recrystallization from $CHCl_3$/pentane was carried out and gave colorless crystals in 70% yield of $[Au_8Cl_2\{\mu_3\text{-}(MeO)_2PCHP(OMe)_2\}_2\{\mu_4\text{-}(MeO)_2PCP(OMe)_2\}_2]$ as the $CHCl_3$ solvate (**3**).[8]

The structure of **3** was determined by X-ray diffraction[9] and is a new type of cage containing four fused nine-membered rings. Each ring may be viewed as forming one face of a tetrahedron; Au atoms 1–6 are thus held in a proximity approaching octahedral (Au···Au ranging from 2.938 (1) to 4.085 (1) Å, Figure 1). Each of the four dmopm ligands has lost either one or two methylene protons. The atom order in two of the rings is AuPCAuPCAuPC, as proposed for **2**, while in the other two AuPCAuPCPAuC is found. Each ring contains at least one Au–P–CH–Au fragment (structure **3** and Figure 2a) and at least one of the hitherto unknown Au–P–C*–Au fragments (structure **3** and Figure 2b). The phosphorus atom of each of the doubly deprotonated ligands that is not involved in ring formation is bonded to an exocyclic Au–Cl unit. There is Au–Au bonding between the pairs of gold atoms bound to doubly deprotonated C*, as is clear from the distances Au(2)–Au(3) = 2.938 (1) Å and Au(4)–Au(6) = 2.951 (1) Å and the angles Au(2)–C(17)–Au(3) = 87.8 (9)° and Au(4)–C(38)–Au(6) = 88.4 (9)°.[10] This secondary bonding between Au(I) centers is a common phenomenon.[3e]

Given the structure of **3** and formula of **2**, it is possible to understand the complex chemistry involved. It seems that the alkynyl ligand in **1** is sufficiently basic to deprotonate the dmopm ligand with formation of *t*-BuCCH and "$Au_2(CC\text{-}t\text{-}Bu)\{(MeO)_2PCHP(OMe)_2\}$", which oligomerizes to the crystalline **2**. Further conversion of **2** to **3** in $CHCl_3$ involves additional condensation by loss of *t*-BuCCH, leading to formation of the new $[\mu_4\text{-}(MeO)_2PCP(OMe)_2]^{2-}$ ligand, along with a parallel reaction in which AuCC-*t*-Bu groups react with chloroform to give Au–Cl bonds.[11] It is likely that the very easy double deprotonation of the $(MeO)_2PCH_2P(OMe)_2$ ligand is aided by the electronegative methoxy substituents on phosphorus, which may increase the acidity of the CH_2P_2 protons compared to those of the more common $R_2PCH_2PR_2$ ligands with R = alkyl or aryl.[6] In turn, this promises a rich chemistry of the ligands $(RO)_2PCH_2P(OR)_2$ in forming and stabilizing unusual cage and cluster complexes of other elements.

Acknowledgment. We thank the NSERC (Canada) for financial support to N.C.P. and R.J.P. and the University of Western Ontario for a fee bursary to R.R.

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(8) Data for **3**: Anal. Calcd for $C_{21}H_{51}Au_8Cl_5O_{16}P_8$: C, 9.85; H, 2.0. Found: C, 9.8; H, 2.1. NMR (CD_2Cl_2): ^1H, δ 3.02 [dt, 1 H, $^2J(PH) = 17$ Hz, $^3J(PH) = 8$ Hz, CHP_2], 3.10 [dt, 1 H, $^2J(PH) = 17$ Hz, $^3J(PH) = 8$ Hz, CHP_2], 3.70–4.03 [m, *Me*O]; ^{31}P, δ 157.7 [m, 1 P], 155.1 [m, 1 P], 150.4 [m, 1 P], 149.9 [m, 1 P] 148.4 [m, 1 P], 145.6 [m, 1 P], 126.4 [m, 2 P].

