Table II. Bond Distances (Å) and Angles (deg) in the Coordination Sphere

| | | | |
|---|---|---|---|
| **Bond Distances** | | | |
| Pd-N(1) | 2.038 (7) | Pd-N(2) | 2.037 (7) |
| Pd-C(8) | 2.190 (9) | Pd-C(20) | 2.172 (9) |
| **Bond Angles** | | | |
| N(1)-Pd-N(2) | 171.4 (3) | N(2)-Pd-C(8) | 97.1 (3) |
| N(1)-Pd-C(8) | 84.5 (3) | N(2)-Pd-C(20) | 83.9 (3) |
| N(1)-Pd-C(20) | 96.4 (3) | C(8)-Pd-C(20) | 168.2 (3) |
| Pd-N(1)-C(1) | 119.3 (6) | Pd-N(2)-C(13) | 120.3 (7) |
| Pd-N(1)-C(5) | 120.3 (6) | Pd-N(2)-C(17) | 119.7 (6) |
| Pd-C(8)-C(7) | 109.9 (6) | Pd-C(20)-C(19) | 107.4 (6) |
| Pd-C(8)-C(9) | 100.1 (6) | Pd-C(20)-C(21) | 103.1 (7) |
| Pd-C(8)-C(10) | 103.8 (6) | Pd-C(20)-C(22) | 107.5 (6) |

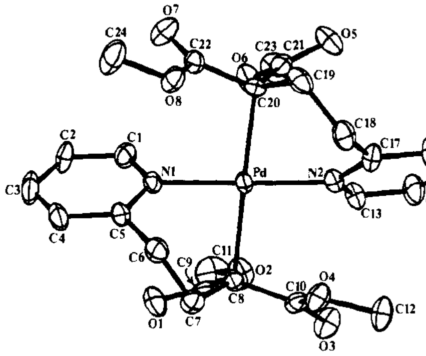

Figure 1.

an empirical absorption correction, based upon $\psi$ scans of reflections near $\chi = 90°$; the minimum relative transmission coefficient was 0.963. Of 3410 unique data measured 1909 had $I > 3\sigma(I)$ and were used in the refinement.

The structure was solved by heavy-atom methods. Refinement was accomplished by full-matrix least squares based on $F$ with weights $w = \sigma^{-2}(F_o)$. All calculations were carried out on a PDP 11/34 computer using the Enraf-Nonius Structure Determination Package programs.[13] All non-hydrogen atoms were located from difference maps and included as fixed contributions with isotropic $B = 5.0$ Å$^2$ (7.0 Å$^2$ for methyl groups). At convergence, $R = 0.035$, $R_w = 0.041$ for 316 variables. A final difference map exhibited no peaks larger than 0.36 e Å$^{-3}$. Final coordinates are listed in Table I; important distances and angles are given in Table II.

The central Pd atom in **5** is $\sigma$ bonded with two carbon atoms and coordinated by two pyridine nitrogens corroborating the net overall square-planar trans configuration and nearly $C_2$ symmetry (Figure 1). The pyridine rings are tipped by 62.9° (av) out of the coordination plane, as compared to 31.5° in **3**.[10] In both **3** and **5**, the alkyl bridges swing upward on the *same* side of the mean coordination plane. Palladium and the coordinating atoms [N(1,2); C(8,10)] form an approximate plane with displacements less than ±0.2 Å, with the metal atom nearly in the best plane. Neither the Pd–N (average 2.038 Å) nor Pd–C (average 2.181 Å) bond lengths are unusual; all are similar to those found in analogous five-membered ring complexes.[10] Intraannular N–Pd–C angles average 84.2°, over 4° larger than those found in **3**. The pyridine rings are planar and exhibit normal angles and bond distances. The six-membered cyclic ring, however, adopts a twist boat conformation.

