies of **1-5** were also performed on the Varian XL-100 spectrometer operating in the Fourier transform mode, and again the spectrometer was locked on ²H in the solvent, CDCl₃.

1,1,2,2-Tetramesityldisilane (2). This compound was prepared by a method analogous to the preparation of 1,1,2,2-tetramesityldiphosphine.²⁵ Under dry N₂, bromomesitylene (25.0 g, 0.13 mol) was added dropwise to Mg metal (12.0 g, 0.5 mol) which was just covered with dry THF. After the addition was complete, an additional 100 ml of THF was added, the Grignard reagent was stirred for 1 hr, and then trichlorosilane (6.37 ml, 0.063 mol) was added dropwise. Following an overnight reflux, the mixture was cooled and then poured into NH₄Cl aq. The suspension was extracted with benzene and the solvent was removed to yield crude **2** (3.51 g). Recrystallization from a benzene-EtOH mixture yielded pure **2** (2.67 g, 15.8%) m.p. 230-233.5°. The ¹H NMR spectrum featured signals at δ (CDCl₃) 2.175 (6 H, s), 2.20 (12 H, s), 5.50 (1 H, s), and 6.70 (4 H, s). (Found: C, 80.83; H, 8.70; Si, 10.73. Calc. for C₃₆H₄₆Si₂: C, 80.80; H, 8.67; Si, 10.50%.)

1,1,2,2-Tetakis(2,6-dimethylphenyl)disilane (3). This compound was prepared by a procedure analogous to the preparation of **2** except in the substitution of 2,6-dimethylbromobenzene for bromomesitylene. The pure product (4.45 g, 31.1%) decomposed over the range 223-231°. The ¹H NMR spectrum featured signals at δ (CDCl₃) 2.20 (12 H, s), 5.58 (1 H, s), and 6.73-7.30 (6 H, m). (Found: C, 80.45; H, 8.29; Si, 11.54. Calc. for C₃₂H₃₈Si₂: C, 80.27; H, 8.00; Si, 11.73%.)

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