An Improved Synthesis for Novel Hexaruthenium Cluster Compound Bearing Two Quadruply-Bridging CO Ligands: Synthesis, Characterization, and X-Ray Structural Analysis of $Ru_6(CO)_{12}(\mu_3\text{-}H)(\mu\text{-}CO)(\mu_4\text{-}\eta^2\text{-}CO)_2[\eta^5\text{-}C_5H_4(SiMe_3)]$

Yun Chi,[1,3] Chi-Jung Su,[1] Wen-Cheng Tseng,[1] Shie-Ming Peng,[2] and Gene-Hsiang Lee[2]

The hexaruthenium cluster compound $Ru_6(\mu_3\text{-}H)(CO)_{15}[C_5H_4(SiMe_3)]$ (2), possessing two $\mu_4\text{-}\eta^2\text{-}CO$ ligands and with the $Ru[C_5H_4(SiMe_3)]$ fragment located at the apex of the central tetrahedral framework, was prepared in low yield by refluxing a toluene solution of $C_5H_5(SiMe_3)$ with excess $Ru_3(CO)_{12}$. This unique complex was characterized by spectroscopic methods and by X-ray structural analysis. The possible mechanism leading to its formation is discussed.

INTRODUCTION

Transition-metal clusters bearing a quadruply-bridging CO ligand form a very interesting class of organometallic compounds [1]. The unusual $\mu_4\text{-}\eta^2\text{-}CO$ ligand on these cluster compounds is bound to three metal atoms through the carbon atom and to a fourth metal atom through its oxygen atom. This type of bonding is reminiscent of the metal–carbonyl interaction on the steps of metal and alloy surfaces [2]. In addition, because of

---

[1] Department of Chemistry, National Tsing Hua University, Hsinchu 30043, Republic of China.
[2] Department of Chemistry and Instrumentation Center, National Taiwan University, Taipei 10764, Taiwan, Republic of China.
[3] To whom correspondence should be addressed.

507

the existence of strong multisite interaction between CO and metal atoms, the C–O bond strength is weakened substantially [3]. As a result, it is implicated as a key intermediate for the reduction of CO on metal clusters [4] and the formation of carbido clusters through direct CO bond cleavage [5].

In attempts to investigate the chemistry of metal cluster compounds containing this type of μ_4-CO ligand, our research group has prepared a series of heterometallic carbonyl clusters with the formula $LMM'_3(\mu$-H) $(CO)_{12}(M = W, Mo; M' = Os, Ru; L = C_5H_5, C_5Me_5)$, via condensation of group 6 metal hydride complexes $LM(CO)_3H$ $(M = W, Mo; L = C_5H_5, C_5Me_5)$, with $Ru_3(CO)_{12}$ or with $Os_3(CO)_{10}(NCMe)_2$ [6]. The X-ray diffraction studies indicated that the Ru-containing cluster complexes show the unusual butterfly core arrangement bearing the μ_4-η^2-CO ligand, and that the corresponding Os-containing complexes exhibit only the tetra- hedral core arrangement with terminal and doubly bridging CO ligands. The formation of such diverse structures is attributed to the weaker metal– metal bond strength for the second-row ruthenium metal and the presence of stronger interligand repulsion between the CO ligands on the Ru atoms and between the CO ligands and the ancillary ligand (e.g., C_5H_5 or C_5Me_5) on the group 6 metal atom. Thus, these complexes offer the oppor- tunity to observe the structural perturbations arising from systematic changing of the transition metal atoms and the corresponding electronic and steric effects of the ancillary ligands [6].

In this paper, we wish to report the synthesis and characterization of a diruthenium complex $Ru_2(CO)_4[C_5H_4(SiMe_3)]_2$ (1) and a hexaruthe- nium cluster $Ru_6(\mu_3$-H$)(CO)_{15}[C_5H_4(SiMe_3)]$ (2) possessing two μ_4-η^2- CO ligands. The isolation of 1 strongly suggested the formation of hydride intermediate complex $[C_5H_4(SiMe_3)]Ru(CO)_2H$ [7], which then conden- sed with the excess $Ru_3(CO)_{12}$ present in the solution, as the principle pathway leading to the Ru_6 cluster compound 2. Thus, the basic synthetic methodology of 2 is akin to that of the cluster-building reactions using metal hydride complexes as building blocks [8].

RESULTS AND DISCUSSION

The trimethylsilylcyclopentadiene reacted with stoichiometric amount of ruthenium complex $Ru_3(CO)_{12}$ in refluxing toluene for 2 hours, affording a yellow orange trimethylsilylcyclopentadienyl complex $Ru_2(CO)_4$ $[C_5H_4(SiMe_3)]_2$ (1) in 92% yield. However, when the concentration of the ruthenium complex $Ru_3(CO)_{12}$ was increased threefold, the formation of a dark brown hexaruthenium cluster compound $Ru_6(\mu_3$-H$)(CO)_{15}$ $[C_5H_4(SiMe_3)]$ (2) was observed in 16% yield, together with the expected

dinuclear complex **1** in 61% yield and a ruthenium hydride cluster complex $Ru_4(CO)_{13}(\mu\text{-}H)_2$ generated from serendipitous hydrogenation of $Ru_3(CO)_{12}$ [9].