The six-membered palladacycle **5** is different from previous examples in that it has *two* (rather than one) trans six-membered cyclometalated rings and most were acetate or halo-bridged dimers.[5-7] It thus appears that the original assumption, that metal ions preferring square-planar geometry favor five-membered rings, is unnecessarily limiting. Six-membered rings can readily accommodate the square-planar central metal; in fact, when compared with analogous five-membered ring complexes, no unusual or unfavorable geometries or interactions can be found. Further, **5** is prepared by identical reaction conditions and in similar yields to the five-membered ring counterparts. Studies in competitive complexation as well as in higher order cyclometalated complexes are in progress.

**Acknowledgment.** We wish to thank the National Science Foundation for partial support of this work and Reilly Tar for generous samples of 2-vinylpyridine as well as Johnson Matthey Inc. for inclusion in their metals loan program.

**Registry No.** 4, 86480-63-9; 5, 86480-64-0; $PdCl_2$, 7647-10-1; dimethyl sodiomalonate, 108-59-8; 2-vinylpyridine, 100-69-6.

**Supplementary Material Available:** Tables of coordinates for hydrogen atoms, bond distances and bond angles, anisotropic thermal parameters, and structure factors and experimental details (22 pages). Ordering information is given on any current masthead page.

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[13] Frenz, B. A.; Okaya, Y. "Enraf-Nonius Structure Determination Package"; Enraf-Nonius: Delft, Holland, 1980.

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# Linked Bis($\mu$-phosphido) and Related Ligands for Metallic Clusters. 1. Application to the Hexacarbonyldiiron Moiety

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*Received April 29, 1983*

*Summary:* Three bis-bridged diiron species, $\mu,\mu^1$-(1-X-2-Y-benzene)hexacarbonyldiiron [X = Y = PPh (**3a**); X = S, Y = PPh (**3b**); X = Y = S (**3c**)], have been prepared by the thermal reaction of the corresponding dihydro forms of the ligand and excess nonacarbonyldiiron. Reduction of **3a–c** with 2 equiv of electrons gives rise to a dianion in which cleavage of an Fe–X bond has occurred in contrast to the reductive behavior of $(\mu\text{-}PPh_2)_2Fe(CO)_6$ which cleaves an Fe–Fe bond. The dianion derived from **3a** reacts with excess trifluoracetic acid to give the phosphorus and iron-protonated species. Methylation (MeI) of $(\mathbf{3a})^{2-}$ gave a complex with the structure assigned as $[1\text{-}P(Me)Ph\text{-}\mu\text{-}2\text{-}PPhC_6H_4][\mu\text{-}C(O)Me](CO)_5Fe_2$. An analogous complex was obtained on methylation of $(\mathbf{3b})^{2-}$.

Phosphido-bridged transition-metal complexes have attracted considerable attention recently owing to the enhanced stability of the phosphido bridge relative to the traditional tertiary phosphine ligand.[1] In particular, it has been hoped that special stability could be accorded to various phosphido-bridged bimetallic and cluster complexes and ultimately lead to the development of new transition-metal catalysts.

Although bis(phosphido)-bridged bimetallic complexes with the general structure A (X = Y = $PR_2$) have been

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[1] Finke, R. G.; Gaughan, G.; Pierpont, C.; Cass, M. E. *J. Am. Chem. Soc.* 1981, *103*, 1394 and references therein.

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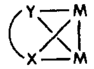
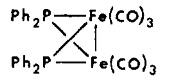
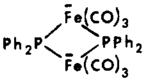
A


B

prepared,[2] few complexes in which the phosphido moieties are bound to the transition metal in both a bridging and chelating manner, as in structure B (X = Y = PR), are known.[3] Linked species are anticipated to be more stable with respect to fragmentation or dissociation of the phosphorus groups as well as conformationally more rigid than analogous compounds with structure A. We now report the convenient synthesis of a compound with linked phosphido bridges (type B) and the demonstration of a pronounced difference in its chemistry compared to that of the analogous compound with structure A.

