and others[6] shows that fluorocarbon complexation, although weak, may be a common phenomenon in the coordination chemistry of fluorocarbons, at least for coordinatively unsaturated metal centers. The ^{19}F coordination shift and, in suitable cases, the observation of coupling with the coordinated C–*F* nucleus seem to be useful spectroscopic criteria of binding.

Experimental Section

General Considerations. All manipulations were performed under dry nitrogen by employing standard Schlenk techniques. Dichloromethane was distilled from CaH_2; ether was distilled from sodium benzophenone ketyl and stored under nitrogen over molecular sieves. [(cod)Ir-$(PPh_3)_2$]SbF_6 was synthesized by a literature procedure.[18] 8-Fluoroquinoline was synthesized from *o*-fluoroaniline (Aldrich) by a modification of the Skraup reaction.[19]

Synthesis of [$IrH_2(PPh_3)_2(L)$](SbF_6) (L = 8-Fluoroquinoline). A solution of (η^4-1,5-cyclooctadiene)bis(triphenylphosphine)iridium(I) hexafluoroantimonate (213 mg, 0.201 mmol) and 8-fluoroquinoline (29.6 mg, 0.201 mmol) in 20 mL of dry, degassed dichloromethane was cooled to 0 °C, and hydrogen was bubbled through for 40 min. The volume of the pale yellowish green solution was reduced in vacuo to 5 mL, and the product was precipitated by addition of 20 mL of ether. The resulting pale greenish white powder was collected on a fritted disk, washed with ether, and dried in vacuo: yield 168 mg (76%); mp 181 °C dec. The product can be purified by precipitation from a concentrated dichloromethane solution with diethyl ether.

Spectroscopy. ^1H NMR spectra were obtained on a JEOL FX-90Q or a Bruker WM 250 spectrometer; chemical shifts are reported in δ (ppm) relative to Me_4Si. ^{19}F NMR spectra were obtained on a Bruker 500-MHz instrument ($CFCl_3$ internal standard). ^{31}P NMR spectra were obtained on a Varian CFT-20 instrument (85% H_3PO_4 external standard). IR spectra were recorded on a Nicolet 5S-X Fourier transform instrument.

Crystallography. A crystal of $IrSbP_2F_7NC_{45}H_{38}$ gave the data shown in Table I. Data (9361 points) were collected at room temperature by using a scan width of 1.2° below $K\alpha_1$ and 1.2° above $K\alpha_2$ to a maximum 2θ value of 60.0°. The intensities of 3 standard reflections were remeasured after every 97 reflections, and as the intensities of these reflections showed less than 6% variation, corrections for decomposition were deemed unnecessary. Data were corrected for Lorentz, polarization, and background effects. Heavy-atom positions were located by using direct methods.[20] Least-squares refinement followed by a difference Fourier synthesis allowed location of the remainder of the non-hydrogen positions. Hydrogen atom positions were calculated by assuming a C–H bond length of 0.97 Å and were included in the final refinement with fixed isotropic thermal parameters. Positions of the Ir–*H* atoms were not apparent from a difference Fourier synthesis. Anomalous dispersion corrections were made for Ir, Sb, and P. Scattering factors were taken from Cromer and Mann.[21]

The final cycle of refinement minimized $(|F_o| - |F_c|)^2$ and led to a final agreement factors: $R = 5.5\%$, $R = 100(\sum|F_o| - |F_c|/\sum|F_o|)$. A weight equal to $1/|F|$ was introduced in the final cycles of refinement: R_w = 7.0%.

Acknowledgment. We thank the donors of the Petroleum Research Fund, administered by the American Chemical Society, for funding.

Registry No. [$IrH_2(PPh_3)_2(L)$](SbF_6) (L = 8-fluoroquinoline), 108970-14-5; [Ir(COD)$(PPh_3)_2$](SbF_6), 91410-27-4.

Supplementary Material Available: Calculated H positions (Table S2) and anisotropic thermal parameters (Table S3) (3 pages); F_o vs. F_c values (Table S1) (68 pages). Ordering information is given on any current masthead page.

---

(18) Shapley, J. R.; Schrock, R. R.; Osborn, J. A. *J. Am. Chem. Soc.* **1969**, *91*, 2816.

