$$\mathbf{2} + e^- \overset{E_1^{0'}}{\rightleftharpoons} \mathbf{2}^- \qquad (1)$$

$$\mathbf{2}^- + e^- \overset{E_2^{0'}}{\rightleftharpoons} \mathbf{2}^{2-} \qquad (2)$$

$$\mathbf{2}^{2-} \xrightarrow{k} 2\,\mathbf{1}^- + \text{'[Mo(CO)}_2\text{]'} \qquad (3)$$

The two redox potentials and the rate constant of the decomposition reaction of $\mathbf{2}^{2-}$ were estimated by computer simulation to be $E_1^{0'} = -1.20$ V, $E_2^{0'} = -1.53$ V, and $k = 0.19\ s^{-1}$. On the basis of these redox potentials, it is reasonable to assume that not the Mo atom but the two Co centers undergo reduction. Hence, the valence balance of $\mathbf{2}^-$ can be roughly expressed as $Co^{III}Mo^0Co^{II}$, which suggests that the Mo bridge assists electronic interaction between the Co sites to form a thermodynamically favorable mixed-valence state. This is supported by the electronic spectrum of $\mathbf{2}^-$, generated by the reduction of **2** with Na in THF, in which a broad band at 1160 nm ($\varepsilon = 60\ mol^{-1}dm^3cm^{-1}$) is attributed to intervalence transfer.[9] Detailed studies on the optical and magnetic properties of the reduced forms of **2** are in progress

Experimental Section

2: All manipulations were carried out under nitrogen or argon. $BF_3 \cdot OEt_2$ (0.134 mL, 95.0 %, 1.0 mmol) was added dropwise to a stirred solution of **1**[10] (0.106 g, 0.40 mmol) and $[Mo(CO)_3(py)_3]$[11] (0.084 g, 0.20 mmol) in diethyl ether (30 mL) at room temperature, and the mixture was stirred for 2 h. The solvent was evaporated under vacuum, and the components of the residue were separated by thin-layer chromatography on silica gel with toluene/hexane (2/1) as eluent. The component in the first band was eluted with toluene and recrystallized from hexane to give 50 mg (0.074 mmol, 37 %) of fine brown crystals of **2**. Elemental analysis calcd for $C_{24}H_{18}Co_2MoO_2S_4$ (%): C 42.39, H 2.99, S 18.85; found: C 42.18, H 2.82, S 18.59; ^{1}H NMR (270 MHz, $CDCl_3$, 25 °C): $\delta = 5.15$ (s, 10 H, Cp), 6.34 (dd, J(H,H) = 5.5, 3.3 Hz, 4 H, Ph), 6.79 (dd, 4 H, Ph); IR (KBr disk): $\tilde{\nu} = 1860$, 1923 cm^{-1} (CO).

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[5] X-ray strucure analysis of **2**: A crystal (0.20 × 0.20 × 0.60 mm) was mounted in a glass capillary, and data were collected at 296 K on a Rigaku AfC7R diffractometer with graphite-monochromated $Mo_{K\alpha}$ radiation ($\lambda = 0.7107$ Å) and rotating-anode generator. Crystal data: $C_{24}H_{18}O_2MoCoS_4$, $M_r = 680.45$, triclinic, space group $P\bar{1}$, $a = 12.040(3)$, $b = 15.858(4)$, $c = 6.587(2)$ Å, $\alpha = 101.89(2)$, $\beta = 98.86(2)$, $\gamma = 92.78(2)^\circ$, $V = 1211.8(6)$ Å^3, $Z = 2$, $\rho_{calcd} = 1.865\ g\,cm^{-3}$; of 7381 reflections ($6 < 2\theta < 60^\circ$), 7068 were unique. The linear absorption coefficient μ for $Mo_{K\alpha}$ radiation is 22.3 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied and resulted in transmission factors ranging from 0.88 to 1.00. The data were corrected for Lorentzian and polarization effects. The structure was solved by direct methods and expanded by Fourier techniques. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement was based on 4707 observed reflections ($I > 3\sigma(I)$) with unweighted and weighted agreement factors of $R = 0.041$ and $R_w = 0.040$. All calculations were performed with the teXsan crystallographic software package of Molecular Structure Corporation. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-108002. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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The Synthesis and Molecular Structure of the First Two-Coordinate, Dinuclear σ-Bonded Mercury(I) RHgHgR Compound[**]

