Contribution from the Department of Chemistry,
The Ohio State University,
120 West 18th Avenue, Columbus, Ohio 43210

Reaction of THF·BH$_3$ with H$_2$Ru$_4$(CO)$_{13}$ and H$_4$Ru$_4$(CO)$_{12}$; Preparation and Structure of the Tetraruthenium "Butterfly" Cluster HRu$_4$(CO)$_{12}$BH$_2$

Fung-E. Hong, Deborah A. McCarthy, James P. White III,
Charles E. Cottrell, and Sheldon G. Shore*

Received December 7, 1989

Recently we reported the synthesis and structure of the hydridoruthenium carbonyl boride[1] HRu$_6$(CO)$_{17}$B, prepared from the reaction of THF·BH$_3$ with Ru$_3$(CO)$_{12}$. In earlier work from this laboratory, reactions of THF·BH$_3$ with the unsaturated cluster (μ-H)$_2$Os$_3$(CO)$_{10}$ produced the borylidyne carbonyl[2] (μ-H)$_3$-(CO)$_9$Os$_3$(μ_3-BCO) and the trioxyboroxin-supported triosmium methylidyne cluster system[3] [(μ-H)$_3$(CO)$_9$Os$_3$(μ_3-CO)]$_3$B$_3$O$_3$. The possibility of preparing analogous ruthenium cluster complexes has been of interest to us. While the unsaturated cluster (μ-H)$_2$Ru$_3$(CO)$_{10}$ has not been isolated, its transitory existence in situ has been implied[4] in the formation of certain mixed-metal clusters. An early attempt to prepare the ruthenium analogues of the osmium complexes cited above involved the reaction of THF·BH$_3$ with Ru$_3$(CO)$_{12}$ in the presence of a stoichiometric quantity[5] of H$_2$. While the anticipated products were not found, the proton NMR spectrum of the reaction solution indicated that a small amount of the "butterfly" complex[1,6,15] HRu$_4$(CO)$_{12}$BH$_2$ had formed. This observation encouraged us to examine the reaction of THF·BH$_3$ with H$_2$Ru$_4$(CO)$_{13}$ and H$_4$Ru$_4$(CO)$_{12}$, the results of which are reported here.

Experimental Section

All reactions were carried out by using standard vacuum-line techniques or Schlenk techniques under an atmosphere of prepurified N$_2$. CH$_2$Cl$_2$ (Mallinkrodt) was dried with P$_2$O$_5$, distilled, and stored in a sealed flask. THF, Me$_2$O, and hexanes were dried over sodium benzophenone ketyl. KH (Aldrich) in mineral oil dispersions was washed repeatedly with dry pentane to remove the oil, activity 93%, and then stored under dry, oxygen-free nitrogen in a glovebox. H$_4$Ru$_4$(CO)$_{12}$[5] and B$_2$H$_6$[7] were prepared according to published procedures. NMR chemical shifts are referenced to Si(CH$_3$)$_4$ (^{1}H, δ = 0.00 ppm) and BF$_3$OEt$_2$ (^{11}B, δ = 0.00 ppm).

X-ray diffraction data were collected with an Enraf-Nonius CAD4 diffractometer with graphite-monochromated Mo Kα radiation. Unit cell parameters were obtained by a least-squares refinement of the angular settings from 25 reflections well distributed in reciprocal space and lying in a 2θ range of 24–30°.

Crystallographic data are given in Table I. All data were corrected for Lorentz-polarization effects. An empirical absorption correction was made. Crystallographic computations were carried out on a PDP 11/44 computer using SDP (Structure Determination Package). The structure was solved by direct-methods MULTAN 11/82 and difference Fourier syntheses with analytical atomic scattering factors used throughout the structure refinement. All non-hydrogen atoms were refined by using anisotropic thermal parameters. All the hydrogen atoms were located on the difference Fourier map, but only H(3) was fully refined. For H(1), the thermal parameter was refined while the positional parameters were fixed. The resulting thermal parameter was fixed, and the positional parameters were refined to convergence for the final solution. Both the positional and thermal parameters for H(2) were fixed. There were no electron density peaks above 0.75 e/Å^3 in the final difference map.