(9) Crystal data for **3**: $C_{21}H_{51}Au_8Cl_5O_{16}P_8$, $M_r = 2560.4$, triclinic, $P\bar{1}$, $a = 14.152$ (2) Å, $b = 18.373$ (2) Å, $c = 12.151$ (2) Å, $\alpha = 91.17$ (1)°, $\beta = 114.77$ (1)°, $\gamma = 108.16$ (1)°, $V = 2684$ (2) Å, $Z = 2$, $D_m = 3.158$ (2) Mg m^{-3}, $D_c = 3.168$ Mg m^{-3}, $F(000) = 2282$ e, $\lambda(\text{Mo } K\bar{\alpha}) = 0.71073$ Å, $\mu = 214.8$ mm^{-1}, $T = 295$ K. Intensity data were collected on an Enraf-Nonius CAD4F diffractometer by the θ–2θ technique with use of graphite-monochromatized Mo radiation. The structure was solved with SHELXS-86 software. Full-matrix least-squares refinement on F (SHELX76 software, 5677 unique observations with $I > 2.5\sigma(I)$, 436 variables, H atom contributions included but not refined) converged at $R = 0.0355$ and $R_w = 0.0371$. Full details of the structure determination are given in the supplementary material.

(10) Scherbaum, F.; Grohmann, A.; Huber, B.; Krüger, C.; Schmidbaur, H. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 1544.

(11) Manojlović-Muir, Lj.; Muir, K. W.; Treurnicht, I.; Puddephatt, R. J. *Inorg. Chem.* **1987**, *26*, 2418. Payne, N. C.; Puddephatt, R. J.; Ravindranath, R.; Treurnicht, I. *Can. J. Chem.* **1988**, *66*, 3176.

Supplementary Material Available: A description of the crystal structure data collection and refinement and tables of atomic positional and thermal parameters, anisotropic thermal parameters, hydrogen atom parameters, selected torsion angles, selected bond distances and bond angles, and least-squares planes (12 pages); a listing of observed and calculated structure amplitudes (43 pages). Ordering information is given on any current masthead page.

Unambiguous Example of a η^2-Benzyl Group Functioning as a Formal Three-Electron Ligand: Solid-State Molecular Structure of $Cp^*Mo(NO)(CH_2SiMe_3)(\eta^2\text{-}CH_2C_6H_5)$[1]

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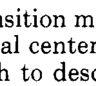
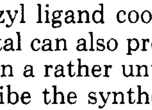
Summary: Sequential treatment at 20 °C of $Cp^*Mo(NO)(\eta^2\text{-}CH_2Ph)Cl$ in Et_2O first with an equimolar amount of Me_3SiCH_2MgCl and then water affords $Cp^*Mo(NO)(CH_2SiMe_3)(\eta^2\text{-}CH_2Ph)$, which is isolable in 82% yield as red, prismatic crystals. A single-crystal X-ray crystallographic analysis of the product complex has confirmed its formulation and has determined that it possesses a "three-legged piano stool" molecular structure. The chemically most interesting result of this analysis is the establishment of the intramolecular dimensions of the $Mo\text{–}(\eta^2\text{-}CH_2Ph)$ grouping, which indicate that the η^2-benzyl group functions as a three-electron ligand via C–Moσ bonds involving its methylene and ipso carbon atoms.

When a benzyl group functions as a formal three-electron donor to a transition-metal center, it usually attaches to the metal in a η^3 fashion to form a linkage that may be represented as shown in A. The first example of such a link was invoked for $CpMo(CO)_2(\eta^3\text{-}CH_2Ph)$ ($Cp = \eta^5\text{-}C_5H_5$)[2] and was confirmed by a single-crystal X-ray crystallographic analysis of its *p*-methylbenzyl analogue, whose molecular structure reveals the existence of the localized bonding within the phenyl ring indicated in A.[3] We now wish to report that a benzyl ligand coordinated in a η^2 manner to a transition metal can also provide three electrons to the metal center in a rather unusual way. Specifically, we wish to describe the synthesis and characterization of $Cp^*Mo(NO)(CH_2SiMe_3)(\eta^2\text{-}CH_2Ph)$ ($Cp^* = \eta^5\text{-}C_5Me_5$), a diamagnetic 18-valence-electron complex in which the metal–benzyl linkage is best represented by the resonance hybrid B (M = Mo).



A



B

$Cp^*Mo(NO)(CH_2SiMe_3)(\eta^2\text{-}CH_2Ph)$ is preparable by the sequential treatment of $Cp^*Mo(NO)(\eta^2\text{-}CH_2Ph)Cl$[4] in Et_2O

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(1) Organometallic Nitrosyl Chemistry. 42. Part 41: Legzdins, P.; Richter-Addo, G. B.; Einstein, F. W. B.; Jones, R. H. *Organometallics* **1990**, *9*, 431.

(2) King, R. B.; Fronzaglia, A. *J. Am. Chem. Soc.* **1966**, *88*, 709.