$$C_5H_5(SiMe_3) \ + \ Ru_3(CO)_{12}$$

$$[\eta^5\text{-}C_5H_4(SiMe_3)]Ru(CO)_2H$$

(1)   (2)
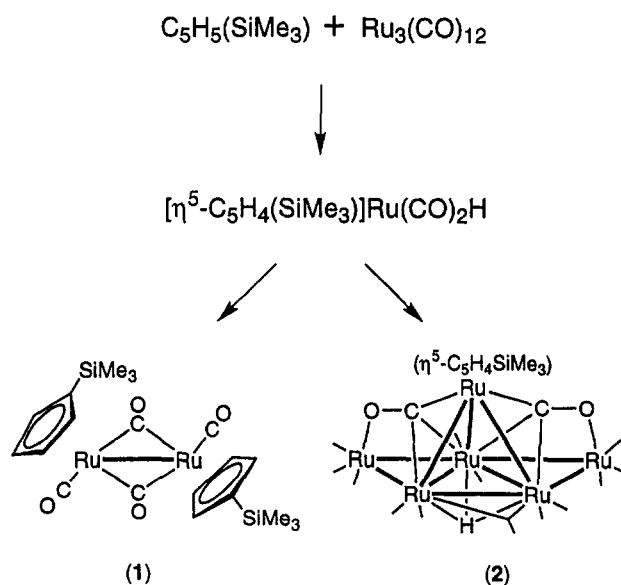
Compounds **1** and **2** were characterized by IR and NMR spectroscopies. As depicted in Scheme 1, compound **1** possesses two bridging CO ligands and two terminal CO ligands at the *trans* positions, which is also verified by a single crystal X-ray diffraction study [10]. Consistent with this assignment were the strong IR ν(CO) absorptions in the range 2005–1940 cm^{-1} due to the terminal CO ligands, and the absorption at 1787 cm^{-1} for the bridging CO ligands. The lack of more than one bridging CO absorption clearly confirms that complex **1** exists primarily as the *trans* bridged isomer as shown in Scheme 1. In addition, the ^{13}C NMR spectrum at 243 K gave a broad CO signal at δ 223.0. Upon lowering the temperature to 195 K, this signal completely collapses into the baseline. This fluxional behavior can be understood in terms of the rapid terminal-bridging CO exchange and the possible existence of several other unbridged isomers, which are analogous to that established for the related Fe_2 and Ru_2 complexes [11].

For the Ru_6 cluster complex **2**, the ^{1}H NMR spectrum showed three peaks at δ 5.49, 5.38, and 0.66 in a 2:2:9 ratio due to the $C_5H_4(SiMe_3)$ ligand, and a downfield signal at δ −24.59 attributed to a bridging hydride ligand. The FAB mass spectrum gave a molecular ion peak corresponding

to $C_{25}H_{15}O_{15}Si_1Ru_6$, while the IR data in C_6H_{12} solution exhibited the expected terminal CO absorptions in the range 2092–1969 cm^{-1} and edge-bridging CO signal at 1870 cm^{-1} and a broad absorption at the lower frequency region at near 1457 cm^{-1}, showing the presence of novel μ_4-η^2-CO ligands.

Complex **2** was further characterized by single-crystal X-ray diffraction study. Our result suggests that it possesses two crystallographically distinct, but structurally similar molecules. A perspective view of one of these molecules is depicted in Fig. 1. The molecule consists of a tetrahedral Ru$_4$ arrangement with two basal Ru–Ru edge each coordinated by a pendant Ru(CO)$_3$, while the third Ru–Ru edge is linked to a symmetrically bridging CO ligand. The C$_5$H$_4$(SiMe$_3$) ligand resides at the apical position of the
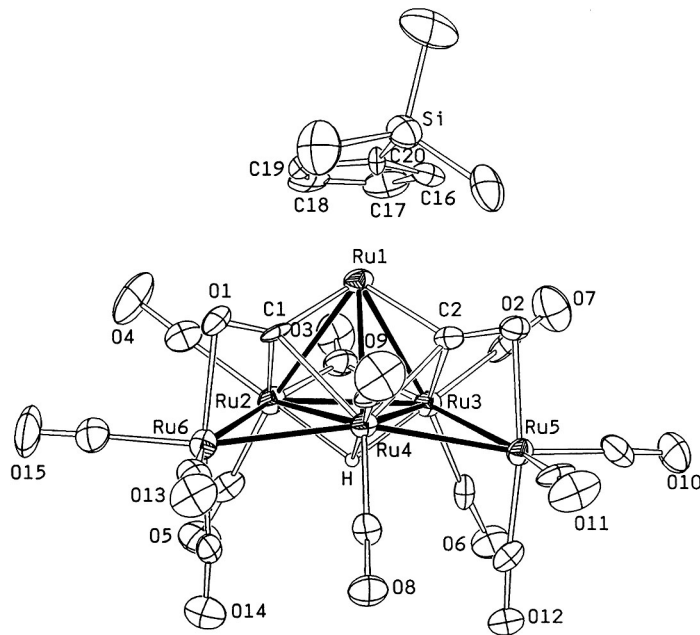