Table I. Crystallographic Data for HRu$_4$(CO)$_{12}$BH$_2$

chem formula	C$_{12}$H$_3$O$_{12}$Ru$_4$B	vol, Å^3	977.8
MW, g mol^{-1}	754.5	density (calc), g cm^{-3}	2.562
space group	*Cm*	radiation	Mo Kα (0.710730 Å)
Z	2		
t, °C	−50	abs coeff, cm^{-1}	30.24
a, Å	10.432 (2)	$R_F = \sum[\lvert F_o\rvert - \lvert F_c\rvert]/\sum\lvert F_o\rvert$	0.020
b, Å	15.709 (2)		
c, Å	6.472 (1)	$R_{wF} = (\sum w[\lvert F_o\rvert - \lvert F_c\rvert]2/\sum w\lvert F_o\rvert^2)^{1/2}$	0.028
β, deg	112.86 (2)		

Table II. Positional Parameters and Their Estimated Standard Deviations for HRu$_4$(CO)$_{12}$BH$_2$[a]

atom	*x*	*y*	*z*	*B*, Å^2
Ru(1)	1.014	0.000	0.997	1.799 (7)
Ru(2)	0.58258 (4)	0.000	0.71605 (7)	1.996 (7)
Ru(3)	0.79638 (3)	0.09239 (2)	1.04923 (6)	1.805 (4)
B	0.7986 (6)	0.000	0.796 (1)	2.0 (1)
H(1)	0.895 (9)	0.000	0.71 (2)	2.1
H(2)	0.693	0.000	0.582	4.0
H(3)	0.81 (1)	0.000	1.20 (2)	2 (2)*
C(1)	1.1158 (5)	0.000	1.312 (1)	2.6 (1)
C(2)	1.1211 (4)	−0.0930 (3)	0.9541 (8)	2.58 (8)
C(3)	0.4781 (6)	0.000	0.901 (1)	2.8 (1)
C(4)	0.4719 (5)	0.0909 (3)	0.5330 (8)	2.86 (9)
C(5)	0.6483 (4)	0.1477 (3)	1.1042 (8)	3.01 (8)
C(6)	0.8047 (5)	0.1777 (2)	0.8455 (8)	2.63 (7)
C(7)	0.9396 (5)	0.1431 (3)	1.3085 (8)	2.79 (8)
O(1)	1.1763 (6)	0.000	1.5027 (9)	3.7 (1)
O(2)	1.1871 (4)	−0.1485 (3)	0.9370 (7)	4.57 (9)
O(3)	0.9173 (5)	0.500	1.0133 (9)	3.9 (1)
O(4)	0.9053 (4)	0.3559 (3)	0.4264 (7)	4.26 (9)
O(5)	0.5646 (4)	0.1845 (3)	1.1371 (7)	5.00 (9)
O(6)	0.8121 (5)	0.2241 (2)	0.7163 (7)	4.13 (8)
O(7)	0.5209 (4)	0.3246 (3)	0.4566 (8)	4.4 (1)

[a] Values marked with an asterisk are for atoms that were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as $(4/3)[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