(19) Sveinbjornsson, A.; Bradlow, H. L.; Oae, S.; Vanderwerf, C. A. *J. Org. Chem.* **1951**, *16*, 1450.

(20) Main, P.; Fiske, S. J.; Hull, S. E.; Lessinger, L.; Germain, G.; DeClerq, J. P.; Woolfson, M. M. "Multan"; University of York: York, England, 1980. Stewart, J. M., Ed. "The XRay System-Version of 1980", Tech. Rep. TR446; Computer Center, University of Maryland: College Park, MD, 1980.

(21) Cromer, D. T.; Mann, I. B. *Acta Crystallogr., Sect. A: Cryst. Phys., Diffr., Theor. Gen. Crystallogr.* **1968**, *A24*, 321.

Contribution from the Department of Chemistry,
University of South Carolina, Columbia, South Carolina 29208

An Unusual Example of Intramolecular Hydrogen Bonding in a Metal Carbonyl Cluster Compound. Synthesis and Crystal and Molecular Structure of $Ru_6(CO)_{16}(\mu\text{-}CO)_2(\mu\text{-}OH)_2(\mu_4\text{-}S)$

Richard D. Adams,* James E. Babin, and Miklos Tasi

Received December 12, 1986

It is well-known that compounds containing hydroxyl groups readily engage in hydrogen bonding to Lewis bases, B:.[1] Hy-

$$\mathrm{B\!:\!-\!-\!-\overset{\delta+}{H}\!-\!\overset{\delta-}{O}}$$

drogen bonding can play an important role in determining the overall structure, bonding and reactivity of the compounds involved.[1] In metal carbonyl cluster compounds, hydroxyl ligands invariably adopt bridging coordination modes.[2] We now wish to report that we have prepared and structurally characterized the new hexaruthenium cluster $Ru_6(CO)_{16}(\mu\text{-}CO)_2(\mu\text{-}OH)_2(\mu_4\text{-}S)$ (**1**), which has two bridging hydroxyl ligands and have obtained evidence for the existence of a significant intramolecular hydrogen bonding interaction between them.

Experimental Section

General Procedures and Materials. All reactions were performed under nitrogen atmosphere. $Ru_3(CO)_{12}$ was purchased from Strem Chemical Co. and was used as received. $Ru_3(CO)_9(\mu_3\text{-}CO)(\mu_3\text{-}S)$ was prepared as previously reported.[3]

TLC separations were performed in air on 0.25-mm Kieselgel 60 F_{254} (E. Merck) purchased from Bodman Chemicals. UV irradiation was performed by using an external high-pressure mercury lamp. IR spectra were recorded on a Nicolet 5 DXB FT IR spectrometer. UV-vis spectra were measured on a Cary 210 spectrophotometer. ^1H NMR spectra were run on IBM NR-80 and Bruker AM-300 spectrometers operating at 80 and 300 MHz, respectively. Elemental analyses were performed by MicAnal, Tucson, AZ.

Synthesis. A 50-mL THF solution of 30 mg (0.04 mmol) of $Ru_3(CO)_9(\mu_3\text{-}CO)(\mu_3\text{-}S)$, 31 mg (0.040 mmol) of $Ru_3(CO)_{12}$, and 20 μL of distilled water was irradiated for 6 h at 25 °C in the presence of a nitrogen purge. The brown solution was evaporated to dryness in vacuo, and the residue was extracted with CH_2Cl_2. The undissolved residue was dissolved in THF and cooled to –20 °C. This yielded 13 mg of $Ru_4(CO)_{11}(\mu_4\text{-}S)_2$ (34%).[3] The CH_2Cl_2 extract was chromatographed on a Florisil column with CH_2Cl_2 as solvent. A brown band and a blue band were separated. The brown band was further separated by TLC (hexane/CH_2Cl_2, v/v 75/25). This yielded the following compounds in order of elution: 19 mg of $Ru_3(CO)_{12}$ and 3 mg of $Ru_6(CO)_{17}(\mu_4\text{-}S)_2$ (12%).[4] The blue compound was recrystallized from CH_2Cl_2 at 0 °C to yield 5 mg of $Ru_6(CO)_{16}(\mu\text{-}CO)_2(\mu\text{-}OH)_2(\mu_4\text{-}S)$ (**1**) (8%). IR (ν(CO) in hexane): 2117 (vw), 2089 (s), 2051 (vs), 2042 (m), 2030 (w), 2020 (w), 1993 (w), 1876 (w) cm^{-1}. IR (ν(OH) in CD_2Cl_2): 3687 (m), 3602 (w) cm^{-1}. UV-vis (in CH_2Cl_2): λ_{max} 368 nm, ϵ 870; λ_{max} 679 nm, ϵ 5150. ^1H NMR (δ in toluene-d_8): –3.35 (s, 2 H). This resonance was not changed by cooling to –40 °C. Anal. Calcd for $Ru_6SO_{20}C_{18}H_2$: C, 18.37; H, 0.17. Found: C, 18.45; H, 0.33.

