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METAL—SILICON BONDED COMPOUNDS

X *. THE MOLECULAR STRUCTURES OF 2,2,4,4,6,6,8,8-OCTAMETHYL-2,4,6,8-TETRASILA-1,5-MERCURACYCLOOCTANE AND BIS(TRIPHENYLSILYL) MERCURY

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Summary

The crystal and molecular structure determinations of 2,2,4,4,6,6,8,8-octamethyl-2,4,6,8-tetrasila-1,5-mercuracyclooctane, $Hg_2Si_4C_{10}H_{28}$ (I) and of bis(triphenylsilyl)mercury, $(Ph_3Si)_2Hg$ (II), are reported. The structures have been determined from single-crystal X-ray data collected by counter methods. Both molecules crystallize in the space group $P\bar{1}$ with one centrosymmetric molecule per unit cell. Each structure contains linear Si—Hg—Si groups, with mercury—silicon distances of 2.490(4) Å in I and 2.503(4) Å in II. In compound I the Si—Hg—Si groups are linked by methylene bridges which form an eight member ring in the chair conformation. The cell dimensions for compound I are a 6.277(2), b 8.408(2), c 9.274(4) Å, α 92.75(3), β 94.79(3) and γ 100.14(2)° with $R_1 = 0.062$ for 1809 observed reflections. The cell dimensions for compound II are a 9.999(4), b 11.727(8), c 7.654(5) Å, α 99.87(5), β 115.35(4) and γ 98.41(4)°, with $R_1 = 0.081$ for 2394 observed reflections.

Introduction

The existence of compounds containing Si—M bonds, where M is a main group metal, has been postulated for many years but has been demonstrated conclusively by structural studies only in the past few years. Compounds which have been shown to contain direct bonds of this type include the trimethylsilyllithiumhexamer [1] with multicenter silicon—lithium bonding, the bis(trimethylsilyl)magnesium · DME complex comprised of a tetrahedral magnesium

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* For part IX see ref. 2.

atom bound to two silicon atoms and to two oxygen atoms [2], and three compounds, 2,2,4,4,6,6,8,8-octamethyl-2,4,6,8-tetrasila-1,5-mercuracyclooctane ($Hg_2Si_2C_{10}H_{28}$) [3], lithium tetrakis(dimethylphenylsilyl)mercurate [3], and bis(trimethylsilyl)mercury [4] which contain direct silicon—mercury bonds. Evidence also has been reported for the insertion of silicon into a carborane cage in which the silicon enters into electron deficient bonding in a manner similar to that of many other elements [5,6].

In this paper and in subsequent papers [7,8] detailed studies of the structures of several of these metal—silicon bonded systems are presented. We report here the structure of the cyclic 8-membered ring compound 2,2,4,4,6,6,8,8-octamethyl-2,4,6,8-tetrasila-1,5-mercuracyclooctane (I) and the structure of bis(triphenylsilyl)mercury (II). Following papers will deal with the structure of the two mercurates $Li_2Hg(SiMe_2Ph)_4$ and $Li_2Hg(SiMe_3)_4$ [7] and with the structure of the $(LiSiMe_3)_6$ hexamer [8].

Experimental

All reactions were carried out using standard inert atmosphere and dry box techniques. 2,2,4,4,6,6,8,8-octamethyl-2,4,6,8-tetrasila-1,5-mercuracyclooctane (I) was prepared by the method of Bettler and Urry [9]. Greenish-yellow crystals were obtained when I was recrystallized from hot cyclohexane in a sealed tube. Bis(triphenylsilyl)mercury (II) was prepared by the method of Neumann and Bleukat by reaction of di-t-butylmercury with triphenylsilane in toluene [10]. The reaction was run in a sealed tube placed in an oil bath at 70° C for 48 hours. Pale yellow crystals of II formed during the course of the reaction and were removed directly from the reaction mixture. In both cases suitable single crystals were placed in thin walled capillaries in the dry box, sealed with grease or sealing wax and, after removal from the dry box, sealed with a torch.