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Michael Bendikov, Karl Klinkhammer, and
Yitzhak Apeloig*

In memory of Nicolai S. Vyazankin

Most of the known mercury compounds have a formal +2 oxidation state.[1–5] σ-Bonded organomercury(I) compounds with a +1 oxidation state for example RHgHgR, R = organic substituent, have been frequently cited in the literature as

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short-lived transients, but not even one of these compounds has been yet fully characterized.[2–5] There are about 50 known crystal structures of dimercury compounds having a Hg_2 unit, which formally may indicate the presence of a Hg^I-Hg^I bond.[2] However, most of these compounds are ionic and have complex oligomeric structures. For example, in crystalline Hg_2Cl_2 each mercury atom is coordinated to six chlorine atoms, two close and four more distant.[5] The two closest known compounds that can be considered to have a Hg^I-Hg^I bond are $[Hg_2(Me_6C_6)_2][AlCl_4]_2$, which has a Hg_2^{2+} unit with a Hg–Hg bond distance of 251.5 pm,[6a] and the tetranuclear $(np_3)Co\text{-}Hg\text{-}Hg\text{-}Co(np_3)$ complex $((np_3 = N(CH_2CH_2PPh_2)_3)$ with a Hg–Hg distance of 265.1 pm.[6b] However, the first compound is ionic and the second is at best a distant model for the long-sought RHgHgR species. We report herein the synthesis and the structural characterization of $[(Me_3SiSiMe_2)_3Si]_2Hg_2$ (**1**), the first known nonionic two-coordinate, dinuclear σ-bonded mercury(I) compound.

In the course of our studies[7] towards the preparation of new polysilane dendrimers[8] we required mercury silyl compounds with highly branched polysilane skeletons. Triorganylsilyl(alkyl)mercury compounds are usually prepared by the reaction of tBu_2Hg with a triorganylsilane, leading to the formation of a mixture of bis(triorganylsilyl)mercury(II) (**2**) and of a triorganosilyl(*tert*-butyl)mercury(II) (**3**) [Eq. (1)].[9–11]

$$R_3SiH + tBu_2Hg \rightarrow \underset{\mathbf{2}}{R_3Si\text{–}Hg\text{–}SiR_3} + \underset{\mathbf{3}}{R_3Si\text{–}Hg\text{–}tBu} \qquad (1)$$

For example, reaction of tBu_2Hg with $(Me_3Si)_3SiH$ or with tBu_3SiH leads exclusively to the formation of the known $[(Me_3Si)_3Si]_2Hg$[12] or $(tBu_3Si)_2Hg$,[13] respectively. Similarly, reaction of $(Me_3SiMe_2Si)_3SiH$ (**4**) (prepared by the reaction of Me_3SiMe_2SiLi[14] with $HSiCl_3$ in hexane in a ratio of 3:1) with tBu_2Hg in a 1:1 ratio yields the expected mercury(II) compound **5** [Eq. (2)].

$$\underset{\mathbf{4}}{(Me_3SiMe_2Si)_3SiH} \xrightarrow{tBu_2Hg} \underset{\mathbf{5}}{(Me_3SiMe_2Si)_3Si\text{–}Hg\text{–}tBu} \qquad (2)$$