(3) Cotton, F. A.; LaPrade, M. D. *J. Am. Chem. Soc.* **1968**, *90*, 5418.

0276-7333/90/2309-0882$02.50/0

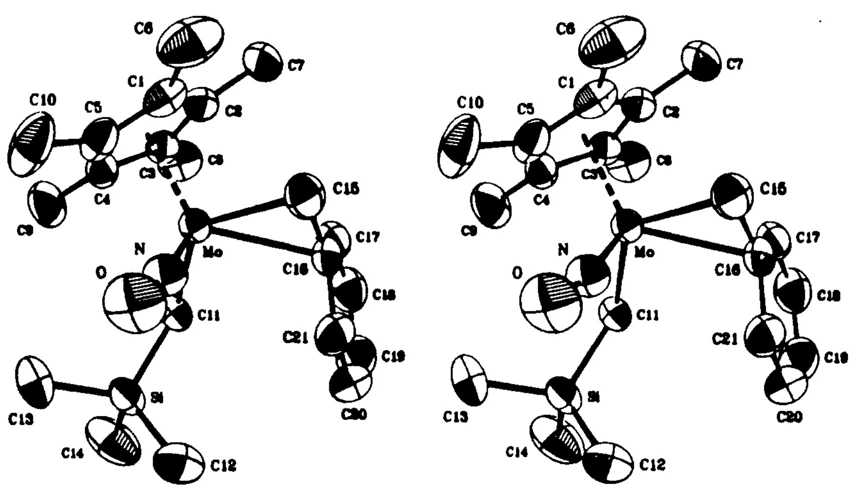

Figure 1. Stereoview of the solid-state molecular structure of Cp*Mo(NO)(CH_2SiMe_3)(η^2-CH_2Ph), hydrogen atoms having been omitted for clarity. Selected bond lengths (Å) and angles (deg): Mo–C(15) = 2.188 (2), Mo–C(16) = 2.473 (2), Mo–C(17) = 3.004 (2), Mo–C(21) = 3.165 (3), Mo–Cp* = 2.058 (1), Mo–C(11) = 2.218 (2), Mo–N = 1.760 (2), N–O = 1.217 (2), C(15)–C(16) = 1.450 (3), C(16)–C(17) = 1.397 (3), C(16)–C(21) = 1.417 (3), C(17)–C(18) = 1.375 (4), C(18)–C(19) = 1.372 (4), C(19)–C(20) = 1.371 (5), C(20)–C(21) = 1.375 (4); N–Mo–C(11) = 96.15 (8), N–Mo–C(15) = 90.87 (10), N–Mo–C(16) = 99.34 (8), N–Mo–Cp* = 121.42 (7), Mo–N–O = 169.7 (2), C(15)–Mo–C(16) = 35.60 (8), Mo–C(15)–C(16) = 82.97 (12), Mo–C(16)–C(15) = 61.43 (11), Mo–C(16)–C(17) = 97.98 (13), Mo–C(16)–C(21) = 105.70 (14).

at ambient temperatures first with an equimolar amount of Me_3SiCH_2MgCl and then water as summarized in eq 1. The product complex is isolable in 82% yield by re-

$$\text{Cp*Mo(NO)}(\eta^2\text{-CH}_2\text{Ph})\text{Cl (in Et}_2\text{O)} \xrightarrow[\text{2. H}_2\text{O}]{\text{1. Me}_3\text{SiCH}_2\text{MgCl}} \text{Cp*Mo(NO)(CH}_2\text{SiMe}_3)(\eta^2\text{-CH}_2\text{Ph}) \quad (1)$$

moval of volatiles from the final reaction mixture in vacuo, extraction of the remaining residue with 5:2 hexane–CH_2Cl_2, and fractional crystallization of these extracts. Cp*Mo(NO)(CH_2SiMe_3)(η^2-CH_2Ph) forms red, prismatic crystals[5] that are reasonably air-stable and soluble in all common organic solvents. One of these crystals has been subjected to an X-ray crystallographic analysis[6] in order to establish the solid-state molecular structure of the compound. The chemically most interesting results of this analysis are presented in Figure 1.