The crystals were mounted on a Syntex $P2_1$ four-circle diffractometer. All X-ray data were obtained with Mo-K_α radiation (λ 0.71069 Å) which had been monochromatized using a highly oriented graphite crystal in the parallel mode. Lattice constants were obtained by least squares refinement based on 15 reflections centered by a programmed routine. Intensity data were collected by the $\theta - 2\theta$ scan technique with a scan rate of 3.91° /min and a scan range of $2\theta(\text{Mo-}K_{\alpha 1}) - 1.0°$ to $2\theta(\text{Mo-}K_{\alpha 2}) + 1.0°$; background counts were collected for a time equal to one-half the scan time. Standard deviations of intensities were assigned as follows:

$$\sigma(I) = [\sigma^2(I)_{\text{counter}} + (0.04 \times I)^2]^{1/2}$$

$\sigma^2(I)$counter = $C + 4B$, C = total counts, I = net intensity, B = total background counts.

Structure determination of $Hg_2Si_4C_{10}H_{28}$

Rotation and oscillation photographs indicated the triclinic crystal system. The refined lattice constants are a 6.277(2), b 8.408(2), c 9.274(4) Å, α 92.75(3), β 94.79(3), γ 100.14(2)°, V 479.15 Å^3 and $\rho_{\text{calc}} = 2.294$ g cm^{-3} for $Z = 1$. Independent data totalling 2382 reflections were collected to a maximum 2θ of 55°. The intensities of three standard reflections measured every 47 reflections showed a decrease of 15% during the data collection for which correaction was made by a step function. Of the 2382 reflections there were 1809 reflections with $I > 3\sigma(I)$, which were used throughout the solution and refinement process.

The structure was solved by conventional heavy atom techniques [11] in the space group $P\bar{1}$. A three dimensional Patterson function gave the position of the mercury atom, and Fourier and difference syntheses established the remaining non-hydrogen positions. Full matrix anisotropic least squares refinement on F yielded discrepancy factors: $R_1 = \Sigma||F_0| - |F_c||/\Sigma|F_0| = 0.062$ and $R_2 = [\Sigma w(|F_0| - |F_c|)^2/\Sigma w|F_0|^2]^{1/2} = 0.076$ and an error of fit equal to 2.56. Hydrogen atoms were assigned positions 0.97 Å from the carbon atoms in the expected geometry with the methyl groups in the staggered conformation relative to the mercury atom and two carbon atoms bonded to the neighboring silicon atom. Subsequent full-matrix anisotropic least squares refinement of non-hydrogen atomic positional parameters and anisotropic temperature factors, with fixed contributions from the hydrogen atoms having isotropic thermal parameters assigned values 10% higher than those of the heavy atoms to which they are attached, did not alter the discrepancy factors reported above.

The final difference synthesis contains three peaks with residual electron density of $\sim 4\ e/\text{Å}^3$. Each peak was within 1 Å of the mercury and therefore could be considered residual electron density from the mercury. The maximum residual electron density not associated with any atom in the structure was 2.0 $e/\text{Å}^3$. The location of these peaks did not suggest the presence of any additional atoms. The rather large residual electron density observed may be attributed to the fact that no absorption correction was made due to the fact that the crystal did not have well-defined faces. Attempts to refine the structure in the acentric space group $P1$ did not improve the discrepancy factors reported above.

Atomic coordinates and anisotropic thermal parameters are given in Table 1 and bond distances and angles in Table 2. Structure amplitudes (×10) are available *.

Structure Determination of $(Ph_3Si)_2Hg$

Rotation and axial photographs indicated the triclinic crystal system. The refined lattice constants are a 9.999(4), b 11.727(8), c 7.654(5) Å, α 99.87(5), β 115.35(4), γ 98.41(4)° and V 774.4 Å^3 with $Z = 1$ and ρ_{calc} 1.54 g cm^{-3}. Independent data totalling 3835 reflections were collected to a maximum of 2θ of 55°. The intensities of three standard reflections measured every 97 reflections showed no significant decrease in intensity during data collection. Of the 3835 reflections there were 2394 reflections with $I > 2.5\sigma(I)$, which were used throughout the solution and refinement process.

