

to room temperature, the precipitated mercuric chloride (7.4 g, 27 mmol) was filtered off. The solution was concentrated to 20 ml and cooled to room temperature, and 8.5 g (22 mmol, 76% yield) of brilliant yellow crystals of 2-biphenyltellurium trichloride separated. The product, recrystallized from dry chloroform, melted with decomposition at 157°. Anal. Calcd for $C_{12}H_9TeCl_3$: C, 37.23; H, 2.34; Cl, 27.47. Found: C, 36.80; H, 2.39; Cl, 27.28.

Method II. To 10.0 g (43.0 mmol) of 2-bromobiphenyl in 100 ml of dry diethyl ether cooled to ca. -60° under nitrogen, 20 ml of 2.2 *M* *n*-butyllithium in hexane was slowly added with efficient stirring. The solution became yellow at first; then a pale yellow finely divided solid separated. The mixture was stirred for 1 hr as it was allowed to warm to room temperature. The resulting suspension was added dropwise with stirring to a solution of 11.3 g (42.0 mmol) of tellurium tetrachloride in 150 ml of dry diethyl ether under nitrogen. A greenish-brown solid separated during the addition and the mixture was cooled externally with ice water as required to keep the reaction mixture at or below room temperature. Stirring was continued for 1 hr after which the mixture was filtered and the solid was extracted with 50 ml of hot, dry chloroform. After reduction of the volume to 15 ml and standing overnight, 2.6 g (6.7 mmol, 16% yield) of yellow crystals of 2-biphenyltellurium trichloride (mp 157°) resulted.

Dibenzotellurophene Dichloride, III. A 2.409-g (6.222-mmol) sample of recrystallized 2-biphenyltellurium trichloride was placed in an open test tube and heated in refluxing nitrobenzene (ca. 210°). The solid melted, and hydrogen chloride was evolved from the liquid for about 5 min, after which a grayish yellow solid residue remained. The heating was continued an additional 20 min to ensure completeness of the reaction. The yield of unrecrystallized product was 2.174 g (6.199 mmol, 99.6%). Recrystallization from ethylene bromide gave pale yellow crystals melting with decomposition at 354° .⁹ Anal. Calcd for $C_{12}H_8TeCl_2$: C, 41.10; H, 2.30. Found: C, 40.88; H, 2.38.

Dibenzotellurophene, I. A 1.244-g (3.55-mmol) sample of dibenzotellurophene dichloride, 25 ml of chloroform, and a solution of 4.4 g (20 mmol) of potassium pyrosulfite in 75 ml of water were placed in a 300-ml flask equipped with a magnetic stirrer. The dichloride dissolved as the reduction proceeded and the chloroform layer acquired a deep yellow color. After 2 hr of stirring, the separated chloroform layer was dried over $MgSO_4$ powder and filtered, and the solvent was allowed to evaporate in a slow stream of dry nitrogen.¹⁰ The dibenzotellurophene crystallized as pale yellow needles melting at 94.5° .¹¹ The yield was 0.821 g (2.94 mmol, 82.5%). Anal. Calcd for $C_{12}H_8Te$: C, 51.51; H, 2.88. Found: C, 51.72; H, 2.80. The crystal symmetry is that of the orthorhombic space group $P2_12_12_1$ with four molecules in the unit cell for which $a = 12.620 \text{ \AA}$, $b = 16.191 \text{ \AA}$, and $c = 4.637 \text{ \AA}$. The calculated density is 1.962 g cm^{-3} .

Dibenzotellurophene Dibromide. To a solution of 0.155 g (0.558 mmol) of dibenzotellurophene in 15 ml of carbon tetrachloride, 2.0 ml of 0.29 *M* Br_2 in carbon tetrachloride was added dropwise with stirring. The pale yellow, finely divided solid which separated weighed 0.242 g (0.550 mmol) and represented a 98.6% yield. Recrystallization from ethylene bromide gave small, brilliant yellow plates which melted with decomposition at 355° .¹² Anal. Calcd for $C_{12}H_8TeBr_2$: C, 32.78; H, 1.83. Found: C, 32.95; H, 1.84.