The bis($\mu$-phosphido)diiron complex **1** has been prepared either by the thermal reaction of pentacarbonyliron and tetraphenyldiphosphine[2c] or by the reaction of disodium tetracarbonylferrate with chlorodiphenylphosphine.[2d] The corresponding dianion **2** has also been described.[2e] We report here the preparation of the benzo-linked species **3a**, analogous to **1**, from the reaction of


1


2

*o*-bis(phenylphosphino)benzene with excess nonacarbonyldiiron (Scheme I).[4a] Complex **3a**, an air-stable, golden yellow solid, was isolated in 58% yield after chromatography of the reaction mixture on alumina.[4b] Consistent with the assigned structure in which the phosphorus atoms bear no hydrogen atoms and each phosphorus atom is coordinated to two iron atoms, the $^1$H-coupled $^{31}$P NMR spectrum of **3a**, consists of only a singlet at $\delta$ 138.2. Additionally, the presence of only aromatic hydrogen absorptions in the $^1$H NMR spectrum is consistent with the absence of any iron hydride bonding.

The bonding in bridged binuclear complexes has been the subject of theoretical investigations, particularly that between the two metals in the neutral, as well as in the reduced state.[5] With regard to compound **1**, X-ray crystallographic data verified the prediction of a nonplanar molecule with a bent Fe–Fe bond for the neutral species and the prediction of a planar molecule with no Fe–Fe bond for the dianionic species **2**.[2e] Cleavage of the Fe–Fe bond upon addition of two electrons to **1** is understandable in view of calculations indicating that the LUMO for neutral **1** is largely Fe–Fe antibonding.[5] The conformational change upon reduction, resulting in the planarity of the dianion, is apparently a reflection of the minimization of electron repulsion between the two iron atoms, as well as between the phosphorus and iron atoms.

In view of the similarity of the structures of complexes **1** and **3a**, dianion formation analogous to that observed for **1** might initially be anticipated for complex **3a**. The linking of the two phosphido groups by a benzo bridge, however, provides a geometric constraint such that a dianion formed by cleavage of the Fe–Fe bond in **3a** cannot possess a planar $P_2Fe_2^{2-}$ backbone. The chemical importance of this inhibition to planarity proves to be great and leads to the exhibition by **3a** of a reductive course distinct from that observed for **1**.

Treatment of **3a** in THF with either excess sodium or 2 equiv of sodium naphthalenide yielded a burgundy red solution of a dianion that was readily transformed back to **3a** upon exposure to air. The infrared spectrum[6] of the dianion with a bridging carbonyl absorption at 1680 cm$^{-1}$ was distinct from that of **2** and strongly suggested that the two dianions possessed fundamentally different structures.[7] The $^{31}$P NMR spectrum (THF), which featured two doublets at $\delta$ 193.5 and 32.1 ($J_{P-P}$ = 4 Hz), eliminated from consideration a symmetrical species analogous to **2** and suggested structure **4a**, the result of cleavage of a Fe–P bond. Consistent with structure **4a**, the addition of 1 equiv of acetic or trifluoroacetic acid to the dianion gave a monoanion whose $^1$H-coupled $^{31}$P NMR spectrum indicated protonation at phosphorus ($^1J_{P-H}$ = 334 Hz). The monoanion is believed to have structure **5** based on the presence of a bridging carbonyl absorption (1680 cm$^{-1}$) in its infrared spectrum.[8] The addition of a further equivalent of acid gave an air-sensitive species tentatively assigned structure **6**, in view of its IR spectrum and somewhat poorly resolved $^1$H NMR absorption for Fe–H at $\delta$ −11.1 (d, $^2J_{P-H}$ = 56 Hz).[9]

Methylation of **4a** ($CH_3I$, THF, 25 °C) also first occurs at phosphorus, but in contrast to the course of protonation, the second methyl group ultimately is bound in the product to the carbon of a carbonyl group, giving the pale orange complex **7a**, as revealed by $^1$H NMR, $^{13}$C NMR, and infrared spectroscopy.[10] As observed for **5** and **6**, complex **7a** exists in isomeric forms based upon the $^{31}$P and $^1$H NMR spectra.