Crystallographic Analyses. Crystals of **1** were obtained by slow evaporation of solutions in CH_2Cl_2 solvent at 0 °C. Diffraction measurements were made on a Rigaku AFC6 automatic diffractometer by using Mo $K\alpha$ radiation. The unit cell was determined and refined from 25 randomly selected reflections obtained by using the AFC6 automatic search, center, index, and least-squares routines. Crystal data, data collection parameters, and results of the analyses are listed in Table I. All data processing was performed on a Digital Equipment Corp. MICROVAX II computer by using the Molecular Structure Corp. TEXSAN structure solving program library. Neutral-atom scattering factors were obtained from the standard sources.[5a] Anomalous dispersion corrections

---

(1) Cotton, F. A.; Wilkinson, G. *Advanced Inorganic Chemistry*; Wiley: New York, 1980; pp 219–224 and references therein.

(2) (a) Adams, R. D.; Babin, J. E.; Kim, H. S. *Inorg. Chem.* **1986**, *25*, 1122. (b) Carty, A. J.; MacLaughlin, S.; Taylor, N. J. *J. Chem. Soc., Chem. Commun.* **1981**, 476. (c) Aime, S.; Deeming, A. J.; Hursthouse, M. B.; Julius Backer-Dirks, J. D. *J. Chem. Soc., Dalton Trans.* **1982**, 1625. (d) Johnson, B. F. G.; Lewis, J.; Raithby, P. R.; Zuccaro, C. *J. Chem. Soc., Dalton Trans.* **1980**, 716.

(3) Adams, R. D.; Babin, J. E.; Tasi, M. *Inorg. Chem.* **1986**, *25*, 4514.

(4) Adams, R. D.; Babin, J. E.; Tasi, M. *Inorg. Chem.*, in press.

0020-1669/87/1326-2561$01.50/0

Table I. Crystallographic Data for the Structural Analysis

(A) Crystal Data

compd	**1**	α, deg	105.02 (1)
formula	$Ru_6SO_{20}C_{18}H_2$	β, deg	109.67 (1)
T, °C (±3 °C)	25	γ, deg	71.84 (1)
space group	$P\bar{1}$	V, Å^3	1480.3 (8)
a, Å	12.265 (2)	M_r	1168.7
b, Å	14.292 (2)	Z	2
c, Å	9.582 (1)	ρ_{calcd}, g cm^{-3}	2.62

(B) Measurement of Intensity Data

radiation: λ(Mo Kα), Å	0.7093
monochromator	graphite
detector aperature, mm	
horiz	2.0
vert	2.0
cryst faces	010, 0$\bar{1}$0, 100, $\bar{1}$00 001, 00$\bar{1}$, 10$\bar{1}$, $\bar{1}$01
cryst size, mm	0.16 × 0.29 × 0.32
cryst orientation: direction; deg from ϕ axis	[102]; 10.0°
reflctns measd	$h,\pm k,\pm l$
max 2θ, deg	50.0
scan type	moving cryst–stationary counter
ω-scan width: $A = (A + 0.347 \tan \theta)°$	1.1
bkgd	one-fourth scan time at each end of scan
ω-scan rate,[a] deg/min	4.0
no. of reflcns measd	5521
no. of data used ($F^2 \geq 3.0\sigma(F^2)$)	4690

