4274 *Inorg. Chem.* **1988**, *27*, 4274–4276

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

Preparation of $(\mu\text{-H})_3\mathrm{Os}_3(\mathrm{CO})_9(\mu_3\text{-C})(\mathrm{B}_5\mathrm{H}_8)$ and $(\mu_3\text{-H})_3\mathrm{Os}_3(\mathrm{CO})_9(\mu_3\text{-C})(\mathrm{C}_2\mathrm{B}_{10}\mathrm{H}_{11})$: "Alkylation" of Pentaborane(9) and 1,2-Dicarbadodecaborane by a Triosmium Methylidyne Cluster

Joseph R. Wermer, Deng-Yang Jan, Thomas D. Getman, Eric Moher, and Sheldon G. Shore*

Received August 3, 1988

In the presence of BF_3 the cluster complex $[(\mu\text{-H})_3\mathrm{Os}_3(\mathrm{CO})_9(\mu_3\text{-C})]_3(\mathrm{O}_3\mathrm{B}_3\mathrm{O}_3)$ undergoes Friedel–Crafts type reactions with $\mathrm{B}_5\mathrm{H}_9$ and with 1,2-$\mathrm{C}_2\mathrm{B}_{10}\mathrm{H}_{12}$ to form the boron-substituted complexes $(\mu\text{-H})_3\mathrm{Os}_3(\mathrm{CO})_9(\mu_3\text{-C})(\mathrm{B}_5\mathrm{H}_8)$ and $(\mu\text{-H})_3\mathrm{Os}_3(\mathrm{CO})_9(\mu_3\text{-C})(\mathrm{C}_2\mathrm{B}_{10}\mathrm{H}_{11})$, respectively. Substitution occurs at the apical site of the B_5 square pyramid of the pentaborane(9) derivative, the site expected to be most susceptible to electrophilic attack. The carbon–boron bond is readily hydrolyzed, and the triosmium methylidyne cluster $(\mu\text{-H})_3\mathrm{Os}_3(\mathrm{CO})_9(\mu_3\text{-CH})$ is formed. In the case of the carborane derivative, substitution is believed to occur at the 9-B site. This compound is resistant to hydrolysis, and it appears to be stable in air. There is no apparent reaction between 1,7-$\mathrm{C}_2\mathrm{B}_{10}\mathrm{H}_{12}$ and $[(\mu\text{-H})_3\mathrm{Os}_3(\mathrm{CO})_9(\mu\text{-C})]_3(\mathrm{O}_3\mathrm{B}_3\mathrm{O}_3)$ in the presence of BF_3.

Introduction

The complex $[(\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu_3\text{-C})]_3(\mathrm{O}_3\mathrm{B}_3\mathrm{O}_3)$ (**1**) has a molecular structure that consists of a trioxyboroxin unit $(\mathrm{O}_3\mathrm{B}_3\mathrm{O}_3)$ to which three triosmium methylidyne cluster units $((\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu_3\text{-C}\text{–})$ are bound[1] (Figure 1). This compound is a potentially useful reagent in the preparation of derivatives of triosmium methylidyne clusters. Its reactions with BCl_3 and BBr_3 in $\mathrm{CH}_2\mathrm{Cl}_2$ produce the halo complexes $(\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu_3\text{-CX})$ (X = Cl (**2**), Br (**3**)).[1] These reactions are believed to occur through a sequence that involves initial coordination of the boron halide to the oxygen of the C–O–B unit followed by transfer of halogen to the carbon atom with rupture of the carbon–oxygen bond.[2-4]

Complex **1** reacts with benzene in the presence of BF_3 to form the triosmium phenylmethylidyne cluster $(\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu\text{-CPh})$ (**4**)[1] via a Friedel–Crafts type reaction. In view of the known ability of some boron hydrides[5] and carboranes[6] to undergo Friedel–Crafts reactions with electrophilic displacement of B–H hydrogen, it seemed that **1** might be useful in linking a borane or a carborane with the triosmium methylidyne cluster unit $(\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu_3\text{-C}\text{–})$ through the formation of a carbon–boron bond. We report here results from studies of the reaction of **1** with the boron hydride $\mathrm{B}_5\mathrm{H}_9$ and the carboranes 1,2-$\mathrm{C}_2\mathrm{B}_{10}\mathrm{H}_{12}$ and 1,7-$\mathrm{C}_2\mathrm{B}_{10}\mathrm{H}_{12}$ in the presence of BF_3.