The unusual reductive cleavage observed for **3a** was similarly observed for the $\mu$-thio analogues **3b** and **3c**.[11]

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[2] (a) Hayter, R. G. *J. Am. Chem. Soc.* **1964**, *86*, 823. (b) Yasufuku, K.; Yamazaki, H. *Bull. Chem. Soc. Jpn.* **1970**, *43*, 1588. (c) Job, B. E.; McLean, R. A. N.; Thompson, D. T. *Chem. Commun.* **1966**, 895. (d) Collman, J. P.; Rothrock, R. K.; Finke, R. G.; Rose-Munch, F. *J. Am. Chem. Soc.* **1977**, *99*, 7381. (e) Ginsburg, R. E.; Rothrock, R. K.; Finke, R. G.; Collman, J. P.; Dahl, L. E. *Ibid.* **1979**, *101*, 6550. (f) Ginsburg, R. E.; Berg, J. K.; Rothrock, R. K.; Collman, J. P.; Hodgson, K. O.; Dahl, L. F. *Ibid.* **1979**, *101*, 7218. (g) Collman, J. P.; Rothrock, R. K.; Finke, R. G.; Moore, E. J.; Rose-Munch, F. *Inorg. Chem.* **1982**, *21*, 146. (h) Vahrenkamp, H. *Angew. Chem., Int. Ed. Engl.* **1978**, *17*, 379. (i) Carty, A. J. *Adv. Chem. Ser.* **1982**, *No. 196*, 163. (j) Breen, M. J.; Geoffroy, G. L. *Organometallics* **1982**; *1*, 1437 and references therein. (k) Meek, D. W.; Waid, R.; Tau, K. D.; Kirschner, R. M.; Morimoto, C. N. *Inorg. Chim. Acta* **1982**, *64*, L221.

[3] (a) Flood, T. C.; DiSanti, F. J.; Campbell, K. D. *Inorg. Chem.* **1978**, *17*, 1643. (b) Charrier, C.; Guihem, J.; Mathey, F. *J. Org. Chem.* **1981**, *46*, 3.

[4] (a) Compound **3a** has been previously prepared in 3% yield from the reaction of 1,2,3-triphenyl-1,2,3-triphosphaindane and $Fe_3(CO)_{12}$: King, R. B.; Reimann, R. H. *Inorg. Chem.* **1976**, *15*, 184. (b) Our IR and $^{31}$P NMR spectroscopic data for **3a** agree well with that reported by King; however, we observe a melting point of 196–197 °C dec (lit. mp 200–201 °C dec).

[5] (a) Teo, B. K.; Hall, M. B.; Fenske, R. F.; Dahl, L. F. *Inorg. Chem.* **1975**, *14*, 3103. (b) Burdett, J. K. *J. Chem. Soc., Dalton Trans.* **1977**, 610.

[6] IR (THF): 1980 (s), 1940 (s), 1905 (vs), 1875 (s), 1790 (m), 1765 (m), 1680 (w) cm$^{-1}$.