Dibenzotellurophene Diiodide. Method I. To a solution of 0.217 g (0.775 mmol) of dibenzotellurophene in 20 ml of carbon tetrachloride, 25 ml of 0.033 *M* I_2 in carbon tetrachloride was added dropwise with stirring. The finely divided, deep orange precipitate which separated weighed 0.405 g (0.760 mmol) and represented a 98% yield. Recrystallization from ethylene bromide gave small, brilliant, garnet red plates which melted with decomposition at 335° .¹³ Anal. Calcd for $C_{12}H_8TeI_2$: C, 27.01; H, 1.51. Found: C, 27.23; H, 1.64. The crystals are triclinic with space group symmetry $P\bar{1}$. There are two molecules in the unit cell for which $a = 8.807 \text{ \AA}$, $b = 8.182 \text{ \AA}$, $c = 12.001 \text{ \AA}$, $\alpha = 121.16^\circ$, $\beta = 101.63^\circ$, and $\gamma = 103.05^\circ$.¹⁴ The calculated density is 2.660 g cm^{-3} .

Method II. Dibenzotellurophene dichloride (0.598 g, 1.70 mmol) and potassium iodide (1.80 g, 10.8 mmol) were ground together in a mortar under 20 ml of 2-butanone for about 5 min. The dark red-brown solution was decanted through a glass filter and the solvent was permitted to evaporate. The product consisted mainly of deep red prismatic crystals of the diiodide along with some colorless crystals of potassium chloride and/or iodide. The latter were readily leached out by several rinsings with water, leaving 0.749 g (1.40 mmol, 83% yield) of the diiodide. These crystals are considerably larger and quite different in habit when compared to those crystallized from ethylene

bromide in method I. However, elemental analysis and X-ray diffraction data show that the products from the two methods are chemically identical.

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Registry No. 2-Biphenylmercury chloride, 10271-66-6; 2-aminobiphenyl, 90-41-5; mercury(II) chloride, 7487-94-7; tellurium tetrachloride, 10026-07-0; 2-bromobiphenyl, 2052-07-5; dibenzotellurophene dibromide, 55493-63-5; dibenzotellurophene diiodide, 1818-19-5; I, 244-98-4; II, 55493-62-4; III, 1818-18-4.

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- (8) The melting point of this compound has been reported in three previous communications: Courtot and Bastani¹ gave $165-167^\circ$; G. Wittig and W. Herwig, *Chem. Ber.*, **88**, 962 (1955), gave $161-162.5$, $162.5-163$, and $161-162^\circ$ for products prepared by three different methods; G. Bähr and R. Gelins, *ibid.*, **91**, 812 (1958), gave $166-167^\circ$.
- (9) The melting point of the dichloride is given as $333-335^\circ$ in ref 3, but ref 1 gives a decomposition temperature of about 200° .
- (10) Solutions of dibenzotellurophene become turbid when exposed to moist air, probably due to oxidation at the tellurium atom.
- (11) The melting point of dibenzotellurophene is given as $91-92^\circ$ in ref 1 and 93° in ref 2.
- (12) The decomposition temperature of the dibromide is given as $210-220^\circ$ in ref 1.
- (13) The melting point of the diiodide is given as $335-340^\circ$ in ref 3.
- (14) A complete structural study of dibenzotellurophene diiodide has been reported: J. D. McCullough, *Inorg. Chem.*, **14**, 1142 (1975).

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Five-Coordinate Complexes of Ruthenium(II) with Di(tertiary phosphines)

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Several six-coordinate complexes of ruthenium(II) with diphosphine ligands have been reported in the last years. The major work has been done by Chatt and Hayter,¹ who prepared complexes of the general formula $[RuX_2(diphosph)_2]$ and $[RuXY(diphosph)_2]$, where X and Y are anionic ligands such as halogenides, pseudohalogenides, hydrides, or σ -bonded alkyl and aryl groups. The di(tertiary phosphines) used are generally of the type $R_2P(CH_2)_nPR_2$ ($n = 1$, $R = C_6H_5$; $n = 2$, $R = CH_3, C_2H_5, C_6H_5$). No five-coordinate complexes of the type $[RuX(diphosph)_2]^+$ appear to be isolated. However, the chemical behaviors of some of these octahedral complexes, which can react with neutral ligands to give six-coordinate cationic derivatives of the type $[RuX(L)(diphosph)_2]^+$,¹⁻³ indirectly suggest the possibility of formation of five-coordinate cations $[RuX(diphosph)_2]^+$. With the aim of obtaining such unsaturated species, we have begun an investigation on the complexes formed by ruthenium(II) with a series of bidentate ligands.