Synthesis of HRu$_4$(CO)$_{12}$BH$_2$. H$_4$Ru$_4$(CO)$_{12}$ (130.0 mg, 0.180 mmol) was added to a 50-mL flask, and CH$_2$Cl$_2$ (15 mL), THF (1.06 mmol), and B$_2$H$_6$ (0.52 mmol) were then condensed into the flask at −196 °C. The solution was stirred at 40 °C for 4 days and then chromatographed on a silica column with hexanes under an N$_2$ atmosphere. The first yellow band was found to contain HRu$_4$(CO)$_{12}$BH$_2$ and H$_4$Ru$_4$(CO)$_{12}$. Successive recrystallization from CH$_2$Cl$_2$ solutions yielded 81.6 mg (0.108 mmol) of the "butterfly" cluster (60% based on H$_4$Ru$_4$(CO)$_{12}$). Dark yellow crystals of HRu$_4$(CO)$_{12}$BH$_2$ for X-ray diffraction studies were obtained by slow evaporation of solvent from a CH$_2$Cl$_2$ solution. ^{11}B NMR spectrum (glyme-d_{10}, 30 °C): 113.5 ppm (t, J_{BH} = 65 Hz). ^{1}H NMR spectrum (glyme-d_{10}, 30 °C): −8.5 (q, 1:1:1:1, J_{BH} = 65 Hz, Ru–H–B), −21.15 ppm (s, Ru–H–Ru). IR spectrum (ν_{CO}, C$_6$H$_6$): 2074 vs, 2063 vs, 2052 m, 2030 s, 2017 m, 2003 m cm^{-1}. Mass spectrum (FAB): highest intensity peak calculated for ^{1}H$_3$^{11}B$_1$^{12}C$_{12}$^{16}O$_{12}$^{102}Ru$_4$ *m/e* = 758; found *m/e* = 756 (M − 2).

Synthesis of K[HRu$_4$(CO)$_{12}$BH]. An Me$_2$O (3 mL) solution of HRu$_4$(CO)$_{12}$BH$_2$ (24.1 mg, 31.9 μmol) and KH (1.5 mg, 37.3 μmol) was stirred at −78 °C for 24 h. After removal of Me$_2$O, the solid was washed with hexanes and filtered, giving a yellow solution containing HRu$_4$(CO)$_{12}$BH$_2$ and a brown solid that was identified from its NMR spectra as K[HRu$_4$(CO)$_{12}$BH] (21.7 mg, 29.8 μmol, 85.7% based on HRu$_4$(CO)$_{12}$BH$_2$). ^{11}B NMR spectrum (glyme-d_{10}, 30 °C): 140.9 ppm (d, J_{BH} = 80 Hz). ^{1}H NMR spectrum (glyme-d_{10}, 20 °C): −6.6 (d, br, J_{BH} = 80 Hz, Ru–H–B), −20.81 ppm (s, Ru–H–Ru). IR spectrum (ν_{CO}, THF): 2024 vs, 2000 s, 1986 s, 1969 m, 1925 m cm^{-1}.

2-D Chemical Exchange. To confirm chemical exchange between the hydrides of K[HRu$_4$(CO)$_{12}$BH] at −6.6 and −20.8 ppm, a two-dimensional chemical exchange experiment[8] was performed with a mixing time

---

(1) Hong, F.-E.; Coffy, T. J.; McCarthy, D. A.; Shore, S. G. *Inorg. Chem.* **1989**, *28*, 3284.

(2) (a) Shore, S. G.; Jan, D.-Y.; Hsu, L.-Y.; Hsu, W.-L. *J. Am. Chem. Soc.* **1983**, *105*, 5923. (b) Jan, D.-Y.; Shore, S. G. *Organometallics* **1987**, *6*, 428. (c) Jan, D.-Y.; Hsu, L.-Y.; Workman, D. P.; Shore, S. G. *Organometallics* **1987**, *6*, 1984.

(3) Shore, S. G.; Jan, D.-Y.; Hsu, W.-L.; Hsu, L.-Y.; Kennedy, S.; Hoffman, J. C.; Lin Wang, T.-C.; Marshall, A. G. *J. Chem. Soc., Chem. Commun.* **1984**, 392.

(4) (a) Lavigne, G.; Papageorgiou, F.; Bergounhou, C.; Bonnet, J. J. *Inorg. Chem.* **1983**, *22*, 2485. (b) Lindsell, W. E.; Knobler, C. B.; Kaesz, H. D. *J. Chem. Soc., Chem. Commun.* **1985**, 1171.