The structure was solved by conventional heavy atom techniques [11] in the space group $P\bar{1}$, with the mercury atom at the origin. The data were corrected

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* The table of structure factors, supplementary tables and figures have been deposited as NAPS Document No. 03598 (31 pages). Order from NAPS, c/o Microfiche Publications, P.O. Box 3513, Grand Central Station, New York, N.Y. 10017. A copy may be secured by citing the document number, remitting $ 7.75 for photocopies or $ 3.00 for microfiche. Advance payment is required. Make checks payable to Microfiche Publications. Outside the U.S. and Canada add postage of $ 3.00 for photocopy and $ 1.00 for microfiche.

TABLE 1

ATOMIC COORDINATES AND ANISOTROPIC THERMAL PARAMETERS FOR $Hg_2Si_4C_{10}H_{28}$ [a, b]

Atom	x	y	z	B
Hg (1)	0.1142 (1)	0.1901 (1)	0.0401 (1)	
Si (2)	0.3177 (7)	0.1048 (5)	0.2584 (4)	
Si (3)	−0.0958 (7)	−0.2699 (5)	0.1775 (5)	
C (4)	−0.0663 (35)	−0.4442 (23)	0.2641 (21)	
C (5)	0.3498 (39)	−0.3393 (29)	0.1277 (28)	
C (6)	0.1683 (26)	−0.0941 (20)	0.3144 (18)	
C (7)	0.3424 (36)	0.2606 (25)	0.4152 (22)	
C (8)	0.6030 (29)	0.0843 (24)	0.2263 (22)	
H (9)	0.0180	−0.4702	0.3493	4.88
H (10)	−0.1002	−0.5378	0.1954	4.88
H (11)	−0.2002	−0.4143	0.2920	4.88
H (12)	0.3114	−0.4341	0.0606	6.79
H (13)	0.4297	−0.3665	0.2145	6.79
H (14)	0.4395	−0.2534	0.0825	6.79
H (15)	0.0336	−0.0736	0.3492	3.77
H (16)	0.2584	−0.1272	0.3935	3.77
H (17)	0.1984	0.2728	0.4392	5.29
H (18)	0.4199	0.3635	0.3883	5.29
H (19)	0.4215	0.2255	0.4986	5.29
H (20)	0.6820	0.1875	0.2012	4.81
H (21)	0.5999	0.0035	0.1475	4.81
H (22)	0.6746	0.0512	0.3137	4.81

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Hg (1)	0.0231 (2)	0.0131 (1)	0.0069 (1)	0.0042 (1)	−0.0008 (1)	0.0014 (1)
Si (2)	0.0225 (12)	0.0136 (7)	0.0067 (5)	0.0050 (7)	−0.0006 (6)	0.0007 (4)
Si (3)	0.0217 (12)	0.0134 (7)	0.0078 (5)	0.0048 (7)	0.0001 (6)	0.0024 (5)
C (4)	0.0410 (68)	0.0145 (29)	0.0114 (25)	0.0031 (35)	0.0035 (32)	0.0039 (21)
C (5)	0.0423 (81)	0.0200 (40)	0.0204 (40)	0.0078 (45)	0.0021 (45)	0.0015 (31)
C (6)	0.0232 (44)	0.0150 (26)	0.0086 (19)	0.0049 (27)	0.0009 (23)	0.0008 (18)
C (7)	0.0429 (74)	0.0180 (33)	0.0109 (24)	0.0074 (39)	−0.0021 (33)	−0.0024 (23)
C (8)	0.0233 (49)	0.0191 (33)	0.0141 (27)	0.0048 (32)	0.0020 (28)	0.0033 (24)