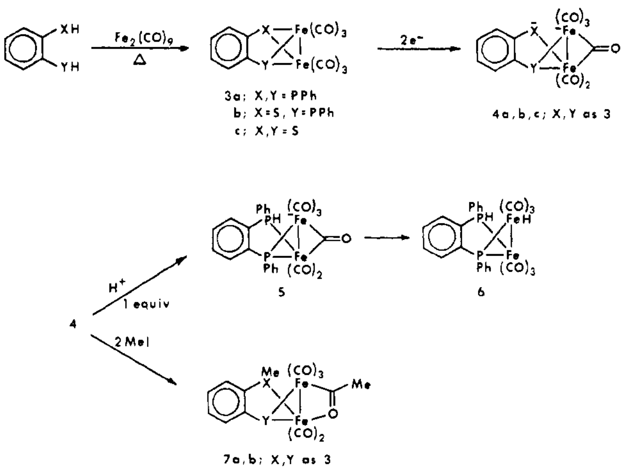
[7] The reductive electrochemical behavior of **3a** was different from that reported for **1**, as well. Reductive cyclic voltammetry of **3a** at a mercury electrode was ill-behaved, whereas under similar conditions the reduction of **1** was reported to be reversible ($E_p$ = −1.26 V vs. SCE$^{2d}$). Reduction of **3a** at a platinum electrode was irreversible but reproducible ($E_p$ = −1.51 V vs. SCE), whereas under similar conditions the reduction of **1** was "totally irreversible". We thank Dr. J. R. Hurst and Professor M. A. Fox for the electrochemical data.

[8] **5**: IR (THF) 1990 (s), 1940 (vs), 1900 (s), 1680 (w) cm$^{-1}$.

[9] Complexes **5** and **6** have thus far been too unstable to isolate and purify for analysis. The $^{31}$P{$^1$H}NMR spectrum (THF) of **6**, as well as that of **5**, contains two sets of doublets of doublets suggesting the presence of isomers: major species $\delta$ 202.4, 57.0 ($J_{P-P}$ = 31 Hz); minor species $\delta$ 202.9, 40.3 ($J_{P-P}$ = 29 Hz). The $^1$H-coupled spectrum gave $J_{P-H}$ = 357 Hz for the phosphorus absorptions at high field.

[10] **7a**: mp 120–122.5 °C; $^1$H NMR ($CDCl_3$) $\delta$ 2.1 (s, 0.6 H), 2.0 (s, 2.4 H), 1.9 (d, $J_{P-H}$ = 7.8 Hz, 2.4 H), 1.75 (d, $J_{P-H}$ = 9.0 Hz, 0.6 H), 7.4 (m, 14 H); $^{13}$C NMR ($CDCl_3$) $\delta$ 212.7 (s), 147.0–128.4 (m), 47.95 (s), 47.7 (s), 15.5 (d, $J_{P-C}$ = 24 Hz); $^{31}$P NMR (THF) major isomer $\delta$ 197.1 (d), 64.9 (d, $J_{P-P}$ = 28 Hz), minor isomer $\delta$ 201.7 (d), 79.0 (d, $J_{P-P}$ = 38 Hz); IR ($C_2Cl_4$) 2045 (s), 2032 (m), 1988 (vs), 1972 (s), 1958 (s), 1482 (w), 1435 (vw) cm$^{-1}$. Anal. Calcd for $C_{26}H_{20}Fe_2O_6P_2$: C, 51.87; H, 3.35. Found: C, 51.91; H, 3.41.

Scheme I



Not unexpectedly, the reduction of **3b** resulted in cleavage of the Fe–S bond in preference to the Fe–P bond to yield **4b**.[12] Subsequent methylation afforded the orange $\mu$-acyl complex **7b**.[13-15]

We are continuing to examine the chemistry of these new complexes and, more generally, the chemical importance of linking phosphido bridges in polynuclear complexes.

**Acknowledgment.** Financial support from the National Science Foundation (Grant CHE81-12090) and the Robert A. Welch Foundation (Grant No. F573) is gratefully acknowledged.

**Registry No.** **3a**, 57139-08-9; **3b**, 86480-53-7; **3c**, 84577-31-1; **4a**, 86480-54-8; **4b**, 86480-55-9; **4c**, 86480-56-0; **5**, 86480-57-1; **6**, 86480-58-2; **7a**, 86480-59-3; **7b**, 86480-60-6; Fe, 7439-89-6; $Fe_2(CO)_9$, 15321-51-4.