(5) Knox, S. A. R.; Koepke, J. W.; Andrews, M. A.; Kaesz, H. D. *J. Am. Chem. Soc.* **1975**, *97*, 3942.

(6) Eady, C. R.; Johnson, B. F. G.; Lewis, J. *J. Chem. Soc., Dalton Trans.* **1977**, 477.

(7) (a) Shapiro, I.; Weiss, H. G.; Schmich, M.; Skolnik, S.; Smith, G. B. L. *J. Am. Chem. Soc.* **1952**, *74*, 901. (b) Toft, M. A.; Leach, J. B.; Himpsl, F. L.; Shore, S. G. *Inorg. Chem.* **1982**, *21*, 1952.

(8) Macura, S.; Ernst, R. R. *Mol. Phys.* **1980**, *41*, 95.

0020-1669/90/1329-2874$02.50/0

Table III. Selected Bond Distances (Å) and Esd's for $HRu_4(CO)_{12}BH_2$

Ru(1)–Ru(3)	2.8220 (4)	Ru(1)–C(1)	1.902 (6)
Ru(2)–Ru(3)	2.8283 (5)	Ru(1)–C(2)	1.924 (4)
Ru(3)–Ru(3)	2.904 (1)	Ru(2)–C(3)	1.908 (2)
Ru(1)–H(1)	1.8 (2)	Ru(2)–C(4)	1.927 (5)
Ru(2)–H(2)	1.7	Ru(3)–C(5)	1.922 (4)
Ru(3)–H(3)	1.73 (5)	Ru(3)–C(6)	1.906 (4)
B–H(1)	1.3 (2)	Ru(3)–C(7)	1.933 (5)
B–H(2)	1.4	C(1)–O(1)	1.147 (9)
Ru(1)–B	2.111 (6)	C(2)–O(2)	1.143 (6)
Ru(2)–B	2.106 (6)	C(5)–O(5)	1.134 (6)
Ru(3)–B	2.195 (5)	C(6)–O(6)	1.134 (6)

Table IV. Selected Bond Angles (deg) and Esd's for $HRu_4(CO)_{12}BH_2$

Ru(1)–Ru(3)–Ru(2)	94.38 (1)	Ru(2)–Ru(3)–B	47.5 (2)
Ru(1)–H(1)–B	83 (6)	Ru(2)–Ru(3)–C(5)	85.4 (1)
Ru(1)–Ru(3)–B	47.8 (2)	Ru(2)–Ru(3)–C(6)	93.9 (2)
Ru(1)–Ru(3)–H(3)	74 (3)	Ru(2)–Ru(3)–C(7)	171.4 (1)
Ru(1)–B–Ru(2)	158.8 (3)	Ru(2)–H(2)–B	85.4
Ru(1)–B–Ru(3)	81.9 (2)	Ru(2)–B–Ru(3)	82.3 (4)
Ru(1)–C(1)–O(1)	179.4 (6)	Ru(2)–Ru(3)–H(3)	83 (4)
Ru(1)–C(2)–O(2)	177.6 (5)	Ru(3)–Ru(2)–B	50.2 (1)
Ru(3)–Ru(1)–C(2)	92.2 (2)	Ru(3)–C(5)–O(5)	176.2 (5)
Ru(3)–Ru(2)–C(3)	89.7 (2)	Ru(3)–C(6)–O(6)	175.2 (4)
Ru(3)–Ru(2)–C(4)	92.0 (2)	Ru(3)–H(3)–Ru(3)	115.1 (4)

of 0.3 s at 37 °C. Cross peaks were observed in the two-dimensional data set, confirming chemical exchange, and the areas of the diagonal and the off-diagonal peaks were used to calculate the first-order rate constant.[9,10] At 37 °C the measured first-order rate constant is 1.1 s^{-1}.