Fig. 1. Molecular structure of Ru$_6(\mu_3$-H)(CO)$_{15}$[C$_5$H$_4$(SiMe$_3$)] (**2**) showing the atomic labeling scheme and the thermal ellipsoids at 30% probability level. Selected bond lengths (Å): Ru(1)–Ru(2) = 2.801(2), Ru(1)–Ru(3) = 2.794(2), Ru(1)–Ru(4) = 2.764(2), Ru(2)–Ru(3) = 2.728(2), Ru(2)–Ru(4) = 2.877(2), Ru(2)–Ru(6) = 2.768(2), Ru(3)–Ru(4) = 2.871(2), Ru(3)–Ru(5) = 2.753(2), Ru(4)–Ru(5) = 2.815(2), Ru(4)–Ru(6) = 2.814(2), Ru(1)–C(1) = 1.92(2), Ru(2)–C(1) = 2.32(1), Ru(4)–C(1) = 2.23(1), Ru(6)–C(1) = 2.33(1), Ru(6)–O(1) = 2.16(1), Ru(1)—C(2) = 1.93(2), Ru(3)–C(2) = 2.29(2), Ru(4)–C(2) = 2.22(2), Ru(5)–C(2) = 2.31(2), Ru(5)–O(2) = 2.15(1), Ru(2)–C(3) = 2.13(2), Ru(3)–C(3) = 2.15(2), C(1)–O(1) = 1.25(2), C(2)–O(2) = 1.23(2), Ru(2)–H = 1.78(13), Ru(3)–H = 1.73(13), Ru(4)–H = 2.03(12).

central tetrahedron. The hydride ligand is located at the basal Ru_3 triangle, as revealed by the difference Fourier synthesis. The Ru atoms on this triangular plane each possesses two terminal CO ligands, while the other two pendent Ru atoms are each coordinated by three terminal CO ligands and by the oxygen atom of a unique μ_4-η^2-CO ligand. The bonding parameters associated with the μ_4-η^2-CO ligands are similar to those of the tetranuclear butterfly cluster complexes mentioned earlier [1, 3, 6] and other polynuclear metal cluster compounds bearing the μ_4-CO ligand [12].

In addition, the structure of 2 appears to possess an idealized mirror plane which bisects the Ru(2)–Ru(3) edge and passes through the bridging CO ligand CO(3), the Ru(1) and Ru(4) atoms, and two terminal CO ligands on the Ru(4) atom. This cluster framework is essentially identical to that of the methylcyclopentadienyl complex $Ru_6(\mu_3$-H)(CO)$_{15}$ (C_5H_4Me) (3) prepared from the reaction of $Ru_3(CO)_{12}$ and cyclohexene [13], the Ru_6 arene complexes $Ru_6(CO)_{15}(C_6H_3Me_3)$ (4) [14] or Ru_6 $(CO)_{15}(C_6Me_6)$ [15] and the $RhRu_5$ heterometallic cluster $RhRu_5(CO)_{15}$ (C_5Me_5) (5) [16] as shown in Scheme 2. In agreement with the symmetrical structure, the ^{13}C NMR spectrum at room temperature exhibited nine CO signals at δ 280.4, 212.3, 205.2, 199.8, 191.5 (J_{CH} = 12 Hz), 190.2, 190.0 (J_{CH} = 12 Hz), 185.4 and 185.2 in a ratio 2:1:2:1:2:2:1:2:2. The partial assignments of CO ligands can be achieved by comparing their chemical shift, integration intensity and the J_{CH} coupling constant. Thus the signal at δ 280.4 is due to the unique μ_4-η^2-CO ligands [17]. The second most downfield signal at δ 212.3 is assigned to the bridging CO ligand, while the signals at δ 191.5 and 190.0 with ratio 2 : 1, which show the characteristic J_{CH} coupling, are obviously coming from the CO ligands located at the equatorial positions, opposite to the bridging hydride ligand. Thus, the complex 2 represents an example among these hexametallic complexes 2-5, for which the ^{13}C NMR spectrum was reported and assigned.

(3)   (4)   (5)
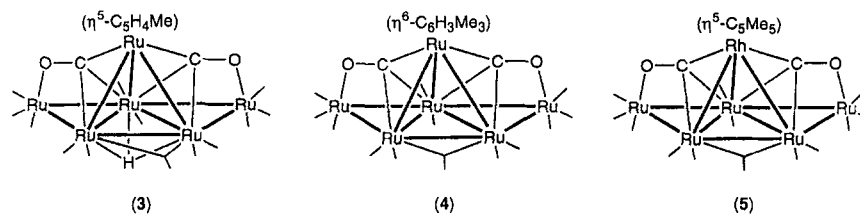
CONCLUSIONS

We present here the facile synthesis of the Ru_2 complex 1 and the Ru_6 cluster 2 which contains two μ_4-η^2-CO ligands. The formation of 1