[a] Standard deviations from the variance-covariance matrix are given in parentheses for the least significant digit(s) in this and the following tables. [b] The form of the anisotropic temperature factor reported here is $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$.

for absorption (range 1.95—2.57, μ 50.7 cm^{-1}) and Fourier and difference syntheses established the remaining non-hydrogen positions. Full-matrix anisotropic least squares refinement on F of the non-hydrogen atoms yielded discrepancy factors $R_1 = 0.069$ and $R_2 = 0.080$. Examination of the phenyl C—C distances at this point revealed distortions in these distances which could be removed by normal least squares refinement (i.e., C—C, 1.11—1.59 Å). Acentric refinement yielded no significant improvement.

The rings were therefore treated as groups with known geometries and refined by full matrix group least squares [11]. The isotropic thermal parameters on each carbon atom were varied individually. The phenyl hydrogen atoms were assigned isotropic thermal parameters which were 10% higher than those of the carbon atoms to which they are attached. Acentric refinement in the space group $P1$ on F was attempted by selectively offsetting paired angles from one another in a variety of ways. This resulted in discrepancy factors of

TABLE 2

BOND DISTANCES AND ANGLES FOR $Hg_2Si_4C_{10}H_{28}$ [a]

Bond	Distance (Å)	Angle	Degrees
Hg (1)—Si (2)	2.505 (4)	Si (2)—Hg (1)—Si (3′)	178.7 (6)
Hg (1)—Si (3′)	2.501 (4)	C (8)—Si (2)—C (6)	109.2 (8)
Si (2)—C (6)	1.89 (2)	C (8)—Si (2)—C (7)	105.9 (9)
Si (2)—C (7)	1.89 (2)	C (6)—Si (2)—C (7)	108.5 (9)
Si (2)—C (8)	1.88 (2)	Hg (1)—Si (2)—C (8)	112.6 (6)
Si (3)—C (4)	1.88 (2)	Hg (1)—Si (2)—C (7)	110.7 (6)
Si (3)—C (5)	1.88 (2)	Hg (1)—Si (2)—C (6)	109.7 (5)
Si (3)—C (6)	1.87 (2)	Si (2)—C (6)—Si (3)	119.2 (9)
Hg (1)—Hg (1′)	3.286 (2)	C (6)—Si (3)—C (5)	109.6 (9)
		C (6)—Si (3)—C (4)	108.5 (8)
		C (5)—Si (3)—C (4)	106.2 (10)
		Hg (1′)—Si (3)—C (4)	111.6 (6)
		Hg (1′)—Si (3)—C (5)	111.4 (8)
		Hg (1′)—Si (3)—C (6)	109.5 (5)

[a] The primed atoms are related to the unprimed atoms by the inversion center at the origin.

R_1 = 0.079 and R_2 = 0.099 and was rejected on the basis of unrealistic thermal parameters for the carbon atoms and high correlations. Final centrosymmetric refinement on F yielded discrepancy factors of R_1 = 0.081 and R_2 = 0.106 and an error of fit of 2.64.

TABLE 3

ATOMIC PARAMETERS AND ANISOTROPIC THERMAL PARAMETERS FOR $(Ph_3Si)_2Hg$ [a]

