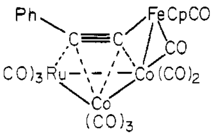
Table I. Capping Reagents and Products[10]

reagent	temp, °C	product	yield, %
$MePH_2$	−20 to +20	1	37
$MeAsH_2$	0	2	38
EtSH	20	3	31
Ph_2Se_2	20	4	22
Ph_2C_2	20	5	73
$Cp(CO)_2FeC_2Ph$	20	6	69
$Cp(CO)_2WC(tol)$	−20 to +20	7	12
$RuCo_2(CO)_{11}$ (decomp)[5]	35	8	46
$KCo(CO)_4/H_3PO_4$	0 to +20	9	56

ature from −15 to +20 °C. Chromatography on a silica gel column afforded first $Co_2WCp(CO)_8C(tol)$[8] and then **7** (40 mg, 12%) as black crystals: mp 196 °C, δ (^1H, $CDCl_3$): Me 2.34, Cp 5.39, C_6H_4 7.31 (m); ν(CO, $CHCl_3$) 2076 (s), 2032 (vs), 2008 (m) 1992 (m), 1860 (w, br), 1795 (w, br) cm^{-1}.

Metal carbonyl fragments are equally well suited for the capping reaction. This had been observed in the thermal decomposition reaction of $RuCo_2(CO)_{11}$[5] where it is likely that $Ru(CO)_n$ fragments are liberated and then added with formation of $Ru_2Co_2(CO)_{13}$ (**8**). It was now used for the preparation of $RuCo_3(CO)_{12}^-$ by addition of $KCo(CO)_4$ in THF and subsequent acidification to give a 56% yield of $HRuCo_3(CO)_{12}$ (**9**).[9] All capping reactions are summarized in Table I.[10]



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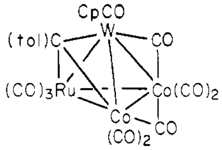

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Although $RuCo_2(CO)_{11}$ is saturated according to the 18-electron rule, it reacts like an unsaturated compound. This is an illustration of the driving force inherent in the capping reaction which in this specific case is made easy by the CO lability on cobalt as well as on ruthenium. It makes possible the elimination of organic substitutents from sulfur and selenium, i.e., the formation of **3** and **4**, which normally requires forcing conditions. And it allows the uncomplicated low-temperature incorporation of reactive units like acetylenes or metal carbonyl reagents to form new clusters whose thermal stability is limited. It is likely that all these capping reactions start with CO substitution. The tendency for capping rather than fragmentation or μ bridging of the cluster must have to do with the fact that the resulting compounds all have a $M_3(CO)_9X$ composition which seems to be especially preferred in metal carbonyl chemistry due to its ideal stereochemical and possibly electronic situation.

The use of the starting cluster $RuCo_2(CO)_{11}$ is favorable in this context since many capping units are four-electron ligands which just means that a $RuCo_2(CO)_9X$ composition will result in the form of the stable compounds **1**–**9**. By contrast, the tendency for capping may be an important aspect in the field of substrate activation by clusters where it is essential to fix but not passivate reaction intermediates in a stereochemically and electronically suitable environment. The interconversions of C–N compounds capping a $Fe_3(CO)_9$ unit[11] and the first $M_3(CO)_9(HCCR) \rightarrow M_3(CO)_9(CCHR)$ isomerizations[12] may serve as examples.

Acknowledgment. This work was supported by the Fonds der Chemischen Industrie and by a generous loan of $RuCl_3$ from Heraeus GmbH.

Registry No. **1**, 86272-85-7; **2**, 86272-86-8; **3**, 86272-87-9; **4**, 86272-88-0; **5**, 86288-23-5; **6**, 86272-89-1; **7**, 86272-90-4; **9**, 24013-40-9; $RuCo_2(CO)_{11}$, 78456-89-0; $MePH_2$, 593-54-4; $MeAsH_2$, 593-52-2; Ph_2Se_2, 1666-13-3; EtSH, 75-08-1; Co, 7440-48-4; Ru, 7440-18-8; Fe, 7439-89-6; W, 7440-33-7.