strongly suggests that the mononuclear hydride fragment $[C_5H_4(SiMe_3)]$ $Ru(CO)_2H$ was the initial product, as the analogous hydride complexes has been documented as the precursor to other Ru_2 complexes through oxidative dehydrogenation reaction. The condensation between the hydride complex $[C_5H_4(SiMe_3)]Ru(CO)_2H$ and $Ru_3(CO)_{12}$ became progressively effective in the presence of excess $Ru_3(CO)_{12}$, which then leads to the isolation of the Ru_6 cluster complex 2. Our experimental observation agrees with the recent report that treatment of $Ru_3(CO)_{12}$ with $Ni(Cp)_2$, which is used as the source for Cp ligand, or with C_5Me_5H lead to the formation of the similar hexanuclear complexes, $Ru_6(\mu_3\text{-}H)(CO)_{15}Cp$ or $Ru_6(\mu_3\text{-}H)$ $(CO)_{15}(C_5Me_5)$ in low yields, respectively [18]. However, in this article, the authors have failed to identify the ruthenium hydride complex as the reaction intermediate. On the other hand, the exact role of the $C_5H_4(SiMe_3)$ ligand with respect to the Cp or the C_5Me_5 ligand in these reactions is apparently due to the greater steric effect [19], which makes the ruthenium hydride complex $[C_5H_4(SiMe_3)]Ru(CO)_2H$ more stable and effective to react with the excess $Ru_3(CO)_{12}$ present in the solution. Attempts to expand this cluster-building reaction by using other trimethyl-silyl substituted cyclopentadiene molecule have encountered many set-backs; thus, the condensation of $C_5H_4(SiMe_3)_2$ with $Ru_3(CO)_{12}$ under similar conditions failed to generate the corresponding bis-trimethylsilyl-cyclopentadienyl cluster $Ru_6(\mu_3\text{-}H)(CO)_{15}[C_5H_3(SiMe_3)_2]$. The Ru_2 complex $Ru_2(CO)_4[C_5H_3(SiMe_3)_2]_2$ appeared to be the only product isolated in all attempts.

The crystal structure of 2 is noteworthy. The existence of two $\mu_4\text{-}\eta^2$-CO ligands in 2 assists the stabilization of the bis-edge bridged tetrahedral cluster framework. This structure is in contrast to the bicapped tetrahedral arrangement as observed in $Os_6(CO)_{18}$ (6) [20], $Os_6(CO)_{15}(C_6H_6)$ (7) [21] or other related osmium cluster complexes [22], where the cluster shows a higher tendency to maintain the maximum number of Os–Os bonds, but fails to undergo rearrangement from bicapped tetrahedral to bis-edge bridged tetrahedral arrangement through the insertion of two CO ligands into the Os–Os bonds (Scheme 3). We propose that the difference in the cluster core arrangement is due to a subtle balance between the metal–metal bond energy vs. the stabilization for formation of the $\mu_4\text{-}\eta^2$-CO interaction. The Os–Os bond energy in the third-row cluster complexes 6 or 7 is much greater [23]; thus, the cluster adopts the bicapped tetrahedral geometry. In contrast, the Ru–Ru bond energy in the Ru_6 complexes 2, 3, and 4 or $RhRu_5$ complex 5 is much weaker with respect to the energy gained from the formation of the $\mu_4\text{-}\eta^2$-CO ligands, because they consist of the second-row transition metal elements. Thus, the steric repulsion between the coordinated ligands induces the insertion of CO ligand

through cleavage of Ru–Ru bond, and favors the generation of a more open *bis*-edge bridged tetrahedral cluster framework.

(6)   (7)
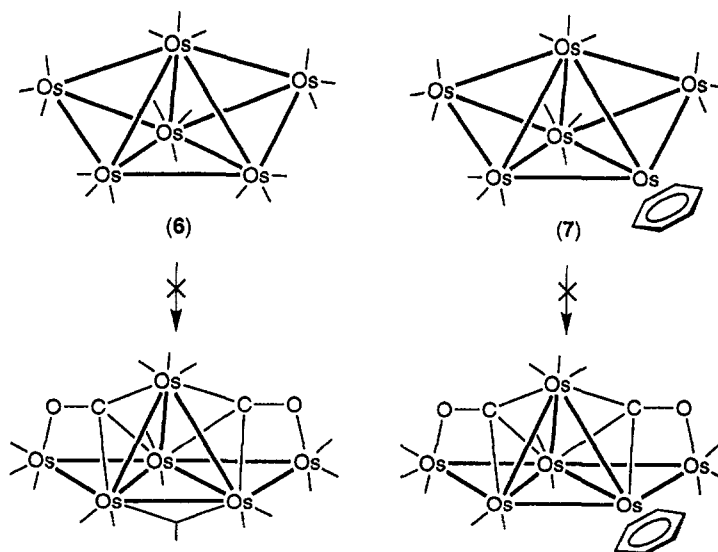
EXPERIMENTAL PROCEDURE

General Information and Materials

Infrared spectra were recorded on a Perkin-Elmer 2000 FT-IR spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AM-400 instrument. ^{1}H and ^{13}C NMR chemical shifts are quoted with respect to internal standard tetramethylsilane. Mass spectra were obtained on a JEOL-HX110 instrument operating in fast atom bombardment (FAB) mode. All reactions were performed under nitrogen atmosphere using deoxygenated solvents dried with an appropriate reagent. Reactions were monitored by analytical thin-layer chromatography (5735 Kieselgel 60 F_{254}, E. Merck) and the products were separated on commercially available preparative thin-layer chromatographic plates (Kieselgel 60 F_{254}, E. Merck). Elemental analyses were carried out at the regional instrumentation center at National Cheng Kung University, Tainan, Taiwan.

Reaction of $C_5H_5(SiMe_3)$ with $Ru_3(CO)_{12}$

A toluene solution (50 mL) of $C_5H_5(SiMe_3)$ (50 μL, 0.31 mmol) and $Ru_3(CO)_{12}$ (200 mg, 0.31 mmol) was heated to reflux for 2 hours, during

which the color changed from orange to dark brown. After removal of
the solvent *in vacuo*, the residue was redissolved in a minimum amount
of CH_2Cl_2 and separated by thin layer chromatography (CH_2Cl_2:
hexane = 1:4), giving 85 mg of orange yellow $Ru_2(CO)_4[C_5H_4(SiMe_3)]_2$
(1, 0.144 mmol, 92%).