(C) Treatment of Data

abs cor	applied	P factor	0.02
coeff, cm^{-1}	30.6	final residuals: R_F, R_{wF}	0.0264, 0.0352
grid	12 × 6 × 12		
transmission coeff		esd of unit wt observn	1.73
max	0.63	largest shift/error value of final cycle	0.12
min	0.44		
no. of variables (refined)	414	largest peak in final diff Fourier, e/Å^3	0.67

[a] Rigaku software uses a multiple-scan technique. If the $I/\sigma(I)$ ratio is less than 10.0, a second scan is made and the results are added to first scan, etc. A maximum of three scans was permitted per reflection.

were applied to all non-hydrogen atoms.[5b] Full-matrix least-squares refinements minimized the function $\sum_{hkl} w(|F_o| - |F_c|)^2$ where $w = 1/\sigma(F)^2$, $\sigma(F) = \sigma(F_o^2)/2F_o$, and $\sigma(F_o^2) = [\sigma(I_{raw})^2 + (PF_o)^2]^{1/2}/Lp$. Compound **1** crystallized in the triclinic crystal system. The space group $P\bar{1}$ was assumed and confirmed by the successful solution and refinement of the structure. The structure of **1** was solved by a combination of Patterson and difference Fourier techniques. All non-hydrogen atoms were refined with anisotropic thermal parameters. The positions of the hydroxyl hydrogen atoms were located and refined.

Results and Discussion

Compound **1** was obtained as a minor product (8% yield) of the reaction of a mixture of the compounds $Ru_3(CO)_9(\mu_3\text{-}CO)(\mu_3\text{-}S)$ and $Ru_3(CO)_{12}$ with H_2O in the presence of UV irradiation. It was characterized by IR and ^1H NMR spectroscopies and by a single-crystal X-ray diffraction analysis. An ORTEP drawing of the molecular structure of **1** is shown in Figure 1. Positional parameters are listed in Table II. Intramolecular distances and selected intramolecular angles are listed in Tables III and IV. The molecule consists of a central rectangular cluster of four ruthenium atoms that contains a quadruply bridging sulfido ligand. Two edges of this cluster, Ru1–Ru3 and Ru2–Ru6, are bridged by $Ru(CO)_4$ groupings and by hydroxyl ligands, O1–H1 and O2–H2. All of the Ru–O bond distances lie in the narrow range 2.116 (3)–2.134 (3) Å. These distances are slightly shorter than those found in the compound $Ru_4(CO)_{10}(\mu_4\text{-}C{=}CH\text{-}i\text{-}Pr)(\mu_3\text{-}OH)(\mu\text{-}PPh_2)$, which contains a triply bridging hydroxyl ligand.[2b]

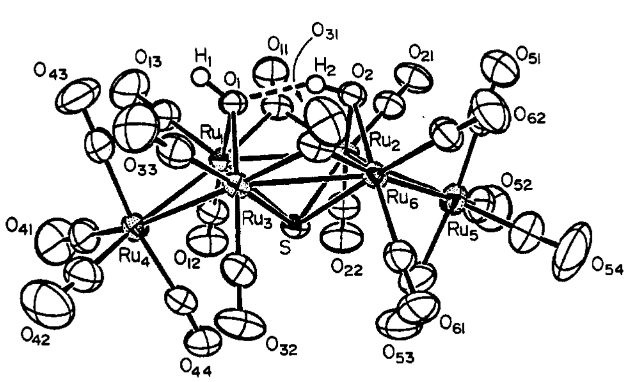

Figure 1. ORTEP diagram of $Ru_6(CO)_{16}(\mu\text{-}CO)_2(\mu\text{-}OH)_2(\mu_4\text{-}S)$ (**1**), showing 50% probability thermal ellipsoids. The ellipsoids of the hydrogen atoms have been reduced for clarity.