Supplementary Material Available: Tables containing the IR and NMR data, the melting points, and the elemental analyses of the new compounds **1**–**7** and crystallographic details and figures of the molecular structures of **5**–**7** (6 pages). Ordering information is given on any current masthead page.

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[8] Beurich, H.; Vahrenkamp, H. *Chem. Ber.* **1982**, *115*, 2385.

[9] Mays, M. J.; Simpson, R. N. F. *J. Chem. Soc.* A **1968**, 1444.

[10] All new compounds have been characterized by satisfactory elemental analyses and IR, NMR, and mass spectra. Crystal structure determinations were performed for **5**–**7**. All these details are given in the supplementary material.

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Characterization and Interconversion of Metal–Phosphorus Single and Double Bonds: Bis(cyclopentadienyl)zirconium and -hafnium Bis(diorganophosphide) Complexes†

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Received March 10, 1983

Summary: Two equivalents of $LiPR_2$ react with $(\eta$-$C_5H_5)_2MCl_2$, affording $(\eta$-$C_5H_5)_2M(PR_2)_2$ (M = Zr, Hf; R = ethyl, cyclohexyl (Cy), or phenyl). ^{31}P NMR and X-ray structural results indicate that these complexes contain both single and double metal–phosphorus bonds which interconvert in solution. Sodium naphthalenide reduction of these complexes produces the corresponding Zr^{III} and Hf^{III} complexes $[(\eta$-$C_5H_5)_2M(PR_2)_2][Na(THF)_n]$; a bis($\mu$-diorganophosphido) heterobimetallic structure is proposed.

Early transition-metal complexes containing terminal diorganoamide ligands have a well-developed preparative and derivative chemistry,[1,2] but the phosphorus analogs do not.[2-7] Both examples[2,3] of structurally characterized

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† Contribution No. 3207.

[1] Lappert, M. F.; Power, P. P.; Sanger, A. R.; Srivastava, R. C. "Metal and Metalloid Amides"; Ellis Horwood Ltd.: Chichester, 1980. Andersen, R. A. *Inorg. Chem.* **1979**, *18*, 1724, 2928; *J. Organomet. Chem.* **1980**, *192*, 189. Bynum, R. V.; Hunter, W. E.; Rogers, R. D.; Atwood, J. L. *Inorg. Chem.* **1980**, *19*, 2368. Blatchford, T. P.; Chisholm, M. H.; Folting, K.; Huffman, J. C. *Inorg. Chem.* **1980**, *19*, 3175. Chisholm, M. H.; Huffman, J. C.; Tan, L. S. *Ibid.* **1981**, *20*, 1859. Hillhouse, G. L.; Bercaw, J. E. *Organometallics* **1982**, *1*, 1025. Chisholm, M. H. *ACS Symp. Ser.* **1981**, *No. 155*, 17. Nugent, W. A. *Inorg. Chem.* **1983**, *22*, 965.

[2] Rocklage, S. M.; Schrock, R. R.; Churchill, M. R.; Wasserman, H. J. *Organometallics* **1982**, *1*, 1332. X-ray structure of $W(CCMe_3)(PHPh)(PEt_3)_2Cl_2$.

[3] Domaille, P. J.; Foxman, B. M.; McNeese, T. J.; Wreford, S. S. *J. Am. Chem. Soc.* **1980**, *102*, 4114. X-ray structure of $TaH(PPh_2)_2(DMPE)_2$.