Atom	x	y	z	B
Hg	0.000	0.000	0.000	a
Si	0.1226 (5)	0.1855 (4)	0.2810 (8)	a
C(1)	0.277 (2)	0.164 (1)	0.503 (2)	5.8 (4)
C(2)	0.523 (5)	0.142 (8)	0.844 (4)	11.0 (7)
C(3)	0.403 (3)	0.133 (3)	0.490 (3)	12.4 (9)
C(4)	0.275 (2)	0.184 (2)	0.687 (3)	13.8 (10)
C(5)	0.526 (4)	0.122 (7)	0.660 (4)	12.9 (9)
C(6)	0.397 (4)	0.173 (5)	0.857 (3)	16.7 (13)
C(7)	0.198 (2)	0.309 (1)	0.190 (2)	6.0 (4)
C(8)	0.301 (4)	0.486 (2)	0.042 (3)	13.1 (9)
C(9)	0.098 (2)	0.331 (2)	0.013 (2)	11.8 (8)
C (10)	0.351 (2)	0.374 (2)	0.293 (2)	11.8 (8)
C (11)	0.149 (3)	0.420 (2)	−0.061 (2)	15.7 (12)
C (12)	0.402 (2)	0.463 (2)	0.219 (3)	14.8 (11)
C (13)	−0.031 (2)	0.231 (1)	0.342 (2)	6.3 (4)
C (14)	−0.260 (2)	0.284 (3)	0.436 (4)	11.7 (8)
C (15)	−0.101 (2)	0.151 (1)	0.412 (3)	13.3 (10)
C (16)	−0.075 (2)	0.338 (1)	0.320 (3)	9.6 (6)
C (17)	−0.216 (2)	0.178 (2)	0.458 (3)	17.7 (14)
C (18)	−0.190 (3)	0.364 (2)	0.366 (4)	9.5 (6)

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Hg	0.0142 (1)	0.0072 (1)	0.0354 (3)	0.0010 (1)	0.0063 (2)	−0.0044 (1)
Si	0.0152 (6)	0.0079 (4)	0.0360 (14)	0.0004 (4)	0.0083 (8)	−0.0046 (6)

[a] Anisotropic thermal parameters are of the form $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$.

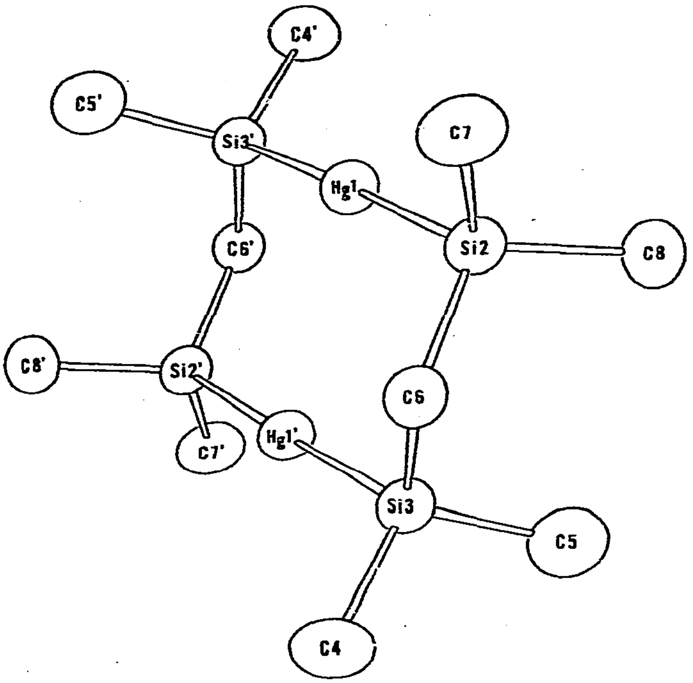

Fig. 1. The molecular structure and labelling of the 2,2,4,4,6,6,8,8-octamethyl-2,4,6,8-tetrasila-1,5-mercuracyclooctane (I).

Nineteen of the peaks in the final difference synthesis contained residual electron density ranging from 1.06 to 2.10 $e/Å^3$. Of these nineteen peaks, two were within 1.0 Å of the mercury atom and therefore could be considered residual electron density from the mercury, and the other seventeen were found to be associated with either apparent disorder of the phenyl groups or with a non-centric model we were unable to identify. Extensive attempts to define a disordered model with partial atomic occupancy were unsuccessful. The maximum residual electron density not associated with any atom in the structure was 0.45 $e/Å^3$. The location of these peaks did not suggest the presence of any additional atoms.

Atomic coordinates and thermal parameters are given in Table 3. The final group positional parameters and the coordinates defining the groups, bond distances, angles, and structure amplitudes are available *.