Table II. Positional Parameters and B(eq) for $Ru_6(CO)_{16}(\mu\text{-}CO)_2(\mu\text{-}OH)_2(\mu_4\text{-}S)$ (**1**)

atom	x	y	z	B(eq), Å^2
Ru1	−0.130375 (30)	0.206107 (25)	0.150478 (39)	2.8
Ru2	0.114072 (31)	0.167226 (25)	0.331933 (37)	2.7
Ru3	−0.049534 (30)	0.245747 (25)	−0.093096 (37)	2.7
Ru4	−0.280422 (31)	0.331746 (27)	−0.056727 (42)	2.9
Ru5	0.325807 (35)	0.225658 (31)	0.397356 (47)	3.2
Ru6	0.193632 (29)	0.204795 (25)	0.095454 (39)	2.9
S	0.013878 (91)	0.300874 (75)	0.17910 (12)	2.7
O1	−0.06003 (26)	0.11720 (21)	−0.02907 (33)	2.5
O2	0.16102 (27)	0.07860 (22)	0.13670 (34)	2.8
O11	−0.03937 (36)	0.03474 (29)	0.33171 (46)	5.9
O12	−0.23375 (39)	0.35406 (29)	0.38740 (46)	6.5
O13	−0.32402 (40)	0.09999 (37)	0.08259 (63)	7.5
O21	0.26584 (37)	0.01138 (28)	0.52676 (42)	5.6
O22	0.05578 (43)	0.31880 (30)	0.59546 (43)	5.9
O31	0.15136 (33)	0.14500 (31)	−0.24408 (41)	5.8
O32	−0.04847 (37)	0.44145(26)	−0.15041 (45)	6.4
O33	−0.15557 (40)	0.18537 (33)	−0.42571 (42)	5.0
O41	−0.50508 (41)	0.38040 (41)	0.04332 (64)	8.6
O42	−0.38350 (54)	0.45007 (39)	−0.31554 (54)	8.1
O43	−0.32729 (39)	0.13880 (33)	−0.26748 (54)	5.1
O44	−0.20547 (35)	0.51035 (27)	0.17945 (44)	4.4
O51	0.45257 (39)	0.00514 (33)	0.31832 (57)	7.0
O52	0.39649 (49)	0.23126 (44)	0.73619 (54)	6.8
O53	0.18151 (46)	0.44508 (32)	0.44085 (52)	5.7
O54	0.53755 (51)	0.28554 (51)	0.37745 (79)	9.6
O61	0.25018 (36)	0.39099 (28)	0.08250 (52)	6.6
O62	0.42185 (37)	0.09716 (33)	0.01247 (62)	8.4
C11	−0.02484 (43)	0.09881 (35)	0.29568 (53)	3.7
C12	−0.19460 (44)	0.29778 (36)	0.29929 (58)	4.4
C13	−0.25145 (47)	0.14068 (40)	0.10680 (63)	4.7
C21	0.20954 (44)	0.07037 (37)	0.45418 (52)	3.6
C22	0.07883 (46)	0.25927 (37)	0.49787 (54)	4.0
C31	0.11674 (42)	0.17668 (34)	−0.14064 (52)	3.7
C32	−0.04949 (41)	0.36837 (36)	−0.12877 (52)	3.8
C33	−0.11810 (44)	0.20830 (36)	−0.29921 (54)	3.9
C41	−0.42115 (47)	0.36156 (42)	0.00668 (65)	4.4
C42	−0.34269 (52)	0.40697 (41)	−0.21848 (64)	4.5
C43	−0.30992 (43)	0.20974 (41)	−0.18930 (64)	3.9
C44	−0.22775 (42)	0.44409 (36)	0.09251 (55)	3.8
C51	0.40320 (46)	0.08560 (46)	0.34740 (65)	4.4
C52	0.37179 (53)	0.22820 (48)	0.61084 (70)	4.4
C53	0.23122 (56)	0.36408 (46)	0.42059 (62)	4.0
C54	0.45871 (56)	0.26509 (53)	0.38604 (78)	5.3
C61	0.22629 (42)	0.32235 (37)	0.08629 (58)	4.1
C62	0.33838 (44)	0.13507 (37)	0.04880 (62)	4.6
H1	−0.1106 (56)	0.0827 (47)	−0.0935 (69)	6.2
H2	0.0973 (48)	0.0706 (40)	0.0675 (59)	3.9

The hydrogen atoms on the hydroxyl ligands were located and refined; O1–H1 = 0.89 (6) Å and O2–H2 = 0.86 (5) Å. Interestingly, H2 was found to be directed toward atom O1 at the distance O1···H2 = 1.83 (5) Å. This distance is consistent with the formation of a hydrogen-bonding interaction.[1] This is supported by the observation of two O–H stretching vibrations that occur at 3687 (m) and 3602 (w) cm^{-1} in its infrared spectrum. The ^1H NMR spectrum of **1** in toluene-d_8 showed only a broad

---

(5) *International Tables for X-ray Crystallography*; Kynoch: Birmingham, England, 1975; Vol. IV: (a) Table 2.2B, pp 99–101; (b) Table 2.3.1, pp 149–150.