However, if silane **4** is used in at least a twofold excess relative to tBu_2Hg, only **1** having a Hg^I-Hg^I bond is obtained [Eq. (3)]. The addition of one equivalent of **4** to the silyl(alkyl)mercury(II) compound **5** also leads to the formation of **1**. It is therefore reasonable to assume that **5** is formed as an intermediate in the formation of **1** in the reaction shown in Equation (3).

$$2\,\underset{\mathbf{4}}{(Me_3SiMe_2Si)_3SiH} + tBu_2Hg \longrightarrow \underset{\mathbf{1}}{(Me_3SiMe_2Si)_3Si\text{–}Hg\text{–}Hg\text{–}Si(SiMe_2SiMe_3)_3} \qquad (3)$$

The X-ray crystal structure of **1**[15] (Figure 1) confirms the molecular formula of **1** and shows that the Si-Hg-Hg-Si fragment is linear. The Hg–Hg bond length in **1** of 265.69(1) pm is similar to that in $(np_3)Co\text{-}Hg\text{-}Hg\text{-}Co(np_3)$ (265.1 pm),[6b] but is significantly longer than in the ionic compound $[Hg_2(Me_6C_6)_2][AlCl_4]_2$ (251.5 pm)[6a] and in the oligomeric compounds Hg_2F_2 (250.8(1) pm),[16,17] Hg_2Cl_2 (252.6(6) pm),[17] and Hg_2Br_2 (249.0(10) pm),[5] and is slightly shorter than in Hg_2I_2 (267 pm).[17]

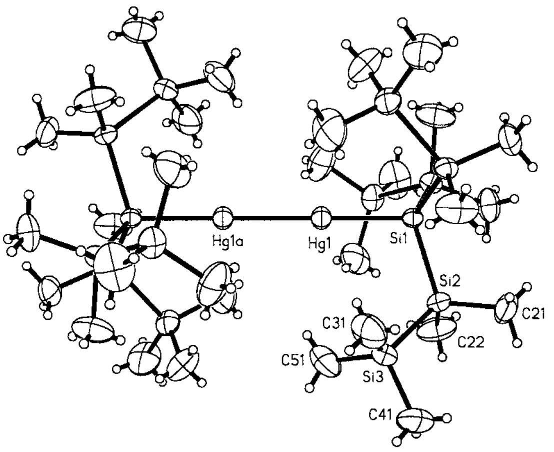

Figure 1. ORTEP diagram of **1**. Selected bond lengths [pm] and angles [°]: Hg1–Hg1a 265.69(8), Hg1–Si1 248.5(2), Si1–Si2 234.9(3), Si2–Si3 234.5(2), Si2–C22 187.3(6), Si2–C21 187.7 (5); Si1-Hg1-Hg1a 180.0, Si2-Si1-Si2 111.65(4), Si2-Si1-Hg1 107.19(5), Si3-Si2-Si1 111.65(7), C22-Si2-C21 107.0(3), C22-Si2-Si3 104.4(2), C21-Si2-Si1 111.65(7), C31-Si3-C41 108.8(3), C51-Si3-C41 108.1(3), C31-Si3-Si2 114.1(2), C41-Si3-Si2 108.1(2).

The $(Me_3SiMe_2Si)_3SiHg$ fragments in **1** have regular bond lengths and angles. The Hg–Si distance of 248.5(1) pm in **1** is similar to that in $(Me_3Si)_2Hg$ (250 pm),[18] indicating the absence of significant steric repulsions between the six Me_3SiMe_2Si groups or of strong electronic interactions with the Hg^I-Hg^I bond.

Some information on the nature of the novel Hg^I-Hg^I bond is obtained from spectroscopy. While, dialkylmercury compounds R_2Hg are usually colorless, disilylmercury compounds $(R_3Si)_2Hg$ are colored, their colors ranging from green to yellow, showing two absorption bands in the region of 300–400 nm.[19] The silyl(alkyl)mercury(II) derivative **5** is yellow and exhibits the expected two bands at 348 and 398 nm. The crystals of **1** on the other hand are red and the UV/Vis spectra of the red hexane solution of **1** reveals three bands at 334, 434, and 530 nm. By comparison with the spectrum of **5** we tentatively assign the red-shifted absorption at 530 nm to the Hg–Hg fragment, implying that the HOMO and LUMO of **1** are the σ and σ* orbitals of the Hg–Hg bond.