Results and Discussion

$(\mu\text{-H})_3\mathrm{Os}_3(\mathrm{CO})_9(\mu_3\text{-C})(\mathrm{B}_5\mathrm{H}_8)$. In the presence of BF_3, pentaborane(9), $\mathrm{B}_5\mathrm{H}_9$, is "alkylated" by the $(\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu_3\text{-C}\text{–})$ unit of **1** to form $(\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu_3\text{-C})(\mathrm{B}_5\mathrm{H}_8)$ (**5**) (reaction 1).

$$[(\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu_3\text{-C})]_3(\mathrm{O}_3\mathrm{B}_3\mathrm{O}_3) + \mathrm{B}_5\mathrm{H}_9 \xrightarrow{\mathrm{BF}_3} \underset{\mathbf{5}}{(\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu_3\text{-C})(\mathrm{B}_5\mathrm{H}_8)} \qquad (1)$$

Synthesis of **5** was achieved by stirring a slurry of **1** in liquid $\mathrm{B}_5\mathrm{H}_9$ in the presence of BF_3. It was isolated in 33% yield. Equivalent yields of **5** were also obtained with $\mathrm{CH}_2\mathrm{Cl}_2$ as a solvent. Another route to **5** is the reaction of the triosmium chloromethylidyne cluster **2** with $\mathrm{B}_5\mathrm{H}_9$ in the presence of AlCl_3; however, the preferred synthesis is given by reaction 1 because of the easy preparation of **1** compared to the preparation of **2**. Isolated as a light orange solid, **5** appears to be stable at room temperature under vacuum but it reacts with the moisture in the air to form the triosmium methylidyne cluster $(\mu\text{-H})_3(\mathrm{CO})_9\mathrm{Os}_3(\mu_3\text{-CH})$ (**6**). In glacial acetic acid **5** is cleanly converted to **6**. Complex **5** is soluble in $\mathrm{CH}_2\mathrm{Cl}_2$ and CHCl_3; it is slightly soluble in hexane.

FT-ICR mass spectra of **5** reveal a parent mass of 906 (calculated *m*/*e* for ${}^{11}\mathrm{B}_5{}^{12}\mathrm{C}_{10}{}^{1}\mathrm{H}_{11}{}^{16}\mathrm{O}_9{}^{192}\mathrm{Os}_3$: 906) and a fragmentation pattern that shows sequential separation of $\mathrm{B}_5\mathrm{H}_8$ and CO groups from the $\mathrm{Os}_3\mathrm{C}$ cluster core. A proposed structure for **5** (Figure 2) that contains an apically substituted pentaborane(9) unit is readily deduced from its NMR spectra. The doublet in the ^{11}B NMR spectrum at −12.2 ppm ($J_{\mathrm{B-H}}$ = 140 Hz) is assigned to the four basal boron atoms of the B_5 square pyramid, while the singlet at −25.3 ppm is assigned to the apical boron atom. Upon proton spin decoupling, the doublet collapses to a singlet. The ^{1}H NMR spectrum of **5** consists of a signal at −19.23 ppm, assigned to the Os–H–Os bridging hydrogens, a broad single resonance at −0.86 ppm, which is assigned to the B–H–B bridging hydrogens, and a 1:1:1:1 quartet at 3.06 ppm ($J_{\mathrm{B-H}}$ = 140 Hz), due to the terminal basal B–H hydrogens.