Table III. Intramolecular Distances (Å) for $Ru_6(CO)_{16}(\mu\text{-}CO)_2(\mu\text{-}OH)_2(\mu_4\text{-}S)$ (**1**)

Ru1–C13	1.871 (5)	Ru5–Ru6	2.7898 (7)
Ru1–C12	1.875 (5)	Ru6–C61	1.874 (5)
Ru1–O1	2.122 (3)	Ru6–C62	1.883 (5)
Ru1–C11	2.149 (5)	Ru6–O2	2.120 (3)
Ru1–S	2.452 (1)	Ru6–C31	2.121 (5)
Ru1–Ru4	2.8074 (7)	Ru6–S	2.474 (1)
Ru1–Ru2	2.8763 (7)	O1–H1	0.89 (6)
Ru2–C22	1.863 (5)	O2–H2	0.86 (5)
Ru2–C21	1.888 (5)	O11–C11	1.139 (6)
Ru2–C11	2.108 (5)	O12–C12	1.127 (6)
Ru2–O2	2.116 (3)	O13–C13	1.139 (6)
Ru2–S	2.466 (1)	O21–C21	1.135 (6)
Ru2–Ru5	2.7961 (6)	O22–C22	1.138 (6)
Ru2–Ru6	2.9701 (6)	O31–C31	1.140 (5)
Ru3–C32	1.871 (5)	O32–C32	1.121 (6)
Ru3–C33	1.873 (5)	O33–C33	1.141 (6)
Ru3–C31	2.116 (5)	O41–C41	1.130 (7)
Ru3–O1	2.134 (3)	O42–C42	1.134 (7)
Ru3–S	2.445 (1)	O43–C43	1.126 (6)
Ru3–Ru4	2.8149 (6)	O44–C44	1.125 (6)
Ru3–Ru6	2.8868 (6)	O51–C51	1.132 (7)
Ru4–C41	1.909 (5)	O52–C52	1.128 (7)
Ru4–C42	1.923 (6)	O53–C53	1.128 (7)
Ru4–C43	1.929 (6)	O54–C54	1.127 (8)
Ru4–C44	1.968 (5)	O61–C61	1.120 (6)
Ru5–C52	1.924 (6)	O62–C62	1.122 (6)
Ru5–C54	1.926 (6)	Ru1–Ru3	3.0570 (6)
Ru5–C51	1.940 (6)	O1···O2	2.608 (4)
Ru5–C53	1.956 (6)	O1···H2	1.83 (5)

single resonance (δ −3.35) for the hydroxyl hydrogen atoms, which suggests that they are rapidly interchanging their environments.

The Ru–Ru bond distances in the central Ru1, Ru2, Ru3, Ru6 portion of the molecule span a wide range; Ru1–Ru2 = 2.8763 (7) Å, Ru3–Ru6 = 2.8868 (6) Å, Ru2–Ru6 = 2.9701 (6) Å, and Ru1–Ru3 = 3.0570 (6) Å. These distances are much longer than those found in the unsaturated cluster $Ru_4(CO)_7(\mu\text{-}CO)_2(PMe_2Ph)_2(\mu_4\text{-}S)_2$ (**2**).[3] The Ru–Ru distances to the $Ru(CO)_4$ groups are considerably shorter and span the narrow range 2.7898 (7)–2.8149 (6) Å. The latter distances are typical of Ru–Ru single bonds.[6] A quadruply bridging sulfido ligand bridges the central portion of the cluster. The Ru–S distances range from 2.445 (1) to 2.474 (1) Å and are similar to those found in compound **2**.