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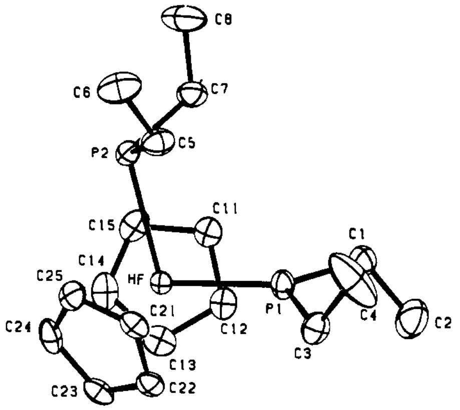
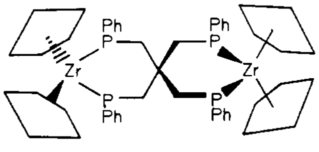


Figure 1. A perspective view of $(\eta\text{-}C_5H_5)_2Hf(PEt_2)_2$, IId, showing 50% probability ellipsoids. Hydrogen atoms are omitted for clarity: Hf–P(1) = 2.488 (1) Å, Hf–P(2) = 2.682 (1) Å, and P(1)–Hf–P(2) = 98.64 (3)°.

complexes containing terminal, π-donor diorganophosphide ligands[4] have trigonal-planar geometries about phosphorus, indicative of metal–phosphorus multiple bonding.

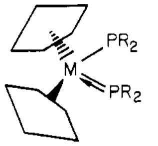
In 1967, Ellerman and Poersch reported[6] that the reaction of $(\eta\text{-}C_5H_5)_2ZrCl_2$ with $C(CH_2PPhNa)_4$ in liquid ammonia produces the bis(diorganophosphide) complex I, and they proposed the structure shown. As part of a



I

broad study of early transition-metal diorganophosphide chemistry,[3,8,9] we have prepared a series of $(\eta\text{-}C_5H_5)_2M(PR_2)_2$ complexes, IIa–f. ^{31}P NMR and X-ray structural results indicate that these complexes contain both single and double metal–phosphorus bonds which interconvert rapidly on the NMR time scale in solution.

The reactions of $(\eta\text{-}C_5H_5)_2MCl_2$ with 2 equiv of $LiPR_2$[10] are conducted in tetrahydrofuran (THF) at 80 °C for M = Hf and at −80 °C for M = Zr to avoid reduction to trivalent products with concomitant formation of P_2R_4.[11] After solvent removal, extraction with *n*-hexane,[12] and filtration to remove LiCl, slow concentration of the red-orange to red-purple solutions affords the crystalline products IIa–f in 75–90% yields. Complexes IIa–f have



IIa, M = Zr, R = Et
b, M = Zr, R = Cy
c, M = Zr, R = Ph
d, M = Hf, R = Et
e, M = Hf, R = Cy
f, M = Hf, R = Ph

been characterized by their ^1H, ^{13}C, and ^{31}P NMR spectra and by full elemental analysis.[13] The molecular structure of IId has been determined by X-ray diffraction[14] and is shown in Figure 1.

The hafnium atom is coordinated to two planar $\eta\text{-}C_5H_5$ rings and the two phosphorus ligands in an unexceptional distorted tetrahedral geometry. The remarkable feature of this structure is the two distinct bonding modes of the diethylphosphide ligands. The geometry about P(2) is pyramidal (sp^3) with an "external" lone pair of electrons, while that of P(1) is trigonal planar (sp^2) with the orthogonal lone pair involved in π bonding with hafnium. This π-donor interaction is substantial, the difference between Hf–P(2) (2.682 (1) Å) and Hf–P(1) (2.488 (1) Å) amounting to nearly 0.2 Å. The trigonal-planar PEt_2 ligand is orthogonal to the HfP_2 plane, allowing the ligand lone-pair maximum overlap with the empty a_1 orbital of the $(\eta\text{-}C_5H_5)_2Hf^{IV}$ moiety, as previously predicted by Lauher and Hoffmann.[15] IIa–f are thus members of the rare class of unsymmetrical 18-electron $(\eta\text{-}C_5H_5)_2MX_2$ complexes, the only previous structurally characterized example being $(\eta\text{-}C_5H_5)_2$ Ti(*p*-nitrobenzoate)$_2$[16] with Ti–O distances/Ti–O–C angles of 2.04 Å/136° and 1.94 Å/157° for the Ti–O single and double bonds, respectively.