We report here the first results, obtained with the di-

Table I. Electronic (Visible) Spectra

Compd	Absorption max, ^a cm ⁻¹ (ϵ_M for soln)
<i>trans</i> -[RuCl ₂ (dpp) ₂]	a: 20,900 (355), 26,000 sh b: 21,300, 24,400 sh
<i>trans</i> -[RuBr ₂ (dpp) ₂]	a: 19,900 (350), 25,800 sh ^b b: 20,000, 25,300 sh
[RuCl(dpp) ₂]PF ₆	a: 18,200 (640), 22,300 (2440), 28,000 sh b: 18,500, 22,200
[RuBr(dpp) ₂]PF ₆	a: 17,500 (640), 21,740 (2430), 27,000 sh b: 18,200, 21,750
[RuCl ₂ (dpp) _{1.5}] ₂	a: 14,700 (420), 21,700 (1440), 27,000 sh b: 14,900, 22,200

^a Key: a, 1,2-dichloroethane solution at 25°; b, Nujol mull.

^b Freshly prepared solution.

phosphines (C₆H₅)₂P(CH₂)_nP(C₆H₅)₂ (*n* = 3, 4), which have been examined in order to study the effect of the length of the alkyl chain on the stereochemistry of the formed complexes. Some five-coordinate complexes have been isolated and these results seem to be interesting in view of the relative scarcity of five-coordinate complexes of ruthenium(II) and in general of d⁶ metal ions.

Experimental Section

All reactions were carried out in an atmosphere of dry nitrogen or argon. The published procedures were used to prepare the ligands 1,3-bis(diphenylphosphino)propane⁴ (dpp) and 1,4-bis(diphenylphosphino)butane⁵ (dpb). The complexes [RuX₂(P(C₆H₅)₃)₃] were prepared by Wilkinson's method.⁶ Microanalyses were performed by Mr. L. Turiaco, Istituto di Chimica Analitica, University of Padua.

Infrared spectra were measured on Beckman IR 11 or Perkin-Elmer 457 instruments; visible spectra were determined on an Optica CF 4RNI spectrophotometer; molecular weights were determined using a Mechrolab osmometer at 37°; conductance data were obtained using a LKB conductance bridge; magnetic susceptibilities were measured by the standard Gouy method; gas absorption was carried out in a standard apparatus.

trans-[RuX₂(dpp)₂] (X = Cl, Br). K₂[RuCl₅(H₂O)] (1 mmol) in 2 ml of water was added to a hot ethanolic solution (50 ml) of dpp (3 mmol) and the resulting mixture was refluxed for about 3 hr. The complexes *trans*-[RuX₂(dpp)₂] precipitated from the reaction mixture and were recrystallized from CH₂Cl₂-tetrahydrofuran. The same products have also been obtained by treating benzene solutions of [RuX₂(P(C₆H₅)₃)₃] with an excess of dpp. Addition of tetrahydrofuran results in the precipitation of the products.

[RuX(dpp)₂]PF₆ (X = Cl, Br). The appropriate *trans*-[RuX₂(dpp)₂] complex (1 mmol) was refluxed in ethanol (100 ml) in the presence of NH₄PF₆ (2 mmol). The resulting red-violet precipitates were recrystallized from tetrahydrofuran-ethanol.

trans-[RuCl(CO)(dpp)₂]PF₆. A solution of [RuCl(dpp)₂]PF₆ in 1,2-C₂H₄Cl₂ was shaken for 1 hr under CO at 25° (1 atm); about 1 mol of CO per mol of Ru was absorbed and the solution became colorless. By evaporating the solvent, white crystals were obtained, which were recrystallized from ethanol.