Electron-counting procedures indicate that **1** has an unusual electron configuration that may help to explain the elongation of the metal–metal bonds. An electron-precise tetranuclear cluster with four metal–metal bonds should contain 64 electrons. If one includes 14 electrons for each *edge*-bridging $Ru(CO)_4$ group,[7] then compound **1** should contain 92 electrons. If one assumes that the bridging hydroxyl ligands serve as three-electron donors and the sulfido ligand serves as a four-electron donor,[8] then it is found that compound **1** contains 94 electrons. It is proposed that the two excess electrons occupy a delocalized antibonding orbital on the central Ru_4 portion of the cluster and that these cause a weakening of the four associated metal–metal bonds. Without calculations, it is not possible to predict quantitatively which bonds will be affected by the greatest amount, but it is believed that the shortness of the Ru2–Ru6 bond compared to the Ru1–Ru3 bond may be a long-range consequence of the hydrogen bonding between the hydroxyl ligands.

Further evidence bearing on the unusual electron configuration of **1** was obtained from its UV–visible spectrum. Compound **1** exhibits two low-energy absorptions, λ_{max} 368 nm (ϵ 870) and λ_{max} 679 nm (ϵ 5150), with the latter undoubtedly accounting for its unusual blue color. It is believed that these absorptions can be attributed to low-energy electronic transitions, possibly between relatively closely spaced antibonding orbitals on the central Ru_4 portion of the cluster. Further studies of this phenomenon are in progress.

Table IV. Selected Intramolecular Bond Angles (deg) for $Ru_6(CO)_{16}(\mu\text{-}CO)_2(\mu\text{-}OH)_2(\mu_4\text{-}S)$ (**1**)