Results

Reactions of $THF{\cdot}BH_3$ with $H_2Ru_4(CO)_{13}$ and with $H_4Ru_4(CO)_{12}$ produce the ruthenium "butterfly" cluster $HRu_4(CO)_{12}BH_2$. This preparation, although requiring a significantly longer period of time, provides a significantly larger yield of $HRu_4(CO)_{12}BH_2$ than previously reported.[6,15] The results of a single-crystal X-ray diffraction analysis of $HRu_4(CO)_{12}BH_2$ are given in Tables I–IV. The molecular structure is shown in Figure 1.

Potassium hydride deprotonates $HRu_4(CO)_{12}BH_2$ to form the anions $[HRu_4(CO)_{12}BH]^-$ and $[HRu_4(CO)_{12}B]^{2-}$. A 2-D chemical exchange experiment at 37 °C involving $[HRu_4(CO)_{12}BH]^-$ indicates exchange of the bridging hydrides.

Discussion

Reaction of $H_2Ru_4(CO)_{13}$ with $THF{\cdot}BH_3$ at 40 °C for 14 days results in the production of $H_4Ru_4(CO)_{12}$ and a very small amount of the butterfly cluster $HRu_4(CO)_{12}BH_2$. However, $HRu_4(CO)_{12}BH_2$ was obtained in 60% yield in the following reaction of $H_4Ru_4(CO)_{12}$ with $THF{\cdot}BH_3$.

$$H_4Ru_4(CO)_{12} + THF{\cdot}BH_3 \xrightarrow[40\ ^\circ C]{CH_2Cl_2} HRu_4(CO)_{12}BH_2 + H_2 + THF \quad (1)$$

In addition, the ruthenium carbide cluster, $Ru_6(CO)_{17}C$,[11] was formed (30% yield) as well as other unidentified products. Formation of the carbide cluster increases with increasing temperature.

$HRu_4(CO)_{12}BH_2$ is moderately air stable, existing in air in CH_2Cl_2 for several days with little decomposition. This is in contrast to the iron analogue,[12] $HFe_4(CO)_{12}BH_2$, which decomposes within minutes of exposure to the air.

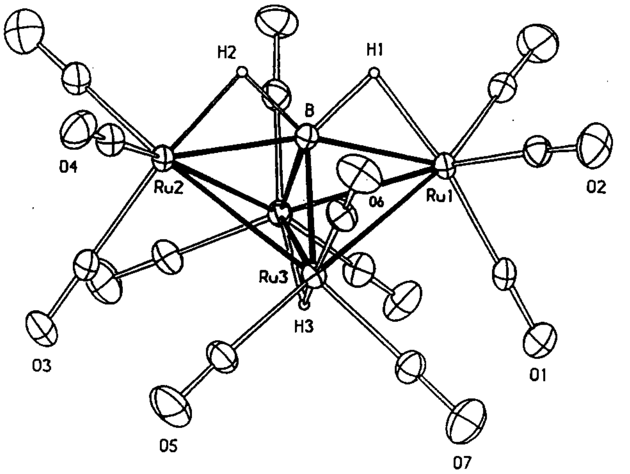


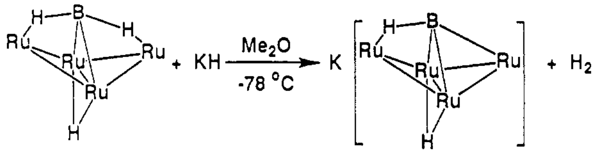
Figure 1. Molecular structure of $HRu_4(CO)_{12}BH_2$ (ORTEP plot with 30% probability ellipsoids).

The molecular structure of $HRu_4(CO)_{12}BH_2$ shown in Figure 1 consists of four $Ru(CO)_3$ groups arranged in a butterfly (dihedral) configuration, with the boron atom residing midway between the ruthenium atoms that define the wing tips. A crystallographically imposed mirror plane passes through Ru(1), B, and Ru(2). The molecule has pseudo-C_{2v} symmetry overall, with atoms B and H(3) residing on the 2-fold axis. This molecule is isostructural with $HFe_4(CO)_{12}BH_2$[12] and accordingly can be considered to be an arachno, four-atom cluster with an interstitial boron or alternatively a 62-electron complex in which the BH_2 ligand contributes five electrons. Selected bond distances and bond angles are shown in Tables III and IV, respectively.