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[11] The orange and magenta complexes **3b** and **3c** were prepared from the reactions of excess nonacarbonyldiiron with 1-thio-2-(phenylphosphino)benzene (73%) and dithiocatechol (69%), respectively. **3b**: mp 140–141 °C; MS, *m/e* 495.8570 ($M^+$) (calcd 495.8555). **3c**: mp 114–116 °C; MS, *m/e* 419.8156 ($M^+$) (calcd 419.8147). Their structures were established by X-ray crystallography (Hassett, K. L.; Davis, R. E.; unpublished results). We thank C. N. Clubb for a sample of 1-thio-2-(phenylphosphino)benzene, the preparation of which will be reported elsewhere.

[12] **4b**: IR (THF) 1962 (s), 1915 (vs), 1875 (s), 1700 (sh), 1665 (w) $cm^{-1}$; $^{31}P$ NMR (THF) $\delta$ 143.5 (s).

[13] **7b**: mp 127–129 °C; IR ($C_2Cl_4$) 2043 (s), 1988 (vs), 1973 (s), 1965 (s), 1930 (s), 1480 (w), 1435 (vw) $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 8.0–7.5 (m, 9 H), 2.73 (s, 3 H), 1.93 (s, 3 H); $^{31}P$ NMR (THF) 190.1 (s); MS, *m/e* 525.9016 ($M^+$) (Calcd 525.9025).

[14] The reduction of **3c** in THF yielded a solution of **4c**, based on its IR spectrum: 1978 (s), 1922 (s), 1881 (s), 1695 (sh), 1660 (w) $cm^{-1}$. Exposure of the solution to air gave a solution with an IR spectrum identical with that of **3c**. Treatment of the dianion with methyl iodide yielded a species with an IR spectrum similar to that observed for **7a** and **7b**: (THF) 2045 (s), 1995 (vs), 1970 (s), 1932 (w) $cm^{-1}$.

[15] While this manuscript was in preparation, we were informed (personal communication) that Wojcicki and his co-workers have observed the generation of a monoanion analogous to **5**, but emanating from the unlinked system **1** by the use of $HBEt_3^-$ as the reducing agent: Yu, Y.-F.; Gallucci, J.; Wojcicki, A. submitted for publication.

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# Syntheses of the Highly Charged Cluster Anions $[Ru_4(CO)_{11}]^{6-}$, $[Ru_6(CO)_{17}]^{4-}$, and $[Ru_6(CO)_{16}]^{6-}$

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*Received May 17, 1983*

*Summary:* Syntheses of the highly charged cluster anions $[Ru_4(CO)_{11}]^{6-}$, $[Ru_6(CO)_{17}]^{4-}$, and $[Ru_6(CO)_{16}]^{6-}$ are described. The anion $[Ru_4(CO)_{11}]^{6-}$ was prepared by the reduction of $Ru_3(CO)_{12}$ and also $[Ru_4(CO)_{12}]^{4-}$ using stoichiometric amounts of $Na[(C_6H_5)_2CO]$. The hexanuclear anions $[Ru_6(CO)_{17}]^{4-}$ and $[Ru_6(CO)_{16}]^{6-}$ were prepared by reducing $[Ru_6(CO)_{18}]^{2-}$ with 2 and 4 equiv of $Na[(C_6H_5)_2CO]$, respectively. Electrical conductivities of tetraphenylphosphonium salts of these anions are consistent with the assigned charges.

Transition-metal carbonyl cluster anions with a charge of 4– or higher are rare.[1] Prior to this report $[Ru_4(CO)_{12}]^{4-}$ was the only ruthenium carbonyl cluster anion cited with a charge as large as 4–. It was prepared[2] through the reduction of $Ru_3(CO)_{12}$. The possibility of obtaining additional highly charged ruthenium carbonyl cluster anions that could be isolated as salts has been of interest to us. We present here the preparation of the new highly charged anions $[Ru_4(CO)_{11}]^{6-}$, $[Ru_6(CO)_{17}]^{4-}$, and $[Ru_6(CO)_{16}]^{6-}$.