Boron-11 NMR spectra of mixtures from reaction 1 indicate that **5** is the principal product in the reaction. The only other product in the reaction mixture that contains B–H bonds is present in significantly smaller concentration. Its ^{11}B NMR spectrum consists of two signals in a 1:1 ratio (d, −12.9 ppm (coupling unresolved due to overlapping resonances); d, −48.4 ppm ($J_{\mathrm{B-H}}$ = 172 Hz)), and its 2D ${}^{11}\mathrm{B}$–${}^{11}\mathrm{B}$ NMR spectrum reveals that the nuclear spins of these boron atoms are spin-coupled. This second B–H-containing product was not identified because it could not be separated from the reaction mixture. It is unlikely that this product is an isomer of **5** in which substitution occurs at a basal site of the B_5 square pyramid. Such an isomer is expected to have a ^{11}B NMR spectrum consisting of a high-field doublet (1 B) and three lower field signals of a singlet (1 B), doublet (1 B), and doublet (2 B). Other boron-containing species in the reaction

---

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

(2) Keister, J. B.; Horling, T. L. *Inorg. Chem.* **1980**, *19*, 2304.

(3) Lappert, M. F. *J. Chem. Soc.* **1958**, 3256.

(4) Dolby, R.; Robinson, H. B. *J. Chem. Soc., Dalton Trans.* **1972**, 2046.

(5) (a) Dupont, J. A.; Hawthorne, M. F. *J. Am. Chem. Soc.* **1962**, *84*, 1804. (b) Hillman, M. *J. Inorg. Nucl. Chem.* **1960**, *12*, 383. (c) Blay, N. J.; Dunstan, I.; Williams, R. L. *J. Chem. Soc.* **1960**, 430. (d) Schaeffer, R.; Schoolery, J. N.; Jones, R. J. *J. Am. Chem. Soc.* **1958**, *80*, 2670. (e) Figgis, B. N.; Williams, R. L. *Spectrochim. Acta* **1959**, *15*, 331. (f) William, R. L.; Dunstan, I.; Blay, N. J. *J. Chem. Soc.* **1960**, 5006. (g) Onak, T.; Williams, R. E. *Inorg. Chem.* **1962**, *1*, 106. (h) Ryschkewitsch, G. E.; Harris, S. W.; Mezey, E. J.; Sisler, H. H.; Weilmuenster, E. A.; Garrett, A. B. *Inorg. Chem.* **1963**, *2*, 890. (i) Onak, T.; Dunks, G. B. *Inorg. Chem.* **1964**, *3*, 1060. (j) Burg, A. B.; Sandhu, J. S. *J. Am. Chem. Soc.* **1965**, *87*, 3787. (k) Zakharkin, L. I.; Kalinin, V. N. *J. Gen. Chem. USSR* (*Engl. Transl.*) **1966**, *36*, 2154. (l) Gaines, D. *J. Am. Chem. Soc.* **1966**, *88*, 4528. (m) Gaines, D.; Martens, J. A. *Inorg. Chem.* **1968**, *7*, 704. (n) Lipscomb, W. N.; *Boron Hydrides*; Benjamin: New York, 1965; pp 171, 180. (o) Holzman, R. T.; Hughes, R. L.; Smith, I. C.; Lawless, E. W. *Production of the Boranes and Related Research*; Academic: New York, 1967; pp 146–177.

(6) (a) Grimes, R. N. *Caroboranes*; Academic: New York, 1970; p 172. (b) Beall, H. *Boron Hydride Chemistry*; Muetterties, E. L., Ed.; Academic: New York, 1975; p 319. (c) Onak, T. *Ibid.*, pp 356, 375. (d) J. A. Potenza; Lipscomb, W. N.; Vickers, G. D.; Schroeder, H. *J. Am. Chem. Soc.* **1966**, *88*, 628. (e) Zakharkin, L. I.; Kalinin, V. N.; Lozovskaya, V. S. *Bull. Acad. Sci. USSR, Div. Chem. Sci.* (*Engl. Transl.*) **1968**, 1683.

0020-1669/88/1327-4274$01.50/0

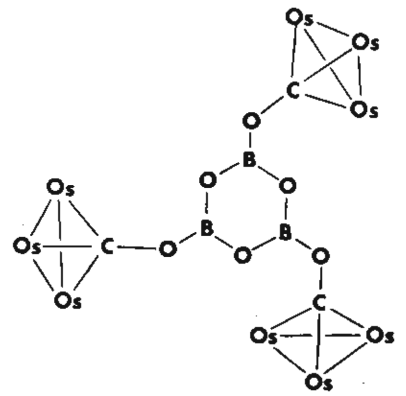

Figure 1. Skeletal arrangement in $[(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-C})]_3(O_3B_3O_3)$ (**1**).