Results

Description of the molecule $Hg_2Si_4C_{10}H_{28}$

The cyclic compound 2,2,4,4,6,6,8,8-octamethyl-2,4,6,8-tetrasila-1,5-mercuracyclooctane is a centrosymmetric molecule containing two linear Si—Hg—Si moieties joined by methylene groups. The molecule is in a chair conformation with the methylene carbons bent above and below the plane formed by

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* See footnote p. 259.

the parallel linear Si—Hg—Si moieties. These features are shown in Fig. 1, which shows the molecular structure and labelling scheme, and in the stereoscopic view of the molecule. (Fig. S1, available with the supplementary material.)

The Si—C bond lengths (Table 2) are equal (1.88(2) Å av.) within experimental error. The average Hg—Si distance is 2.503(3) Å, and the Hg—Hg distance across the ring is 3.286(2) Å. The dihedral angle between either of the planes described by a methylene carbon atom and the two adjacent silicon atoms and the plane containing the two linear Si—Hg—Si units is 130.3°.

Description of the structure of $(Ph_3Si)_2Hg$

Bis(triphenylsilyl)mercury is a centrosymmetric molecule with a linear Si—Hg—Si skeleton. Fig. 2 shows the molecular structure and Fig. S2 (available with the supplementary material) shows a stereoscopic view of the molecule. The triphenylsilyl groups are in a staggered conformation with the phenyl groups tilted in a propeller-like fashion (Fig. S3). The average Si—C bond length is 1.88(2) Å and the Hg—Si distance is 2.490(4) Å.

Discussion

The environments about the silicon atoms in the two compounds are very similar and approach the anticipated tetrahedral geometry. In the ring compound, I, the Si—C distances lie in a very narrow range (1.87(2)—1.89(2) Å). In bis(triphenylsilyl)mercury, (II) the Si—C distances vary over a much greater range, (1.83(1)—1.90(2) Å), but the errors associated with these distances and

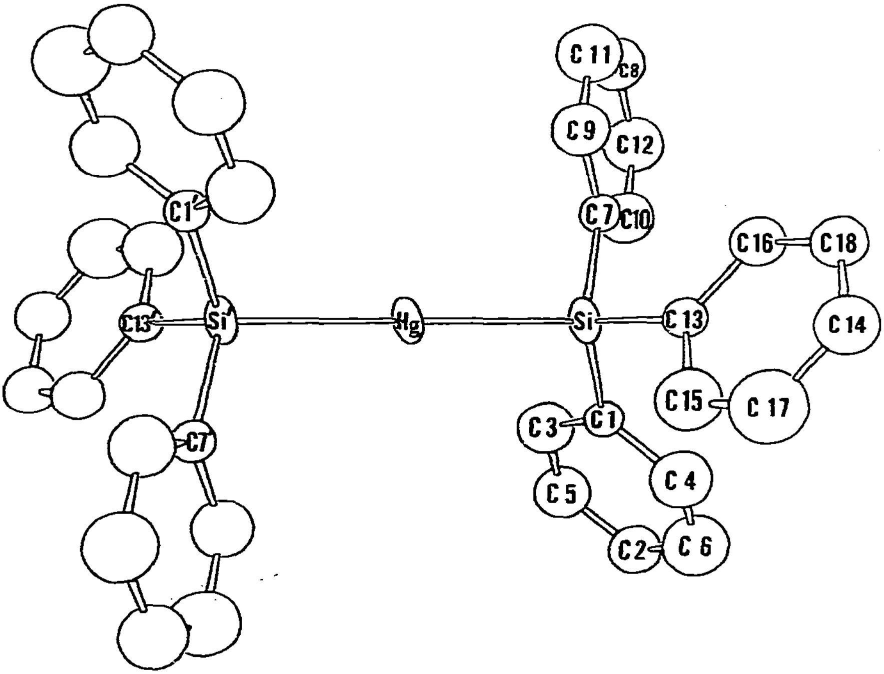
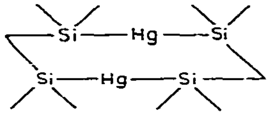


Fig. 2. The molecular structure and labelling of the bis(triphenylsilyl)mercury (II).