Reaction of $C_5H_5(SiMe_3)$ with Excess $Ru_3(CO)_{12}$

A toluene solution (50 mL) of $C_5H_5(SiMe_3)$ (17 μL, 0.104 mmol) and
$Ru_3(CO)_{12}$ (200 mg, 0.313 mmol) was heated to reflux for 2 hours, during
which time the color changed from orange to dark brown. After removal
of the solvent *in vacuo*, the residue was redissolved in a minimum amount
of CH_2Cl_2 and separated by thin layer chromatography (CH_2Cl_2:
hexane = 1:4), giving 14 mg of orange $Ru_4(\mu\text{-}H)_2(CO)_{13}$ (0.018 mmol),
20 mg of black $Ru_6(\mu_3\text{-}H)(CO)_{15}[C_5H_4(SiMe_3)]$ (2, 0.016 mmol, 16%)
and 19 mg of orange yellow $Ru_2(CO)_4[C_5H_4(SiMe_3)]_2$ (1, 0.032 mmol,
61%). Crystals of 2 suitable for X-ray diffraction study were obtained from
dichloromethane/heptane at room temperature.

Spectral data for 1: MS (FAB, ^{102}Ru), m/z 590 (M +). IR(C_6H_{12}):
ν(CO), 2017 (w), 2005 (s), 1969 (vs), 1961 (vs), 1940 (vs), 1787 (vs) cm^{-1};
^1H NMR (CDCl$_3$, 294 K): δ 5.34 (t, J_{HH} = 1.8 Hz, 2H), 5.07 (t, J_{HH} = 1.8
Hz, 2H), 0.28 (s, 9H). ^{13}C NMR (CDCl$_3$, 243 K): δ 223.0 (broad, CO),
96.7 (2C, CH), 94.6 (CSiMe$_3$), 93.9 (2C, CH) 0.40 (3Me). Elemental
analysis for $C_{20}H_{26}O_4Si_2Ru_2$: Calc.: C, 40.80; H, 4.45. Found: C, 40.70;
H, 3.39.

Spectral data for 2: MS (FAB, ^{102}Ru), m/z 1169 (M$^+$). IR(C_6H_{12}):
ν(CO), 2092 (m), 2071 (vs), 2035 (vs), 2025 (m), 2013 (s), 2003 (vw), 1985
(w), 1969 (w), 1870 (w) cm^{-1}; ^1H NMR (CDCl$_3$, 294 K): δ 5.49 (s, 2H),
5.38 (s, 2H), 0.66 (s, 9H), −24.59 (s, 1H). ^{13}C NMR (CDCl$_3$, 294 K): δ
280.4 (2C, μ_4-CO), 212.3, 205.2 (2C), 199.8, 191.5 (d, 2C, J_{CH} = 12 Hz),
190.2 (2C), 190.0 (d, J_{CH} = 12 Hz), 185.4 (2C), 185.2 (2C), 100.7
(CSiMe$_3$), 99.8 (2C, CH), 92.5 (2C, CH) −0.72 (3Me). Elemental analysis
for $C_{23}H_{14}O_{15}SiRu_6$: Calc.: C, 23.72; H, 1.21. Found: C, 23.62; H, 1.18.

X-ray Crystallography

The X-ray diffraction study was carried out on a Nonius CAD-4 dif-
fractometer. Lattice parameters were determined from 25 randomly-
selected high-angle reflections. Three standard reflections were monitored
every 3600 seconds. No significant change in intensities, due to crystal
decay, was observed over the course of data collection. Intensities of the
diffraction signals were corrected for Lorentz, polarization, and absorption

Table I. Crystal Data for the X-ray Diffraction Studies of 2

Formula = $C_{23}H_{14}O_{15}SiRu_6$	Mol. wt. = 1164.86
Crystal system, Space group	Triclinic, $P-1$
$a = 12.830(3)$ Å	$b = 15.782(3)$ Å
$c = 17.108(3)$ Å	$\alpha = 75.61(2)°$
$\beta = 79.40(2)$ Å	$\gamma = 89.96(2)°$
Vol. = 3295(1) Å^3	$Z = 4$
$D_c = 2.349$ g/cm^3	$F(000) = 2200$
2θ (max)	45°
hkl ranges	$-13, 13, 0\ 16, -17\ 18$
Crystal size, mm.	$0.03 \times 0.22 \times 0.22$
μ(Mo-K$_\alpha$) cm^{-1}	27.31
Transmission: max, min.	1.000, 0.919
No. of unique data	8602
Data with $I > 2\sigma(I)$	4763
No. of atoms and parameters	118, 820
Weighting scheme	$w^{-1} = \sigma^2(F_o) + |g|\ F_o^2$
Weight modifier	0.0001
Maximum Δ/σ ratio	0.022
R_F; R_w	0.042; 0.039
GOF	1.28
D-map, max/min, e/Å^3	0.82/-0.77