The dihedral angle between the butterfly wings is 118°. It corresponds well with the value observed for $HFe_4(CO)_{12}BH_2$ (114°), and it is also consistent with the ideal dihedral angle, 109°, for a four-atom, butterfly, arachno cluster derived from an octahedron.[12]

^1H and ^{11}B NMR spectra (Experimental Section) are consistent with the solid-state molecular structure. The ^{11}B NMR spectrum consists of a triplet [113.5 ppm (t, J_{BH} = 65 Hz)], reflecting coupling with bridging hydrogens. The ^1H NMR spectrum consists of a quartet and a sharp singlet [−8.5 (q, 1:1:1:1, J_{BH} = 65 Hz), −21.15 ppm (s)]. The bridge hydrogens in the neutral molecule do not appear to be fluxional in the temperature range studied (25–70 °C).

Removal of an Ru–H–B hydrogen occurs in the following reaction:



(2)

$HRu_4(CO)_{12}BH_2$ $K[HRu_4(CO)_{12}BH]$

Ru = $Ru(CO)_3$

NMR spectra (Experimental Section) indicate that the proton is apparently removed from an Ru–H–B bond, consistent with the deprotonation of $HFe_4(CO)_{12}BH_2$[13a] to form $[HFe_4(CO)_{12}BH]^-$. The ^1H and ^{11}B NMR spectra of $[HRu_4(CO)_{12}BH]^-$ are in accord with recently reported spectra for this anion.[15]

Proton NMR studies of $[HRu_4(CO)_{12}BH]^-$ at elevated temperature suggest that the hydrides are not as fluxional as those in $[HFe_4(CO)_{12}BH]^-$[13] where rapid exchange is observed at 60 °C. In the case of $[HRu_4(CO)_{12}BH]^-$, the proton signals from Ru–H–B and Ru–H–Ru begin to broaden at 40 °C and nearly

---

(9) Abel, E. W.; Coston, T. P. J.; Orrel, K. G.; Sik, V.; Stephenson, D. *J. Magn. Reson.* **1986**, *70*, 34.

(10) Paquette, L. A.; Wang, T. Z.; Luo, J.; Cottrell, C. E.; Clough, A. E.; Anderson, L. B. *J. Am. Chem. Soc.* **1990**, *112*, 239.

(11) Johnson, B. F. G.; Johnston, R. D.; Lewis, L.; Robinson, B. H. *J. Chem. Soc. A* **1968**, 2856.

(12) (a) Wong, K. S.; Scheidt, W. R.; Fehlner, T. P. *J. Am. Chem. Soc.* **1982**, *104*, 1111. (b) Fehlner, T. P.; Housecroft, C. E.; Scheidt, W. R.; Wong, K. S. *Organometallics* **1983**, *7*, 2302.

(13) (a) Rath, N. P.; Fehlner, T. P. *J. Am. Chem. Soc.* **1987**, *109*, 5273. (b) Housecroft, C. E.; Buhl, M. L.; Long, G. J.; Fehlner, T. P. *J. Am. Chem. Soc.* **1987**, *109*, 3323.

collapse into the baseline as the temperature reaches 67 °C, but no new signal that would suggest an averaging of the resonances is observed. However, a 2-D NMR chemical exchange experiment at 37 °C indicates bridging hydrogen exchange with a rate constant of 1.1 s^{-1}.