The title anions were prepared according to the following reduction reactions ((1)–(3)) in which carefully measured stoichiometric ratios of reactants were employed. These reactions produced CO (analyzed mass spectrometrically) in yields of 98–100% of theory.

$$2Ru_3(CO)_{12} + 9Na[(C_6H_5)_2CO] \xrightarrow[60\ ^\circ C]{THF} 1.5Na_6[Ru_4(CO)_{11}] + 7.5CO + 9(C_6H_5)_2CO \quad (1)$$

$$Na_2[Ru_6(CO)_{18}] + 2Na[(C_6H_5)_2CO] \xrightarrow[60\ ^\circ C]{THF} Na_4[Ru_6(CO)_{17}] + CO + 2(C_6H_5)_2CO \quad (2)$$

$$Na_2[Ru_6(CO)_{18}] + 4Na[(C_6H_5)_2CO] \xrightarrow[60\ ^\circ C]{THF} Na_6[Ru_6(CO)_{16}] + 2CO + 4(C_6H_5)_2CO \quad (3)$$

The anion $[Ru_4(CO)_{11}]^{6-}$ was obtained according to reaction 1. It was also produced by reducing $[Ru_4(CO)_{12}]^{4-}$ with 2 equiv of $Na[(C_6H_5)_2CO]$. When $[Ru_6(CO)_{18}]^{2-}$ was reduced with 2 equiv of $Na[(C_6H_5)_2CO]$ (reaction 2) and 4 equiv of $Na[(C_6H_5)_2CO]$ (reaction 3), the anions $[Ru_6(CO)_{17}]^{4-}$ and $[Ru_6(CO)_{16}]^{6-}$ were prepared. Reduction of $[Ru_6(CO)_{17}]^{4-}$ with 2 equiv of $Na[(C_6H_5)_2CO]$ also gives $[Ru_6(CO)_{16}]^{6-}$.

The anions $[Ru_4(CO)_{11}]^{6-}$ and $[Ru_6(CO)_{16}]^{6-}$ appear to be the limiting anions that can be obtained under the conditions described here. When excess reducing agent was employed under the conditions cited above, no new species were obtained. More vigorous conditions might produce carbide systems.[3]

The new anions were isolated as the tetraphenylphosphonium salts $[P(C_6H_5)_4]_6[Ru_4(CO)_{11}]$, $[P(C_6H_5)_4]_4[Ru_6(CO)_{17}]$, and $[P(C_6H_5)_4]_6[Ru_6(CO)_{16}]$. Although highly air sensitive,[4] these salts are stable at room temperature under dry $N_2$. Conductivity measurements of these salts in $CH_3CN$ at room temperature were performed in a controlled atmosphere box. Figure 1 shows conductivities as a function of $(\text{molarity})^{1/2}$. The limiting molar conductivities $\Lambda_0$ = 764, 787, and 514 $cm^2\ \Omega^{-1}\ mol^{-1}$ for $[P(C_6H_5)_4]_6[Ru_4(CO)_{11}]$, $[P(C_6H_5)_4]_6[Ru_6(CO)_{16}]$, and [P-

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[1] Coyle, J. D. In "Comprehensive Organometallic Chemistry"; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon Press: Elmsford, NY, 1982; Vol. 9, pp 441–822.

[2] (a) Bhattacharyya, A. A.; Nagel, C. C.; Shore, S. G. *Organometallics*, in press. (b) Nagel, C. C.; Shore, S. G. *J. Chem. Soc., Chem. Commun.* **1980**, 530.

[3] Hayward, C.-M. T.; Shapley, J. R. *Inorg. Chem.* **1982**, *21*, 3816.

[4] Shriver, D. F. "The Manipulation of Air Sensitive Compounds"; McGraw-Hill: New York, 1969.

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