Table II. Atomic Coordinates and Isotropic Displacement Coefficients for Complex 2; e.s.d.s Refer to the Last Digit Printed

	x	y	z	Beq^a
Ru1	0.73806(11)	0.99873(8)	0.25669(9)	3.03(7)
Ru2	0.52427(11)	1.00940(8)	0.24198(9)	2.96(7)
Ru3	0.67693(11)	1.02929(8)	0.10392(8)	2.76(7)
Ru4	0.64386(10)	0.86039(8)	0.21886(8)	2.63(6)
Ru5	0.77159(11)	0.88626(8)	0.06160(9)	2.92(7)
Ru6	0.46274(11)	0.84451(9)	0.34348(9)	3.36(7)
Si	0.9618(4)	0.9128(4)	0.3605(3)	4.7(3)
C1	0.6250(12)	0.9199(9)	0.3256(9)	2.8(8)
C2	0.7931(12)	0.9429(9)	0.1704(9)	3.1(8)
C3	0.5962(13)	1.1266(10)	0.1571(11)	4.0(10)
C4	0.4803(14)	1.0617(10)	0.3324(11)	4.2(9)
C5	0.3872(14)	1.0237(9)	0.2134(10)	3.7(9)
C6	0.6209(12)	1.0480(8)	0.0057(9)	2.9(8)
C7	0.7940(15)	1.1003(10)	0.0475(9)	4.3(10)
C8	0.5680(12)	0.7803(9)	0.1831(10)	3.3(8)
C9	0.7156(15)	0.7645(11)	0.2770(11)	4.6(10)

Table II. *(Continued)*

	x	y	z	Beq^a
C10	0.8569(13)	0.9400(11)	−0.0417(12)	4.6(11)
C11	0.8224(14)	0.7706(10)	0.0704(11)	4.0(10)
C12	0.6626(13)	0.8607(9)	0.0125(9)	3.1(8)
C13	0.4612(14)	0.7213(11)	0.3866(11)	4.7(10)
C14	0.3685(12)	0.8307(9)	0.2750(10)	3.4(9)
C15	0.3517(14)	0.8616(11)	0.4316(10)	4.6(10)
C16	0.8949(13)	1.0654(11)	0.2418(11)	4.2(10)
C17	0.8221(16)	1.1295(10)	0.2399(12)	5.4(11)
C18	0.7515(14)	1.1089(11)	0.3144(12)	5.0(11)
C19	0.7820(13)	1.0307(12)	0.3663(10)	4.3(10)
C20	0.8753(12)	1.0046(10)	0.3206(9)	3.3(8)
C21	1.0736(18)	0.9653(15)	0.3857(17)	9.3(18)
C22	1.0113(15)	0.8527(14)	0.2827(13)	6.8(13)
C23	0.8806(20)	0.8413(15)	0.4517(14)	8.7(15)
O1	0.5868(9)	0.8833(7)	0.3989(6)	3.8(6)
O2	0.8771(8)	0.9253(6)	0.1317(7)	3.5(6)
O3	0.5947(10)	1.2007(7)	0.1455(8)	6.0(8)
O4	0.4561(13)	1.0934(9)	0.3840(8)	8.2(10)
O5	0.3053(9)	1.0322(8)	0.1971(8)	5.7(7)
O6	0.5934(9)	1.0616(7)	−0.0554(7)	4.6(6)
O7	0.8642(10)	1.1473(8)	0.0119(9)	6.7(8)
O8	0.5247(10)	0.7253(7)	0.1662(8)	5.7(7)
O9	0.7574(12)	0.7098(8)	0.3086(9)	7.8(9)
O10	0.9032(10)	0.9753(9)	−0.1059(8)	6.9(8)
O11	0.8473(12)	0.7027(8)	0.0755(9)	7.2(9)
O12	0.5990(9)	0.8451(7)	−0.0229(7)	4.4(6)
O13	0.4643(11)	0.6480(7)	0.4077(8)	6.7(8)
O14	0.3041(10)	0.8189(8)	0.2402(8)	6.2(8)
O15	0.2862(11)	0.8710(11)	0.4814(9)	8.7(10)
Ru1′	0.24109(10)	0.35668(8)	0.25023(8)	2.61(6)
Ru2′	0.17647(10)	0.41358(8)	0.09963(8)	2.51(6)
Ru3′	0.02634(10)	0.35417(8)	0.23871(8)	2.72(6)
Ru4′	0.14455(10)	0.51504(8)	0.21940(8)	2.62(6)
Ru5′	−0.03350(11)	0.45776(9)	0.34709(9)	3.49(7)
Ru6′	0.26902(11)	0.57966(8)	0.06144(8)	2.85(7)
Si′	0.4526(4)	0.3839(4)	0.3640(3)	5.0(3)
C1′	0.2919(12)	0.4607(9)	0.1669(9)	3.1(8)
C2′	0.1266(11)	0.3954(10)	0.3217(9)	2.9(8)
C3′	0.0967(14)	0.2884(10)	0.1489(10)	3.7(9)
C4′	0.2951(12)	0.3745(10)	0.0390(10)	3.8(9)
C5′	0.1182(13)	0.4502(9)	0.0026(9)	2.9(8)
C6′	−0.1113(13)	0.3603(10)	0.2110(10)	3.4(9)
C7′	−0.0136(12)	0.2530(11)	0.3223(10)	3.7(9)