Table II. Analytical Data and Physical Constants

Compd	Color	Mp, °C	Λ_M^a	Anal., %					
				C		H		X	
				Calcd	Found	Calcd	Found	Calcd	Found
<i>trans</i> -[RuCl ₂ (dpp) ₂] ^b	Pale-orange	205	2	65.00	64.40	5.25	5.08	7.12	7.28
<i>trans</i> -[RuBr ₂ (dpp) ₂]	Pink	200	87 ^c	59.68	60.28	4.78	4.95	14.44	14.88
[RuCl(dpp) ₂]PF ₆ ^d	Red-violet	270	86 ^e	58.62	58.54	4.74	4.70	3.20	3.23
[RuBr(dpp) ₂]PF ₆	Red-violet	260	91	56.35	56.10	4.55	4.36	6.94	7.11
[RuCl(CO)(dpp) ₂]PF ₆	White	285	95	59.57	59.02	4.61	4.66	3.12	3.39
[RuCl ₂ (dpp) _{1.5}] ₂	Light-green	186	1	62.14	60.89	5.22	5.11	8.74	9.17
[RuCl ₂ (CO)(dpp) _{1.5}] ₂ ^f	White	202	1	61.50	60.52	5.04	5.39	8.44	8.26

^a Molar conductance values for ca. 10⁻³ M nitromethane solutions. ^b Calcd: mol wt, 997. Found: mol wt, 960 (in 1,2-dichloroethane). ^c Aged solution. ^d Calcd: mol wt, 1106. Found: mol wt, 570 (in 1,2-dichloroethane). ^e Plot of Λ_e vs. the square root of the equivalent concentration, according to the Onsager equation $\Lambda_0 = \Lambda_e + A\sqrt{N}$, gives $\Lambda_0 = 91.0$ cm² ohm⁻¹ equiv⁻¹ and $A = 173$ cm² ohm⁻¹ equiv⁻¹ N^{-1/2}. ^f Calcd: mol wt, 1679. Found: mol wt, 1630 (in CHCl₃).

[RuCl₂(dpp)_{1.5}]₂. K₂[RuCl₅(H₂O)] (1 mmol) in 2 ml of water was added to a hot ethanolic solution (100 ml) of dpb (4 mmol) and the mixture was refluxed for about 3 hr. The light-green precipitate was washed with diethyl ether and extracted with chlorobenzene. The soluble fraction was repeatedly crystallized from chlorobenzene-diethyl ether. The same product has been obtained by treating a CH₂Cl₂ solution of [RuCl₂(P(C₆H₅)₃)₃] with an excess of dpb and by precipitating with diethyl ether.

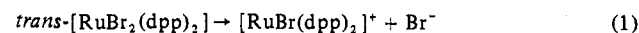
[RuCl₂(CO)(dpp)_{1.5}]₂. Finely powdered [RuCl₂(dpp)_{1.5}]₂ was kept under CO. One mole of CO per Ru atom is rapidly absorbed to give a yellow compound of formula [RuCl₂(CO)(dpp)_{1.5}]₂. Recrystallization from CH₂Cl₂-ethanol results in the formation of the corresponding white isomeric form.

Results and Discussion

The ligand 1,2-bis(diphenylphosphino)propane (dpp) reacts with K₂[RuX₅(H₂O)] in boiling ethanol-water mixture to yield the pink or orange diamagnetic *trans*-[RuX₂(dpp)₂]. The *trans* arrangement for [RuCl₂(dpp)₂] is supported by ir spectra, which show, in the Ru-Cl stretching region, a band (not present in the spectrum of the corresponding bromide) at 315 cm⁻¹, which can be assigned to terminal chlorines in mutually *trans* positions.⁷ Visible solid state spectra of the two *trans*-[RuX₂(dpp)₂] complexes are very similar to each other and have a weak absorption at about 27,000 cm⁻¹, with a shoulder at higher frequencies, which blends into much stronger bands in the uv (Table I). Similar absorption has been found in the spectra of other *trans*-[RuX₂(P)₄] chromophores and has been attributed to ¹A_{1g} → ¹E_g and ¹A_{1g} → ¹A_{2g} transitions in a tetragonally distorted octahedral field.^{1a,8}

In 1,2-dichloroethane solution, *trans*-[RuCl₂(dpp)₂] is monomeric and nonelectrolytic (Table II) and the visible spectrum, very similar to that in the solid state, indicates that the process of dissolution is not accompanied by structural changes.