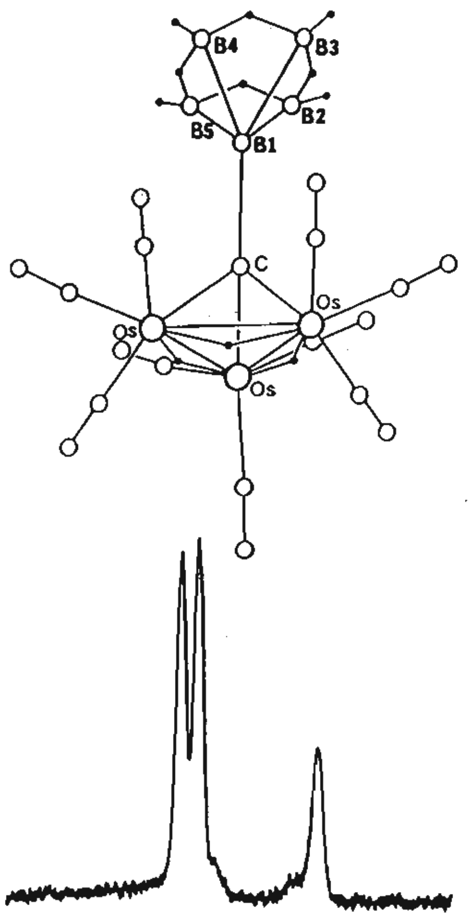

Figure 2. Boron-11 NMR spectrum and proposed structure of $(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})(B_5H_8)$ (**5**).

mixture have resonances at 0.3 ppm, assigned to a species containing BF_3 bound to an oxygen of an ether-like ligand, and at 19.7 ppm, assigned to a boron atom that is in a trigonal environment surrounded by three oxygen atoms,[7] possibly a trioxyboroxin unit $(O_3B_3O_3)$.

Formation of the carbon-boron bond at the apical atom[5c,h] of the B_5 cluster provides support for the synthesis of **5** through a Friedel-Crafts type reaction in which an expected directive effect involves electrophilic attack at the most negative boron atom in B_5H_9,[5n] the apical boron atom. It is suggested that BF_3 assists the reaction by binding to the oxygen on the methylidyne carbon with subsequent heterolytic cleavage to form a cluster carbonium ion or perhaps by causing extreme polarization of the C-O bond

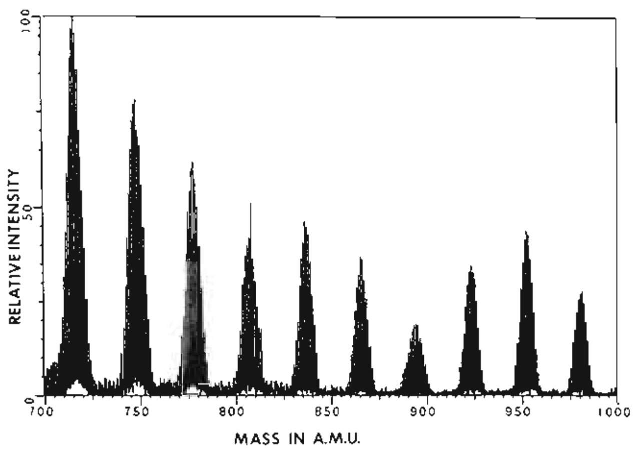

Figure 3. FT-ICR mass spectrum of $(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})(C_2B_{10}H_{11})$ (**7**).