Table II. (Continued)

	x	y	z	Beq^a
C8'	0.0642(13)	0.6143(10)	0.1872(10)	3.9(9)
C9'	0.2131(14)	0.5758(10)	0.2747(11)	4.2(10)
C10'	−0.1399(13)	0.3898(13)	0.4320(11)	4.9(11)
C11'	−0.0256(14)	0.5539(12)	0.3950(11)	4.8(11)
C12'	−0.1308(13)	0.5116(10)	0.2825(11)	4.5(10)
C13'	0.3190(14)	0.6885(10)	0.0697(11)	4.6(10)
C14'	0.1552(13)	0.6331(9)	0.0136(9)	3.2(9)
C15'	0.3463(13)	0.5821(10)	−0.0414(10)	4.2(9)
C16'	0.2834(13)	0.2585(10)	0.3565(10)	4.2(9)
C17'	0.2551(13)	0.2128(10)	0.3013(12)	4.9(10)
C18'	0.3266(14)	0.2393(10)	0.2271(12)	4.7(10)
C19'	0.3999(11)	0.3022(9)	0.2368(10)	3.1(8)
C20'	0.3762(13)	0.3149(10)	0.3170(10)	3.7(9)
C21'	0.5622(17)	0.3168(16)	0.3970(14)	8.1(15)
C22'	0.3625(18)	0.4024(15)	0.4529(13)	7.7(15)
C23'	0.5084(17)	0.4893(13)	0.2891(14)	7.1(14)
O1'	0.3754(8)	0.5037(6)	0.1273(6)	3.6(5)
O2'	0.0920(8)	0.3875(6)	0.3969(6)	3.5(6)
O3'	0.0946(10)	0.2189(7)	0.1361(8)	5.8(7)
O4'	0.3658(10)	0.3504(8)	0.0038(8)	6.8(8)
O5'	0.0910(9)	0.4707(7)	−0.0587(6)	4.1(6)
O6'	−0.1947(9)	0.3559(8)	0.1992(8)	5.9(8)
O7'	−0.0394(10)	0.1911(8)	0.3754(7)	6.2(7)
O8'	0.0204(10)	0.6772(7)	0.1717(8)	6.0(8)
O9'	0.2579(10)	0.6155(8)	0.3082(9)	6.5(8)
O10'	−0.2027(11)	0.3503(10)	0.4841(9)	8.6(10)
O11'	−0.0217(13)	0.6126(9)	0.4205(9)	8.4(10)
O12'	−0.1946(10)	0.5439(8)	0.2465(8)	6.5(8)
O13'	0.3506(13)	0.7546(8)	0.0726(10)	9.0(11)
O14'	0.0935(9)	0.6660(7)	−0.0193(7)	4.7(7)
O15'	0.3924(11)	0.5814(9)	−0.1079(8)	7.6(9)

^{a} Beq is the mean of the principal axes of the thermal ellipsoid.

effects (ψ scans). The structure was solved by using the NRCC-SDP-VAX package. All the non-hydrogen atoms had anisotropic temperature factors, and the hydrogen atoms were placed at the idealized positions with $U_H = U_C + 0.1$. The selective crystallographic refinement parameters are summarized in Table I, while the atomic positional parameters are given in Table II. Tables of nonessential bond distances and angles, tables of anisotropic thermal parameters, and listings of the observed and calculated structural factors are available from the author (Y.C.).