In the case of the dibromo derivative, only for freshly prepared solutions in 1,2-dichloroethane, the visible spectrum resembles that in the solid state. On standing, the weak bands associated to *trans*-[RuBr₂(dpp)₂] disappear and the molar conductance of the solution rises to values typical of univalent electrolytes. If the pink *trans*-[RuBr₂(dpp)₂] is dissolved in ethanol, deep red solutions are rapidly formed, from which, by adding PF₆⁻ ions, the red-violet compound [RuBr(dpp)₂]PF₆ precipitates. Its visible spectrum in 1,2-dichloroethane is coincident with the spectrum of the aged solutions of *trans*-[RuBr₂(dpp)₂] in the same solvent, thus suggesting the occurrence in both solvents of the dissociation reaction 1. The dichloro derivative, which appears to be



undissociated in 1,2-dichloroethane (the visible spectrum remains unchanged after 1 week), yields an analogous [RuCl(dpp)₂]PF₆ when refluxed in ethanol containing PF₆⁻ ions. It should be noted that the dissociation of *trans*-

[RuBr₂(dpp)₂] in 1,2-dichloroethane is not repressed in the presence of excess Br⁻ ions and that addition of X⁻ ions to a 1,2-dichloroethane solution of [RuX(dpp)₂]⁺ produces no detectable changes in the visible spectra even over long periods of time (1 week). Moreover, the dichloro derivative slowly reacts in 1,2-dichloroethane with Br⁻ ions, giving the cation [RuBr(dpp)₂]⁺, probably through a [RuClBr(dpp)₂] intermediate.

Monomeric five-coordinate structures are assigned to the diamagnetic [RuX(dpp)₂]PF₆ complexes, on the basis of their physical properties. Polymeric structures containing halogen bridges are ruled out from ir spectra (in Nujol mull) of [RuCl(dpp)₂]PF₆, which exhibit a single band assignable as Ru-Cl stretching at 280 cm⁻¹, in the range expected for terminal chlorine groups.⁷ Both the chloro and the bromo derivatives behave as uni-univalent electrolytes in nitromethane solutions. The nature of a 1:1 electrolyte of [RuCl(dpp)₂]PF₆ has been confirmed by the method of Hayter.⁹ The slope of the plot of equivalent conductance against the square root of the equivalent concentration is in good agreement with the values calculated for 1:1 electrolytes. Also ionic weight measurements of [RuCl(dpp)₂]PF₆ in 1,2-dichloroethane are consistent with a monomeric structure (see Table II).

Electronic absorption spectra of [RuX(dpp)₂]⁺ complexes, which are very similar to each other and virtually identical both in the solid state and in solution, show two bands at 18,000 and 22,000 cm⁻¹, with a well-developed shoulder at higher frequencies. According to the simple crystal-field model for a five-coordinate d⁶ ion in a strong field, the observed diamagnetism of the [RuX(dpp)₂]PF₆ complexes suggests a more or less distorted square pyramidal geometry. If we assume for the [RuX(dpp)₂]⁺ chromophores a C_{4v} symmetry, the observed bands can be tentatively assigned, on the basis of the energy-levels diagram recently proposed by Sacconi,¹⁰ to the transitions ¹A₁ → ¹B₂, ¹A₁ → ¹E_a, and ¹A₁ → ¹A₂. A fourth transition (¹A₁ → ¹E_b), which is expected for such a symmetry, is probably masked by the charge-transfer bands.

It is interesting to note that attempts to obtain similar five-coordinate cations starting from the *trans*-[RuX₂((C₆H₅)₂P(CH₂)_nP(C₆H₅)₂)] (*n* = 1, 2) have been unsuccessful. Presumably the increased chain length of the dpp ligand causes a larger crowding in the plane of the six-coordinate *trans*-[RuX₂(dpp)₂] complexes, in such a manner that the elimination of one halide ion and the achievement of five coordination becomes favorable.