Extended reaction (30 °C, 48 h) of $HRu_4(CO)_{12}BH_2$ with excess KH results in double deprotonation to form the dianion $[HRu_4(CO)_{12}B]^{2-}$, as evidenced by its NMR spectra. The second proton is apparently removed from the remaining Ru–H–B bond in contrast to Fehlner and co-workers' observation that in the formation of $[Fe_4(CO)_{12}BH]^{2-}$ from $[HFe_4(CO)_{12}BH]^-$ the second proton was apparently removed from the "hinge" Fe–H–Fe bond.[13] This result is in agreement with Fehlner's prediction that the dianion would result from the removal of both protons from the Ru–H–B bonds with the remaining hydrogen bridging the hinge Ru–Ru bond.[14] The ^1H NMR spectrum of $[HRu_4(CO)_{12}B]^{2-}$ consists of a sharp signal at −20.09 ppm, a typical region for Ru–H–Ru bridging hydrides.[5] In $[Fe_4(CO)_{12}BH]^{2-}$ the ^1H NMR spectrum has a broad signal at −8.7 ppm, consistent with an Fe–H–B bridging hydrogen. The ^{11}B NMR spectrum of $[HRu_4(CO)_{12}B]^{2-}$ consists of a broad singlet at 159 ppm, a difference of 18.1 ppm from the ^{11}B signal of the monoanion. The ^{11}B NMR spectrum of $[Fe_4(CO)_{12}BH]^{2-}$ consists of a singlet at 153 ppm, a difference of only 3 ppm from that of the monoanion, indicating that the loss of the second proton has little effect on the boron environment. A difference of 18 ppm from 140.9 to 159 ppm indicates that the removal of the second proton from $[HRu_4(CO)_{12}BH]^-$ has a much greater effect on the environment of the boron atom.

At elevated temperature the hydrogen in $[HRu_4(CO)_{12}B]^{2-}$ is fluxional. At 30 °C a new broad signal at −16.81 ppm is observed and the resonance at −20.09 ppm is broadened. At 50 °C the signal at −20.09 ppm is no longer apparent. No further changes occur in the spectrum at temperatures up to 65 °C, the highest temperature observed. Returning the temperature to 30 °C results in the reappearance of the signal at −20.09 ppm and the disappearance of the signal at −16.81 ppm, with some decomposition to $H_4Ru_4(CO)_{12}$,[5] as evidenced by the appearance of a signal at −17.78 ppm.

The apparent removal of the proton from the second Fe–H–B in the iron dianion to form $[Fe_3(CO)_{12}B]^{3-}$ results in the cluster becoming ^{11}B NMR silent.[13a] On the other hand, removal of the second Ru–H–B proton does not produce the same result.

Reprotonation of the mono- and dianion results in the re-formation of the neutral $HRu_4(CO)_{12}BH_2$ cluster in moderate yields. $H_4Ru_4(CO)_{12}$ was also found in the reprotonation products.

Acknowledgment. We thank the National Science Foundation for support of this work through Grant CHE 88-00515. FAB mass spectra and NMR spectra were obtained at The Ohio State University Chemical Instrumentation Center (funded in part by National Science Foundation Grant No. 79-10019 and National Institutes of Health Grant No. 1 S10 PRO140518-01A).

Supplementary Material Available: Listings of crystal data, bond distances, bond angles, positional parameters, and anisotropic thermal parameters (5 pages); a table of calculated and observed structure factor amplitudes (16 pages). Ordering information is given on any current masthead page.

---

(14) Fehlner, T. P. *Comments on Inorganic Chemistry*; Gordon & Breach Science Publishers Inc.: London, 1988; Vol. 7, No. 6, p 326.

(15) Chipperfield, A. K.; Housecroft, C. E.; Rheingold, A. L. *Organometallics* **1990**, *9*, 681.

Additions and Corrections

1988, Volume 27

Toru Amari, Shigenobu Funahashi, and Motoharu Tanaka*: Dilatometric Studies on Reaction Volumes for the Formation of Nickel(II) Complexes in Aqueous Solution.

Page 3369. The figure just below eq 2 should read 7.1 instead of −7.1.

Page 3370. In the last column in Table IV, the third and fourth entries should read 7.1 and 8.1, respectively, instead of −7.1 and −8.1.—Shigenobu Funahashi