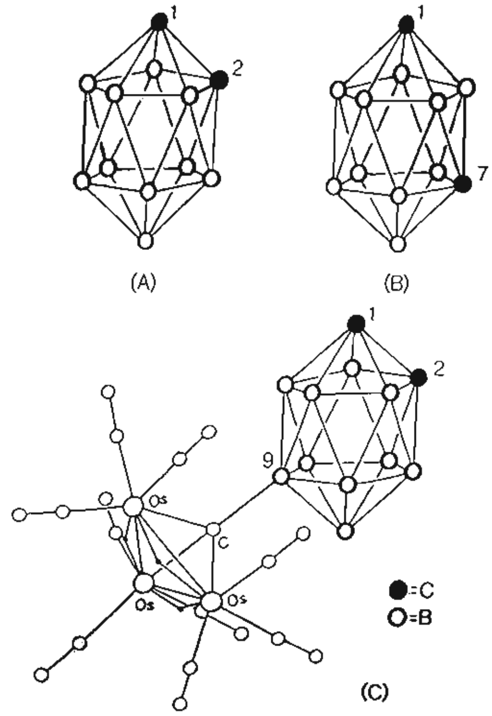

Figure 4. (A) Structure of $1,2\text{-}C_2B_{10}H_{12}$, (B) structure of $1,7\text{-}C_2B_{10}H_{12}$, and (C) proposed structure of $(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})(C_2B_{10}H_{11})$ (**7**). The hydrogen atoms of the carboranes have been omitted for clarity.

to facilitate the Friedel-Crafts type reaction.[4,8]

$(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})(C_2B_{10}H_{11})$. The carborane $1,2\text{-}C_2B_{10}H_{12}$ also reacts with **1** in the presence of BF_3 to form the boron-substituted complex $(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})(C_2B_{10}H_{11})$ (**7**) (reaction 2). On the other hand, there is no reaction, (3), between 1,7-$C_2B_{10}H_{12}$ and **1** in the presence of BF_3. On the basis of analytical HPLC only one isomer, **7**, was produced in reaction 2. It was isolated as a pure material in 32% yield and is soluble in CH_2Cl_2, $CHCl_3$, and acetone.

$$[(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})]_3(O_3B_3O_3) + 1,2\text{-}C_2B_{10}H_{12} \xrightarrow{BF_3} \underset{\mathbf{7}}{(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})(C_2B_{10}H_{11})} \quad (2)$$

$$[(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})]_3(O_3B_3O_3) + 1,7\text{-}C_2B_{10}H_{12} \xrightarrow{BF_3} \text{no reaction} \quad (3)$$

The FT-ICR mass spectrum of **7** reveals the parent ion mass of 988 (calculated m/e for ${}^{11}B_{10}{}^{12}C_{12}{}^{1}H_{14}{}^{16}O_9{}^{192}Os_3$: 988).

---

[7] Bell, R. P.; Edwards, J. O.; Jones, R. B. *The Chemistry of Boron and its Compounds*; Muetterties, E. L., Ed.; Wiley: New York, 1967; p 214.

[8] Seyferth, D.; Hallgren, J. E.; Eschbach, C. S. *J. Am. Chem. Soc.* **1974**, *96*, 1730.

Successive loss of the nine carbonyls from the Os_3 framework was observed in the mass spectrum, leaving a fragment that consists of the carborane attached to the Os_3C core (Figure 3). On the other hand, for complex **5** the parent ion appears in high abundance only at low accelerating potential (9 eV) and the initial fragmentation appears to involve scission of the methylidyne carbon–boron bond. The C–B bond in **7** is much more resistant to hydrolysis than that in **5**. Solid **7** appears to be stable in air.

The structure of **7** could not be deduced from NMR spectra (Experimental Section) because of overlapping resonances that could not be resolved, even by the use of partial relaxation experiments. However, we can argue for a most likely structure, the 9-B substituted isomer shown in Figure 4. The favored site for electrophilic substitution of carboranes is the boron atom farthest from carbon.[6] In electrophilic monohalogenation (Cl_2, Br_2, or I_2 in the presence of an aluminum halide), substitution occurs predominantly at boron atom 9(12) in 1,2-$C_2B_{10}H_{12}$ and at boron atom 9(10) in 1,7-$C_2B_{10}H_{12}$, the boron atoms farthest from both carbons in these carborane molecules.[6e] Selectivity increases with decreasing strength of the electrophile.[6e] The absence of reaction between **1** and 1,7-$C_2B_{10}H_{12}$ under identical conditions in which **1** reacts with 1,2-$C_2B_{10}H_{12}$ suggests that **1** is a weak electrophile compared to Cl_2, Br_2, and I_2. Since we obtain only one isomer of **7**, it is assumed to be the 9-B-substituted isomer.