C13–Ru1–O1	94.8 (2)	C41–Ru4–Ru1	97.3 (2)
C13–Ru1–S	172.8 (2)	C41–Ru4–Ru3	162.9 (2)
C13–Ru1–Ru4	89.4 (2)	C42–Ru4–Ru1	163.4 (2)
C13–Ru1–Ru2	132.3 (2)	C42–Ru4–Ru3	97.6 (2)
C12–Ru1–O1	173.2 (2)	C43–Ru4–Ru1	84.9 (2)
C12–Ru1–S	92.6 (1)	C43–Ru4–Ru3	81.9 (1)
C12–Ru1–Ru4	88.4 (2)	C44–Ru4–Ru1	87.7 (1)
C12–Ru1–Ru2	96.5 (2)	C44–Ru4–Ru3	91.7 (1)
O1–Ru1–C11	91.7 (1)	Ru1–Ru4–Ru3	65.88 (2)
O1–Ru1–S	82.97 (8)	C52–Ru5–Ru6	160.6 (2)
O1–Ru1–Ru4	86.00 (8)	C52–Ru5–Ru2	96.4 (2)
O1–Ru1–Ru2	85.11 (8)	C54–Ru5–Ru6	100.6 (2)
C11–Ru1–S	101.3 (1)	C54–Ru5–Ru2	164.9 (2)
C11–Ru1–Ru4	174.3 (1)	C51–Ru5–Ru6	84.8 (2)
C11–Ru1–Ru2	46.9 (1)	C51–Ru5–Ru2	87.5 (1)
S–Ru1–Ru4	83.61 (3)	C53–Ru5–Ru6	87.4 (2)
S–Ru1–Ru2	54.42 (3)	C53–Ru5–Ru2	87.5 (2)
Ru4–Ru1–Ru2	137.83 (2)	Ru6–Ru5–Ru2	64.24 (2)
C22–Ru2–O2	172.0 (2)	C61–Ru6–O2	172.5 (2)
C22–Ru2–S	88.6 (2)	C61–Ru6–S	91.5 (1)
C22–Ru2–Ru5	89.2 (2)	C61–Ru6–Ru5	88.0 (2)
C22–Ru2–Ru1	93.5 (2)	C61–Ru6–Ru3	96.2 (1)
C22–Ru2–Ru6	126.5 (1)	C61–Ru6–Ru2	127.7 (2)
C21–Ru2–O2	93.6 (2)	C62–Ru6–O2	95.3 (2)
C21–Ru2–S	172.4 (2)	C62–Ru6–S	175.0 (2)
C21–Ru2–Ru5	84.5 (2)	C62–Ru6–Ru5	87.3 (2)
C21–Ru2–Ru1	133.6 (2)	C62–Ru6–Ru3	131.3 (2)
C21–Ru2–Ru6	121.7 (1)	C62–Ru6–Ru2	124.7 (2)
C11–Ru2–O2	92.4 (2)	O2–Ru6–C31	95.2 (1)
C11–Ru2–S	102.1 (1)	O2–Ru6–S	85.35 (8)
C11–Ru2–Ru5	169.5 (1)	O2–Ru6–Ru5	85.11 (9)
C11–Ru2–Ru1	48.1 (1)	O2–Ru6–Ru3	87.42 (9)
C11–Ru2–Ru6	126.2 (1)	O2–Ru6–Ru2	45.43 (8)
O2–Ru2–S	85.65 (8)	C31–Ru6–S	100.4 (1)
O2–Ru2–Ru5	85.02 (8)	C31–Ru6–Ru5	171.7 (1)
O2–Ru2–Ru1	87.61 (8)	C31–Ru6–Ru3	47.0 (1)
O2–Ru2–Ru6	45.54 (8)	C31–Ru6–Ru2	127.3 (1)
S–Ru2–Ru5	87.85 (3)	S–Ru6–Ru5	87.83 (3)
S–Ru2–Ru1	53.99 (3)	S–Ru6–Ru3	53.61 (3)
S–Ru2–Ru6	53.17 (3)	S–Ru6–Ru2	52.91 (3)
Ru5–Ru2–Ru1	141.60 (2)	Ru5–Ru6–Ru3	141.21 (2)
Ru5–Ru2–Ru6	57.77 (2)	Ru5–Ru6–Ru2	57.98 (2)
Ru1–Ru2–Ru6	91.12 (2)	Ru3–Ru6–Ru2	90.61 (2)
C32–Ru3–O1	172.4 (2)	Ru3–S–Ru1	77.24 (3)
C32–Ru3–S	91.4 (1)	Ru3–S–Ru2	115.96 (4)
C32–Ru3–Ru4	88.8 (1)	Ru3–S–Ru6	71.86 (3)
C32–Ru3–Ru6	95.8 (1)	Ru1–S–Ru2	71.59 (3)
C33–Ru3–O1	95.5 (2)	Ru1–S–Ru6	115.87 (4)
C33–Ru3–S	172.6 (2)	Ru2–S–Ru6	73.93 (3)
C33–Ru3–Ru4	89.1 (1)	H1–O1–Ru1	110.66
C33–Ru3–Ru6	132.6 (1)	H1–O1–Ru3	117.37
C31–Ru3–O1	94.5 (1)	Ru1–O1–Ru3	91.8 (1)
C31–Ru3–S	101.5 (1)	H2–O2–Ru2	108.94
C31–Ru3–Ru4	174.9 (1)	H2–O2–Ru6	103.60
C31–Ru3–Ru6	47.1 (1)	Ru2–O2–Ru6	89.0 (1)
O1–Ru3–S	82.90 (8)	O11–C11–Ru2	138.7 (4)
O1–Ru3–Ru4	85.58 (8)	O11–C11–Ru1	136.3 (4)
O1–Ru3–Ru6	85.05 (8)	Ru2–C11–Ru1	85.0 (2)
S–Ru3–Ru4	83.57 (3)	O31–C31–Ru3	137.7 (4)
S–Ru3–Ru6	54.53 (3)	O31–C31–Ru6	136.2 (4)
Ru4–Ru3–Ru6	137.87 (2)	Ru3–C31–Ru6	85.9 (2)

Acknowledgment. The research was supported by the National Science Foundation under Grant No. CHE-8416460. The AM-300 NMR spectrometer was purchased with funds from the National Science Foundation, Grant No. CHE-8411172.

Supplementary Material Available: A table of anisotropic thermal parameters (*U* values) (2 pages); a listing of calculated and observed structure factor amplitudes (32 pages). Ordering information is given on any current masthead page.

---

(6) Bruce, M. I. In *Comprehensive Organometallic Chemistry*; Wilkinson, G., Stone, F. G. A., Abel, E., Eds.; Pergamon: Oxford, 1982; Vol. 4, pp 846–847.

(7) Mingos, D. M. P. *Acc. Chem. Res.* **1984**, *17*, 311.

(8) Adams, R. D.; Babin, J. E.; Natarajan, K. *J. Am. Chem. Soc.* **1986**, *108*, 3518.