The ^{199}Hg chemical shifts in $(R_3Si)_2Hg$ are significantly more deshielded than in other mercury derivatives, the most deshielded value of $\delta = 987$ was previously reported for $[(Me_3Si)_3Si]_2Hg$.[20,21] In **1** the Hg atoms are even more deshielded, absorbing at $\delta = 1142.3$. This value is consistent with the linear correlation which has been found by Oliver et al. between the ^{199}Hg chemical shift and the lowest observed UV absorption of silylmercury(II) compounds.[20] Thus, the ^{199}Hg chemical shift of **1** and its UV absorption at 434 nm nicely fit the correlation line derived by Oliver et al.[20] The chemical shift of the silicon atoms directly bonded to Hg ($\delta = -48.7$) is very similar to that of the α-silicon atoms in $[(Me_3Si)_3Si]_2Hg$ ($\delta = -54.0$),[22] suggesting that the electronic environment of the α-silicon atoms in the two molecules is similar.

The red crystals of **1** show unusual stability. Thus, they can be handled without visible decomposition for several days even in air! However, when **1** is dissolved in diethyl ether or hydrocarbons it rapidly decomposes under light. Irradiation of a solution of **1** in hexane at $\lambda > 330$ nm leads selectively to the corresponding coupling product $[(Me_3SiMe_2Si)_3Si]_2$ (**6**). We are currently attempting to prepare other compounds that possess the novel Hg^I-Hg^I σ bond and are studying the chemistry of **1** further.

Experimental Section

Standard Schlenk techniques were used for all synthesis and all sample manipulations. NMR spectra were recorded in solution at room temperature in $CDCl_3$ or C_6D_6 using Bruker EM-200 or Bruker-400 instruments. Mass spectra (MS) were obtained with a Finnigan MAT TSQ 45 triple-stage guadrupole mass spectrometer. Elemental analyses were performed by the Microanalytical Laboratory of the University of Essen.

$(Me_3SiMe_2Si)_3SiH$ (**4**): A solution of Cl_3SiH (1.35 mmol) in toluene (10 mL) was slowly added under vacuum using Schlenk techniques to a solution of Me_3SiMe_2SiLi[14] (4.1 mmol) in toluene (10 mL). The product was isolated by filtration of the reaction mixture, evaporation of the solvent, and column chromatography.

^{1}H NMR (C_6D_6): $\delta = 0.18$ (27 H, s, $SiMe_3$), 0.34 (18 H, s, $SiMe_2$), 2.81 (1 H, s, SiH); ^{13}C NMR (C_6D_6): $\delta = -1.61$ (Me_3Si), -1.27 (Me_2Si); ^{29}Si NMR (C_6D_6): $\delta = -116.21$ (HSi), -39.93 ($SiMe_2$), -15.69 ($SiMe_3$); MS(EI): *m/z*: 349 $[M^+ - SiMe_3]$.