Experimental Section

Materials. Syntheses in the literature were employed for the preparation of $(\mu\text{-H})_2Os_3(CO)_{10}$,[9] 1,2-$C_2B_{10}H_{12}$,[10a,b] 1,7-$C_2B_{10}H_{12}$,[10c] and diborane(6).[11] Pentaborane(9) (Callery Chemical Co.) was used as received. Boron trifluoride was obtained from the Matheson Chemical Co. It was passed through a −140 °C trap prior to use and stored in a Pyrex tube with a ground-glass stopcock at −196 °C. Methylene chloride was distilled from CaH_2 and stored over P_2O_5 until use. Hexane was purified according to the literature procedure[12] and then distilled from CaH_2 and stored in an evacuated bulb until use. Boron-11 NMR ($\delta(BF_3\cdot OEt_2)$ = 0.00 ppm) and proton NMR (δ(TMS) = 0.00 ppm) spectra were obtained at The Ohio State University Chemical Instrument Center on a Bruker MSL-300 NMR spectrometer at 96.3 and 300 MHz, respectively. Fourier transform ion cyclotron resonance (FT-ICR) mass spectra were obtained at The Ohio State University Chemical Instrument Center on a Nicolet MS1000 spectrometer.

$(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})(B_5H_8)$. A 10-mL reaction vessel was loaded with 117.1 mg of $H_2Os_3(CO)_{10}$ (0.137 mmol) and a Teflon-coated magnetic stir bar. The vessel was evacuated on the vacuum line, and 2 mL of dry CH_2Cl_2 was condensed into the vessel at −196 °C. This was followed by 0.548 mmol of THF and 0.0687 mmol of B_2H_6, both measured as gases. The vessel was warmed to room temperature and the mixture was stirred for 5 h. After about 1 h, complex **1** had begun to precipitate. After 5 h, the volatile components were removed by continuous pumping for 1 h. The vessel was cooled to −196 °C, and 1.0 mL of B_5H_9 (ca. 10 mmol) and 0.274 mmol of BF_3 were condensed into it. The vessel was returned to room temperature, and the mixture was stirred for 40 h. The resultant mixture consisted of an orange solution and an off-white precipitate. Only 0.08 mmol of noncondensable gas was evolved. The volatile materials were removed by pumping for 1 h. In the drybox, the reaction flask was attached to an extractor. The extraction apparatus was attached to the vacuum line, and hexane was distilled onto the product at −196 °C. The vessel was warmed to room temperature, and the compound was washed with hexane. Then the vessel was placed in the drybox, and a new collection flask was attached. Next, the extraction apparatus was attached to the vacuum line, and CH_2Cl_2 was condensed onto the product at −196 °C. The vessel was warmed to room temperature, the product was dissolved in the CH_2Cl_2, and the resulting solution was filtered, leaving behind boric oxide on the frit. The methylene chloride soluble product, **1**, was isolated as a light orange solid in 33% yield (41 mg, 0.045 mmol) and identified as $(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})(B_5H_8)$ as described under Results and Discussion. IR spectrum (in CH_2Cl_2): ν_{BH} = 2599 (w) cm^{-1}; ν_{CO} = 2005 (s), 2013 (s), 2019 (s), 2074 (s), 2079 (s) cm^{-1}; unassigned = 1359 (w), 1409 (w), 1430 (w) cm^{-1}.

Alternatively, **5** was prepared in the following manner. From 256.0 mg of $H_2Os_3(CO)_{10}$ (0.29 mmol), cluster **1** was prepared according to the procedure above. After the volatiles were removed by pumping, CH_2Cl_2 (2 mL), B_5H_9 (0.87 mmol), and BF_3 (0.58 mmol) were condensed into the vessel at −196 °C. The vessel was warmed to room temperature and stirred for 40 h. The volatiles were removed by pumping for 1 h. In the drybox, the reaction vessel was attached to an extractor and the solid products were washed with hexane. The vessel was taken into the drybox, and a new collection flask was attached. The residue on the frit was dissolved in CH_2Cl_2 and the solution filtered, leaving behind boric oxide on the frit. The methylene chloride soluble compound **5** was isolated in 32% yield (83.7 mg, 0.093 mmol).