TABLE 4

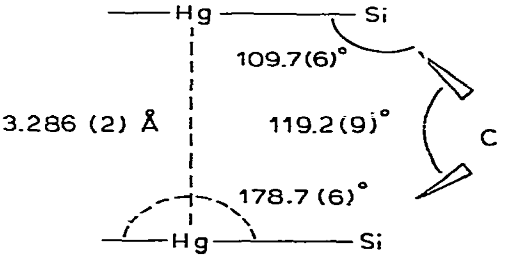
A COMPARISON OF C—Si AND Hg—Si BOND DISTANCES

Compound	C—Si distance (Å)	Hg—Si distance (Å)	ref.
Σ Covalent radii	1.95	2.48	13, 14, 15
$Si(CH_3)_4$	1.888 (20)		16
$Si_2(CH_3)_6$	1.90 (2)		17
$Si[Si(CH_3)_3]_4$	1.889 (3)		18
$Si(C_6H_5)_4$	1.87 (3)		19
Ph_3SiN_3	1.85 (1)		20
$(\eta\text{-}C_5H_5)_2ZrClSi(C_5H_5)_3$	1.913 (av)		21
$Mg(SiMe_3)_2 \cdot DME$	1.879 (7)	Mg—Si 2.630 (2)	2
$Hg[Si(CH_3)_3]_2$		2.500 (5)	4
	1.88 (2) (av)	2.501 (4) 2.505 (4)	this work
$Hg[Si(C_6H_5)_3]_2$	1.898 (20) 1.905 (19) 1.832 (14)	2.490 (4)	this work
$Li_2Hg[Si(CH_3)_3]_4$	1.90 (1) (av)	2.539 (2) 2.548 (2)	7
$Li_2Hg[Si(CH_3)_2(C_6H_5)]_4$	1.89 (1) (av)	2.549 (2) 2.493 (2)	7

the fact that the group least squares refinement was used precludes any serious speculation concerning the deviations observed. The average Si—C distance of 1.88 Å obtained for both compounds is consistent with those reported for normal silicon—carbon bonds some of which are summarized in Table 4.

The angles around the silicon atoms in the ring compound range from 105.9(9) to 112.6(6)° with the largest angle being the Hg(1)—Si(2)—C(8) angle and the smallest the C(7)—Si(2)—C(8) angle. The greatest deviation from tetrahedral geometry occurs around the methylene carbon and is seen in the Si—C—Si angle of 119.2(9)°. This angle and the Hg—Si methylene carbon angle are depicted below.

These deviations from tetrahedral geometry and the observed Hg—Hg distance of 3.286(2) Å may be interpreted in terms of Hg—Hg repulsion and as indicative of a 1.65 Å interactive distance for the mercury atom. This distance is slightly greater than the reported atomic radius of mercury (1.57 Å) and lies between the two values (1.50 and 1.73 Å) suggested by Grdnić [13] for the Van der Waals radius of mercury.



In bis(triphenylsilyl)mercury, all of the angles around silicon are very close to the tetrahedral value (range 107.8(5)° to 111.8(5)°).

The principal purpose of this study is the establishment of an experimental value for the Si—Hg bond distance in compounds containing linear Si—Hg—Si groups. The Si—Hg distances in $Hg_2Si_4C_{10}H_{28}$ and $(Ph_3Si)_2Hg$ are 2.503(4) Å and 2.490(4) Å respectively. These distances are in excellent agreement with the recently reported value of 2.500(3) Å in $(Me_3Si)_2Hg$ [4]. Thus, it would appear that a value of 2.50 Å is typical for the Si—Hg bond for a linear mercury compound. The value predicted from the sum of the covalent radii, using the value of 1.18 Å for silicon [14] and of 1.30 Å for digonal mercury [13,15] yields 2.48 Å, in good agreement with experimental values.

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