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 ^{1}H 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 ^{1}H 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.

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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.

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(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