ACKNOWLEDGMENTS

REFERENCES

1. (a) C. P. Horwitz and D. F. Shriver (1984). *Adv. Organomet. Chem.* **23**, 219; (b) R. D. Adams, J. E. Babin, and M. Tasi (1987). *Angew. Chem. Int. Ed. Engl.* **26**, 685.
2. E. Sappa, A. Tiripicchio, A. J. Carty, and G. E. Toogood (1987). *Prog. Inorg. Chem.* **35**, 437.
3. (a) K. Kwek, N. J. Taylor, and A. J. Carty (1984). *J. Am. Chem. Soc.* **106**, 4636; (b) J. S. Field, R. J. Haines, and F. J. Mulla (1990). *J. Organomet. Chem.* **389**, 227; (c) R. D. Adams, J. E. Babin, and M. Tasi (1988). *Inorg. Chem.* **27**, 2618; (d) P. Brun, G. M. Dawkins, M. Green, A. D. Miles, A. G. Orpen, and F. G. A. Stone (1982). *J. Chem. Soc., Chem. Commun.* 926; (e) B. F. G. Johnson, J. Lewis, M. McPartlin, M. Pearsall, and A. Sironi (1984). *J. Chem. Soc., Chem. Commun.* 1089.
4. (a) E. L. Muetterties and J. Stein (1979). *Chem. Rev.* **79**, 479; (b) K. H. Whitmire and D. F. Shriver (1980). *J. Am. Chem. Soc.* **102**, 1456.
5. (a) C. P. Horwitz and D. F. Shriver (1985). *J. Am. Chem. Soc.* **107**, 8147; (b) P. J. Bailey, B. F. G. Johnson, and J. Lewis (1994). *Inorg. Chim. Acta* **227**, 197; (c) C.-J. Su, P.-C. Su, Y. Chi, S.-M. Peng, and G.-H. Lee (1996). *J. Am. Chem. Soc.* **118**, 3289.
6. (a) Y. Chi, F.-J. Wu, B.-J. Liu, C.-C. Wang, and S.-L. Wang (1989). *J. Chem. Soc., Chem. Commun.* 873; (b) S.-M. Peng, G.-H. Lee, Y. Chi, C.-L. Peng, and L.-S. Hwang (1989). *J. Organomet. Chem.* **371**, 197; (c) Y. Chi, C.-J. Su, L. J. Farrugia, S.-M. Peng, and G.-H. Lee (1994). *Organometallics* **13**, 4167.
7. (a) A. P. Humphries and S. A. R. Knox (1973). *J. Chem. Soc., Chem. Commun.* 326; (b) A. P. Humphries and S. A. R. Knox (1975). *J. Chem. Soc., Dalton Trans.* 1710; (c) S. A. R. Knox (1984). *Pure Appl. Chem.* **56**, 81.
8. (a) J. R. Shapley, G. A. Pearson, M. Tachikawa, G. E. Schmidt, M. R. Churchill, and F. J. Hollander (1977). *J. Am. Chem. Soc.* **99**, 8064; (b) M. R. Churchill, F. J. Hollander, J. R. Shapley, and D. F. Foose (1978). *J. Chem. Soc., Chem. Commun.* 534; (c) M. R. Churchill, F. J. Hollander, R. A. Lashewycz, G. A. Pearson, and J. R. Shapley (1981). *J. Am. Chem. Soc.* **103**, 2430.
9. C. R. Eady, B. F. G. Johnson, and J. Lewis (1977). *J. Chem. Soc., Dalton Trans.* 477.
10. Crystal data: $C_{20}H_{26}O_4Si_2Ru_2$, $M = 588.73$, monoclinic, space group $P\,2_1/c$, $a = 7.809(1)$, $b = 9.7965(9)$, $c = 15.247(2)$ Å, $\beta = 94.47(1)°$, $V = 1162.8(3)$ Å^3, $Z = 4$, $\rho_{calc.} = 1.681$ gcm^{-3}, $F(000) = 583$, $\lambda(Mo-K\alpha) = 0.7107$ Å, $T = 298°$ K, $\mu = 13.97$ cm^{-1}. $R_F = 0.043$, $R_w = 0.043$ and G.O.F. $= 1.22$.
11. (a) L. J. Farrugia and L. Mustoo (1992). *Organometallics* **11**, 2941; (b) R. D. Adams and F. A. Cotton (1973). *J. Am. Chem. Soc.* **95**, 6589.
12. (a) Y. Chi, S.-H. Chuang, L.-K. Liu, and Y.-S. Wen (1991). *Organometallics* **10**, 2485; (b) J.-C. Wang, R.-C. Lin, Y. Chi, S.-M. Peng, and G.-H. Lee (1993). *Organometallics* **12**, 4061; (c) J.-C. Wang, Y. Chi, F.-H. Tu, S.-G. Shyu, S.-M. Peng, and G.-H. Lee (1994). *J. Organomet. Chem.* **481**, 143; (d) C.-J. Su, Y. Chi, S.-M. Peng, and G.-H. Lee (1995). *Organometallics* **14**, 4286.
13. S. L. Ingham, B. F. G. Johnson, C. M. Martin, and D. Parker (1995). *J. Chem. Soc., Chem. Commun.* 159.

14. C. E. Anson, P. J. Bailey, G. Conole, B. F. G. Johnson, J. Lewis, M. McPartlin, and H. R. Powell (1989). *J. Chem. Soc., Chem. Commun.* 442.

15. A. J. Blake, P. J. Dyson, S. L. Ingham, B. F. G. Johnson, and C. M. Martin (1995). *Inorg. Chem. Acta* **240**, 29.

16. J. E. Davies, S. Nahar, P. R. Raithby, and G. P. Shields (1997). *J. Chem. Soc., Dalton Trans.* 13.

17. J. Wang, M. Sabat, C. P. Horwitz, and D. F. Shriver (1988). *Inorg. Chem.* **27**, 552.

18. E. Kolehmainen, K. Rissanen, K. Laihia, Z. A. Kerzina, M. I. Rybinskaya, and M. Nieger (1996). *J. Organomet. Chem.* **524**, 219.

19. (a) M. F. Lappert, C. J. Pickett, P. I. Riley, and P. I. W. Yarrow (1981). *J. Chem. Soc., Dalton Trans.* 805; (b) A. Antinolo, J. M. de Ilarduya, A. Otero, P. Royo, A. M. M. Lanfredi, and A. Tiropicchio (1988). *J. Chem. Soc., Dalton Trans.* 2685; (c) J. Okuda (1992). *Top. Curr. Chem.* **160**, 97.

20. R. Mason, K. M. Thomas, and D. M. P. Mingos (1973). *J. Am. Chem. Soc.* **95**, 3802.

21. J. Lewis, C. K. Li, C. A. Morewood, M. C. Ramirez De Arellano, P. R. Raithby, and W. T. Wong (1994). *J. Chem. Soc., Dalton Trans.* 2159.

22. (a) B. F. G. Johnson, R. A. Kamarudin, F. J. Lahoz, J. Lewis, and P. R. Raithby (1988). *J. Chem. Soc., Dalton Trans.* 1205; (b) R. K. Henderson, P. A. Jackson, B. F. G. Johnson, J. Lewis, and P. R. Raithby (1992). *Inorg. Chim. Acta* **198–200**, 393; (c) J. Lewis, C.-K. Li, C. A. Morewood, M. C. Ramirez de Arellano, P. R. Raithby, and W.-T. Wong (1994). *J. Chem. Soc., Dalton Trans.* 2159.

23. D. Michael, P. Mingos, and D. J. Wales, *Introduction to Cluster Chemistry* (Prentice Hall, Englewood Cliffs, NJ, 1990), Chap. 1.