The $^{31}P\{^1H\}$ NMR spectra of complexes IIa–f at 25 °C all consist of a singlet between 100 and 160 ppm, indicating that the interconversion of the Hf–P single and double bonds is fast on the NMR time scale. This process is quenched at low temperatures only for the bulky bis(dicyclohexylphosphide) complexes, IIb,e. Although the slow

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[4] Several X-ray structures of complexes containing terminal PX_2 groups have also been reported: Hutchins, I. D.; Paine, R. T.; Campana, C. F. *J. Am. Chem. Soc.* **1980**, *102*, 4521. (X_2 = $-N(Me)CH_2CH_2(Me)N-$). Day, V. W.; Tavanaiepour, I.; Abdel-Meguid, S. S.; Kirner, J. F.; Goh, L. Y.; Muetterties, E. L. *Inorg. Chem.* **1982**, *21*, 657 (X = OMe). Weber, D.; Fluck, E.; Von Schnering, H. G.; Peters, K. *Z. Naturforsch., B: Anorg. Chem., Org. Chem.* **1982**, *37B*, 594 (X = $-P(O)(OEt)_2$).

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[9] Baker, R. T.; Whitney, J. F.; Tulip, T. H.; Krusic, P. J.; Wreford, S. S. *Inorg. Chem.*, to be submitted for publication.

[10] $LiPR_2$ reagents are prepared in diethyl ether/hexane from PR_2H and *n*-BuLi as modified after: Issleib, K.; Tzschach, A. *Chem. Ber.* **1959**, *92*, 1118.

[11] At ambient temperature $(\eta\text{-}C_5H_5)_2ZrCl_2$ and $LiPEt_2$ react to produce the dimeric Zr^{III} complex $[(\eta\text{-}C_5H_5)_2Zr(PEt_2)]_2$, which contains two PEt_2 bridges and a metal–metal bond: Issleib, K. Hackert, H. *Z. Naturforsch., B: Anorg. Chem., Org. Chem., Biochem., Biophys., Biol.* **1966**, *21B*, 519 (see also ref 9 for full spectroscopic characterization).

[12] For R = Ph the reaction residue is extracted with benzene and filtered. An equal volume of heptane is added and the solution concentrated in vacuo to yield crystalline IIc,f.

[13] For example, for IIa: ^1H NMR (200 MHz, C_6D_6) δ 5.54 (t, $^3J_{PH}$ = 1.5 Hz, 10 H, C_5H_5), 1.90 (qd, $^3J_{HH}$ = 7.5, $^2J_{PH}$ = 3.0 Hz, 8 H, CH_2CH_3), 1.19 (dtr, $^3J_{PH}$ = 12.5 Hz, 12 H, CH_2CH_3); ^{13}C NMR (100.6 MHz, C_6D_6, gated decoupling) 104.48 (dt, J_{CH} = 173, $^2J_{CH}$ = 6 Hz, 10 C, C_5H_5); 23.81 (td, J_{CH} = 128, J_{CP} = 11 Hz, 4 C, CH_2CH_3), 15.60 ppm (q, J_{CH} = 126 Hz, 4 C, CH_2CH_3); $^{31}P\{^1H\}$ NMR (161.9 MHz, 20% C_6D_6/THF) 130.3 ppm (s). Anal. Calcd for $C_{18}H_{30}P_2Zr$: C, 54.10; H, 7.57; P, 15.50; Zr, 22.83. Found: C, 53.55, 53.39; H, 7.44; P, 14.8, 15.3; Zr, 22.9, 22.7.