According to its coordinatively unsaturated nature, the compound [RuCl(dpp)₂]PF₆ readily adds in 1,2-dichloroethane 1 mol of CO per mol of complex, giving colorless solutions, from which the white diamagnetic solid [RuCl(CO)(dpp)₂]PF₆ may be recovered (ν_{CO} 1930 cm⁻¹). The carbonyl adduct can be assigned a *trans* configuration in view of the position of the Ru-Cl stretching frequency (300 cm⁻¹), which falls in the range expected for terminal chlorine *trans* to a CO group.^{2,7} When the alkyl chain of the diphosphine is lengthened, ruthenium(II) complexes of unusual stoichiometry are obtained. Thus, the interaction of excess of 1,4-bis(diphenylphosphino)butane (dpb) with boiling aqueous ethanolic solutions of K₂[RuCl₅(H₂O)] or with dichloromethane solutions of [RuCl₂(P(C₆H₅)₃)₃] produces green compounds, which, depending on the excess of diphosphine used, appear to contain from 3 to 1.5 mol of diphosphine per ruthenium atom.

Repeated crystallizations of the crude products from chlorobenzene-diethyl ether give finally a green complex of composition RuCl₂(dpb)_{1.5}. The complex is diamagnetic and nonelectrolytic in nitromethane and, on the basis of spectroscopic data, is assigned a binuclear five-coordinate structure with a diphosphine molecule bridging two ruthenium atoms.

The electronic spectrum of [RuCl₂(dpb)_{1.5}]₂ exhibits, both

in the solid state and in solution of 1,2-dichloroethane, two bands at 14,700 and 21,700 cm⁻¹, with a shoulder at higher frequencies. This spectrum shows strong similarities to those reported recently by James¹¹ for the five-coordinate [RuCl₂(P(C₆H₅)₃)₃] (two bands at 20,900 and 13,000 cm⁻¹) and is consistent with the presence of a five-coordinate ruthenium(II) with a P₃Cl₂ donor atom set. *Trans* arrangement of the chlorine atoms is indicated by the appearance of a single band at 320 cm⁻¹ in the Ru-Cl stretching region.

In agreement with a five-coordinate structure, [RuCl₂(dpb)_{1.5}]₂ readily takes up in the solid state 1 mol of CO per ruthenium atom, to give a yellow monocarbonyl derivative [RuCl₂(CO)(dpb)_{1.5}]₂ (ν_{CO} 1990 cm⁻¹; ν_{RuCl} 320 cm⁻¹). When this complex is crystallized from dichloromethane-ethanol, isomerization occurs and a white compound of the same composition is obtained (ν_{CO} 1950 cm⁻¹; ν_{RuCl} 290 and 255 cm⁻¹), in which the carbonyl group is probably *trans* to a chlorine.⁷

Molecular weight determinations for [RuCl₂(CO)(dpb)_{1.5}]₂ (in CHCl₃: found, 1630; calcd, 1679) indirectly confirm the dimeric nature of [RuCl₂(dpb)_{1.5}]₂ which cannot directly be proved, owing to the low solubility of the compound in the usual solvents.

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Registry No. *trans*-[RuCl₂(dpp)₂], 55669-28-8; *trans*-[RuBr₂(dpp)₂], 55669-29-9; [RuCl(dpp)₂]PF₆, 55669-31-3; [RuBr(dpp)₂]PF₆, 55669-33-5; [RuCl(CO)(dpp)₂]PF₆, 55669-35-7; [RuCl₂(dpb)_{1.5}]₂, 55669-36-8; [RuCl₂(CO)(dpb)_{1.5}]₂, 55669-37-9; K₂[RuCl₅(H₂O)], 14404-33-2; [RuCl₂(P(C₆H₅)₃)₃], 15529-49-4.

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Preparation, Properties, and Self-Exchange of Bis(dimethylchlorosilyl)mercury

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Bettler, Sendra, and Urry reported that bis(dimethylchlorosilyl)mercury could be formed at low temperatures, but decomposed above -79°. This is surprising since both bis(trimethylsilyl)mercury and bis(dichloromethylsilyl)mercury are thermally stable at room temperature.^{1,2} This anomalous behavior prompted us to prepare and study bis(dimethylchlorosilyl)mercury.

Reaction of dimethylchlorosilane with di-*tert*-butylmercury at 85° gave a light green solution from which white crystals precipitated on cooling. NMR and mass spectra unequivocally established this material as bis(dimethylchlorosilyl)mercury.³