Reaction of $(\mu\text{-H})_3(CO)_9Os_3(\mu\text{-C})(B_5H_8)$ with Glacial Acetic Acid. An excess of glacial acetic acid, ca. 2 mL, was condensed into a 15-mL round-bottom flask containing 45.0 mg of **5** (0.050 mmol) at −78 °C. The solution was then allowed to warm to room temperature and was heated to 80 °C for 24 h. Volatile components were pumped away, leaving behind a light yellow residue which was then recrystallized from CH_2Cl_2/hexane to give $(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-CH})$ in 90% yield (37.7 mg, 0.045 mmol).

$(\mu\text{-H})_3(CO)_9Os_3(\mu_3\text{-C})(C_2B_{10}H_{11})$. From 187.5 mg of $H_2Os_3(CO)_{10}$ (0.220 mmol), complex **1** was prepared according to the procedure above. After the volatiles had been removed by pumping, the vessel was taken into the drybox and charged with 32.0 mg of 1,2-$C_2B_{10}H_{12}$ (0.226 mmol). The vessel was removed from the drybox, evacuated on the vacuum line, and cooled to −196 °C. Dry CH_2Cl_2 (1–2 mL) was condensed into the reaction vessel along with 0.440 mmol of BF_3. The vessel was allowed to return to room temperature with stirring. The light yellow precipitate gradually disappeared, forming an initially clear, orange solution. After a few minutes, an off-white precipitate began to appear. Over a period of 3 days the amount of precipitate became noticeably larger. After the mixture was stirred for 6 days, the volatile components were removed and the vessel was taken into the drybox and attached to an extractor. The orange CH_2Cl_2 solution was filtered, leaving behind a slightly soluble (in CH_2Cl_2) light yellow material. In the air this material was dissolved in acetone and eluted from a silica gel column with acetone and methylene chloride. Yield: 72 mg (0.073 mmol, 32% based upon $H_2Os_3(CO)_{10}$). IR spectrum (KBr pellet): ν_{BH} = 2607 (w) cm^{-1}; ν_{CO} = 1998 (s, br), 2073 (s) cm^{-1}; unassigned = 602 (w), 696 (w, sh), 1084 (w), 1093 (w), 1106 (w), 1109 (w), 1119 (w) cm^{-1}. Boron-11 NMR spectrum: 0.97 (J = 129 Hz), −5.71 (J = 148 Hz), −10.65 (J = 164 Hz), −13.73 ppm (J = 151 Hz). Proton NMR spectrum: −19.23 ppm (s, Os–H–Os); B–H region not resolved.

Acknowledgment. This work was supported by the National Science Foundation through Grant No. CHE 84-11630. We thank Dr. Steven Mullen and Dr. Alan Marshall for obtaining FT-ICR mass spectra. E.M. thanks the National Science Foundation for support as an undergraduate summer research participant through Grant No. NSF-REU CHE 87-13117. NMR spectra were obtained at The Ohio State University Chemical Instrument Center (funded in part by National Science Foundation Grant No. 79-10019 and National Institutes of Health Grant No. 1 S10 PR0140518-01A).

---

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

(10) (a) Heying, T. L.; Ager, J. W., Jr.; Clark, S. L.; Mangold, D. J.; Goldstein, H. L.; Hillman, M.; Polak, R. J.; Szymanski, J. W. *Inorg. Chem.* **1963**, *2*, 1089. (b) Fein, M. M.; Bobinski, J.; Mayers, N.; Schwartz, N.; Cohen, M. S. *Inorg. Chem.* **1963**, *2*, 1111. (c) Papetti, S.; Obenland, C., Heying, T. L. *Ind. Eng. Chem., Prod. Res. Dev.* **1966**, *5*, 334.

(11) Toft, M. A.; Leach, J. B.; Himpsl, F. L.; Shore, S. G. *Inorg. Chem.* **1982**, *21*, 1952.

(12) Jolly, William L. *The Synthesis and Characterization of Inorganic Compounds*; Prentice-Hall: Englewood Cliffs, NJ, 1970; p 119.