[14] Crystal data for IId grown from hexane solution: $C_{18}H_{30}HfP_2$; *M* 486.88; triclinic; space group $P\bar{1}$ (No. 2); a = 9.494 (2) Å, b = 14.086 (3) Å, c = 7.953 (2) Å; α = 101.15 (2)°, β = 108.12 (2)°, γ = 77.44 (2)°; V = 977.5 (8) Å^3; $Z = 2$; ρ(calcd) = 1.654 g cm^{-3}; T = −100 °C. On the basis of refinement of 310 variables (all non-hydrogen atoms with anisotropic thermal parameters) using 3947 unique reflections with $F_o^2 > 2\sigma(F_o^2)$, R = R_w = 0.027. Full details will be published separately.[9] (See also supplementary material.)

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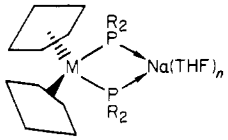
exchange limiting spectrum (presumably two doublets) was not obtained, at −126 °C IIe exhibits two broad ($\Delta\nu_{1/2} \simeq$ 700 Hz) resonances at 270.2 and −15.3 ppm,[17] *a chemical shift difference of 285 ppm for two PCy_2 ligands on the same metal.* These two resonances coalesce at −100 °C, corresponding[18] to an activation energy of 6.0 ± 0.2 kcal/mol.

Reduction of complexes IIa–f by sodium naphthalenide in THF at 25 °C produces the thermally stable Zr^{III} and Hf^{III} complexes $[(\eta\text{-}C_5H_5)_2M(PR_2)_2][Na(THF)_n]$, IIIa–f, as



IIIa, M = Zr, R = Et
b, M = Zr, R = Cy
c, M = Zr, R = Ph
d, M = Hf, R = Et
e, M = Hf, R = Cy
f, M = Hf, R = Ph

determined by ESR spectroscopy.[19-21] The four-membered, inorganic ring structure shown is suggested by the ^{23}Na hyperfine splitting observed in the ESR spectra of complexes IIIa,b. Although complexes IIIa–f are presumably isostructural, ^{23}Na hyperfine splittings were not observed for R = Ph and not resolved for M = Hf, because of the large inherent line widths ($\Delta\nu_{1/2} \simeq 0.5$ mT). Complexes IIId–f are the most thermally stable organohafnium(III) species yet reported;[22] the ESR signal persists for days at room temperature.

Further details of these new metal-containing diphosphines will appear in a subsequent full paper.[9]

Acknowledgment. We wish to thank the following persons for fine technical assistance in the following areas: synthesis, D. W. Reutter, K. H. Richmond, W. M. Gray, and S. A. Hill; X-ray, L. F. Lardear; NMR, J. D. Center, G. Watunya, Drs. D. W. Ovenall, P. J. Domaille, and D. C. Roe; ESR; D. J. Jones and Dr. P. J. Krusic.

Registry No. IIa, 86013-23-2; IIb, 86013-24-3; IIc, 86013-25-4; IId, 86013-26-5; IIe, 86013-27-6; IIf, 86013-28-7; IIIa, 86013-29-8; IIIb, 86013-30-1; IIIc, 86013-31-2; IIId, 86013-32-3; IIIe, 86013-33-4; IIIf, 86013-34-5; $(\eta\text{-}C_5H_5)_2ZnCl_2$, 1291-32-3; $(\eta\text{-}C_5H_5)_2HfCl_2$, 12116-66-4; $LiPEt_2$, 19093-80-2; $LiPCy_2$, 19966-81-5; $LiPPh_2$, 4541-02-0; sodium naphthalenide, 3481-12-7.

Supplementary Material Available: Tables of final fractional atomic coordinates, bond distances and angles, and observed and calculated structure factors (37 pages). Ordering information is given on any current masthead page.

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(17) ^{31}P NMR chemical shifts are positive downfield from external 85% H_3PO_4.

(18) Martin, M. L.; Martin, G. J.; Delpeuch, J.-J. "Practical NMR Spectroscopy"; Heyden: Philadelphia, 1980; Chapter 8, p 339.