1 (**5**): $t Bu_2Hg$ (3.15 mmol) and **4** (6.50 mmol) (for the preparation of **5** 3.40 mmol of **4** were used) were heated under an argon atmosphere up to 120 °C. The reaction mixture was stirred for 4 h, during which time isobutane (5.25 mmol) (2.83 mmol for the preparation of **5**) was formed. Evaporation of volatile compounds from the reaction mixture yielded **1**(67 %) (or **5**(85 %)). Compound **1** could be purified by crystallization from pentane. $[(Me_3SiMe_2)_3Si]_2Hg_2$ (**1**):^{1}H NMR (C_6D_6): $\delta = 0.28$ (27 H, s, $SiMe_3$), 0.48 (18, s, $SiMe_2$); ^{13}C NMR (C_6D_6): $\delta = -0.68$ ($SiMe_3$), 1.37 ($SiMe_2$); ^{29}Si NMR (C_6D_6, 25 °C): $\delta = -49.64$ and -46.02 (SiHg), -30.72 and -30.35 ($SiMe_2$), -14.67 and -13.02 ($SiMe_3$); ^{29}Si NMR (C_6D_6, 60 °C): $\delta = -48.67$ (SiHg), -30.54 ($SiMe_2$), -14.82 ($SiMe_3$). ^{199}Hg NMR (C_6D_6): $\delta = 1142.3$; elemental analysis calcd for $C_{30}H_{90}Si_{14}Hg_2$ (%): C 28.93, H 7.28; found: C 29.34, H 7.47; MS(CI): *m/z*: 1043 $[M^+ - Hg]$. $(Me_3SiMe_2Si)_3SiHg t Bu$ (**5**): ^{1}H NMR (C_6D_6): $\delta = 0.23$ (27 H, s, $SiMe_3$), 0.39 (18 H, s, $SiMe_2$), 1.65 (9 H, s, CMe_3); ^{13}C NMR (C_6D_6): $\delta = -0.83$ ($SiMe_3$), 0.73 ($SiMe_2$), 30.78 (CMe_3), 79.85 (CMe_3); ^{29}Si NMR (C_6D_6): $\delta = -91.4$ (SiHg), -30.5 ($SiMe_2$), -16.3 ($SiMe_3$); ^{199}Hg (C_6D_6): $\delta = -104.5$.

6: A solution of **1** (or of **5**) in hexane was irradiated with a 80-W Hanau mercury vapor lamp (Q-81) equipped with an uranium glass filter ($\lambda >$ 330 nm). After separation of the hexane solution from metallic mercury, **6** was isolated by crystallization from hexane (yield 72–78 %). ^{1}H NMR (C_6D_6): $\delta = 0.28$ (54 H, s, $SiMe_3$), 0.48 (36 H, s, $SiMe_2$); ^{13}C NMR (C_6D_6): $\delta = 1.02$ ($SiMe_3$), 3.40 ($SiMe_2$); ^{29}Si NMR (C_6D_6): $\delta = -87.38$ (Si); -34.83 ($SiMe_2$), -10.97 ($SiMe_3$); MS(CI): *m/z*: 842 $[M^+]$.

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[15] A single crystal of dimensions 0.4 × 0.4 × 0.3 mm was immersed in Nujol, transferred into a capillary and mounted on a Siemens P4 diffractometer. Data were collected at 173 K using $Mo_{K\alpha}$ radiation (0.71069 Å, ω scan): 8588 collected, 2421 unique, and 2418 observed reflections; 81 parameters. $C_{30}H_{90}Hg_2N_2Si_{14}$, $M_r = 1245.46$ g mol^{-1}, rhombohedric, space group $R\bar{3}$ (no. 148), $a = 18.247(3)$, $c = 15.574(4)$ Å, $V = 4491(2)$ Å^3, $Z = 3$, $\rho_{calcd} = 1.382$ g cm^{-3}, $\mu = 5.421$ mm^{-1}, $wR2 = 0.0837$ ($R1 = 0.0355$ based on 1838 reflections with $F_o > 4\sigma(F_o)$). Largest difference peak/hole: $+3.014/-1.940$ e Å^{-3} (near Hg). The structure was solved by Patterson methods using the SHELXTL-PLUS package (G. M. Sheldrick, SHELXTL-PLUS An Integrated System for Solving, Refining and Displaying Structures from Diffraction Data, Rel.5.03, Siemens Analytical X-Ray Instruments Inc., **1989**). Refinement was achieved with the program SHELXL93 employing full-matrix least-squares methods on F_o^2. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were introduced at calculated positions and refined by using a riding model. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-103313. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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