(19) Although we have been unable to crystallize IIIa–f, no other products are detected by ESR or NMR spectroscopy. Comparison of the doubly integrated ESR signal intensities vs. external DPPH in THF indicates that the reductions are quantitive within the experimental error of ca. 20%.

(20) 9.54-GHz ESR data at 25 °C in THF: (g_{av}, $a(^{31}P)$ in mT): IIIa, 1.9902, 1.00, $a(^{91}Zr) \simeq 2.0$, $a(^{23}Na) = 0.18$; IIIb, 1.9872, 1.23, $a(^{91}Zr) \simeq 2.4$, $a(^{23}Na) = 0.20$; IIIc, 1.9860, 1.14, $a(^{91}Zr) \simeq 2.3$; IIId, 1.9753, 1.10; IIIe, 1.9666, 1.28; IIIf, 1.9653, 1.24.

(21) The low-temperature ESR spectra of complex IIIc and several titanium analogues (generated from $(\eta\text{-}C_5H_5)_2MCl_2$ and excess $NaPR_2$) have been reported previously.[7]

(22) Lappert, M. F.; Raston, C. L. *J. Chem. Soc., Chem. Commun.* **1980**, 1284.

Metal vs. Ligand Exchange upon Reaction of $[(\eta^5\text{-}C_5H_5)Mo(CO)_3]^-$ with $(\mu_3\text{-}S)Co_3(CO)_7LX$ Clusters. A Two-Step Synthesis of the Optically Active Cluster $(\mu_3\text{-}S)Mo(\eta^5\text{-}C_5H_5)Co_2(CO)_6[\mu\text{-}1,2\text{-}\eta^2\text{-}C(R^1)N(R^2)]$

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Received March 11, 1983

Summary: The $(\mu_3\text{-}S)Co_3(CO)_7LX$ clusters react with $(\eta^5\text{-}C_5H_5)Mo(CO)_3^-$ to afford either $(\eta^5\text{-}C_5H_5)Mo(CO)_2LX$ complexes or the heteronuclear cluster $(\mu_3\text{-}S)Mo(\eta^5\text{-}C_5H_5)Co_2(CO)_6LX$, depending on the ability of the bridging ligand to stabilize the binuclear intermediates. The X-ray structure analysis of the $MoCo_2$ cluster shows that it spontaneously forms optically active crystals. The catalytic activities of four clusters in hydroformylation have been examined.

Transition-metal cluster compounds constitute an area of organometallic chemistry currently under intense investigation, largely due to their potential utilization in homogeneous catalysis where they can function either as the catalytically active species or may serve as precursors for the release of reactive fragments.[1-3] Concerning the latter point it may be noted that most of the known clusters are composed of individual fragments linked by metal–metal bonds with the occasional participation of other elements (carbon, sulfur, phosphorus, ...) bonded to the metal atoms. The ability of clusters to act as catalysts has been debated, and although plausible mechanisms have been proposed,[2] definitive proof that they can be active as a structural whole does not seem to have been obtained.[4] This has led to considerable efforts directed toward the synthesis of optically active clusters[5] which would be suitable species with which to examine the process of optical induction in cluster-catalyzed reactions because such intrinsically chiral compounds should provide the proof that clusters considered as an entity can be catalytically active.

Our approach to the synthesis of chiral clusters was based on the discovery that chirality could be obtained directly by treating with $Co_2(CO)_8$ compounds such as $R^1C(S)NR^2R$.[3] With secondary thioamides[6] ($R^1 \neq R^2 \neq$ H, R^3 = H), thiocarbonyl chloride[7] (R^1 = Cl, $R^2 = R^3$ = Me) and primary thioamides[8] ($R^1 \neq$ H, $R^2 = R^3$ = H),

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(7) Mahé, C.; Patin, H.; Benoît, A.; Le Marouille, J. Y. *J. Organomet. Chem.* **1981**, *216*, C15.

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