Not surprisingly, the solid-state molecular structure of Cp*Mo(NO)(CH_2SiMe_3)(η^2-CH_2Ph) is that of a "three-legged piano stool", the midpoint of C(15)–C(16) constituting the terminus of one of the legs. The intramolecular dimensions involving the Mo–Cp*, Mo–CH_2SiMe_3, and Mo–NO portions of the molecule are normal and comparable to those found in related compounds.[7,8] The bond lengths and angles in the Mo–CH_2Ph part of the molecule, however, are distinctive and have led us to formulate this linkage as depicted in B above for the following reasons.

(1) The complex is diamagnetic, and yet the benzyl ligand is not monodentate, as it would be if it were functioning as a one-electron donor to the 15-valence-electron Cp*Mo(NO)(CH_2SiMe_3) fragment.

(2) All the hydrogen atoms of the benzyl ligand have been located and refined; none are situated so that a C–H–Mo agostic interaction[9] can be invoked.

(3) The observed Mo–C bond lengths of 2.188 (2) and 2.473 (2) Å, respectively,[10] indicate that the methylene and ipso carbons of the benzyl ligand are bonded quite strongly to the metal center. On the other hand, the ortho carbons of the benzyl's phenyl ring (i.e. C(17) and C(21)) are not since they are more than 3 Å away from the molybdenum atom.

(4) The four phenyl C–C distances from C(17) to C(21) are essentially identical and rule out the existence of localized bonding within the phenyl ring of the type shown in A. Furthermore, the observed bond lengths of 1.371(5)–1.375(4) Å suggest the presence of delocalized multiple bonding extending over these carbons.[11] Consistently, the remaining C–C bond lengths within the planar benzyl ligand are longer.

Representation of the M–CH_2Ph interaction by resonance hybrid B thus indicates that two electrons have formally been removed from the phenyl ring's π system and are being provided to the metal center via a carbon-to-metal σ donor bond. This bond, combined with the other, normal C–M σ covalent bond shown in either limiting form of B, thus enables the benzyl ligand to function as a formal three-electron donor and permits the metal to attain the favored 18-valence-electron configuration.

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(4) Dryden, N. H.; Legzdins, P. Manuscript in preparation.

(5) Anal. Calcd for $C_{21}H_{33}NOSiMo$: C, 57.39; H, 7.57; N, 3.19. Found: C, 57.15; H, 7.60; N, 3.22. IR (Et_2O): ν_{NO} 1597 (s) cm^{-1}. ^1H NMR (300 MHz, C_6D_6) δ 7.35 (t, 1 H, J = 7.5 Hz, H_{para}), 6.65 (t, 2 H, J = 7.8 Hz, H_{meta}), 6.21 (d, 2 H, J = 7.3 Hz, H_{ortho}), 3.24 (d, 1 H, J = 4.5 Hz, CH_AH_BPh), 2.00 (d, 1 H, J = 4.5 Hz, CH_AH_BPh), 1.53 (s, 15 H, C–CH_3), 0.30 (s, 9 H, $Si(CH_3)_3$), −0.64 (d, 1 H, J = 13.1 Hz, $CH_AH_BSiMe_3$), −4.49 (d, 1 H, J = 13.1 Hz, $CH_AH_BSiMe_3$). $^{13}C\{^1H\}$ NMR (75 MHz, C_6D_6): δ 133.8 (C_{ortho}), 130.5 (C_{para}), 128.6 (C_{meta}), 115.7 (C_{ipso}), 108.4 (C–CH_3), 46.1 (CH_2Ph), 19.5 (CH_2Si), 10.6 (C–CH_3), 3.8 (Si–CH_3). Low-resolution mass spectrum (probe temperature 150 °C): m/z 441 [P^+].

(6) Crystals of Cp*Mo(NO)(CH_2SiMe_3)(η^2-CH_2Ph) are triclinic, with a = 8.3901 (10) Å, b = 9.3937 (17) Å, c = 14.9556 (19) Å, α = 74.017 (11)°, β = 98.009 (9)°, γ = 86.849 (12)°, D_c = 1.31 g cm^{-3}, Z = 2, and space group $P\bar{1}$. Of 5092 unique reflections measured at 22 °C to $2\theta_{max}$ = 55° with Mo Kα graphite-monochromated radiation on an Enraf-Nonius CAD4-F diffractometer, 4328 (85.0%) were considered to be observed with $I \geq 3\sigma(I)$. The structure was solved by conventional heavy-atom methods and refined by full-matrix least-squares procedures with absorption-corrected data, the transmission factors ranging from 0.81 to 0.89. Refinement of all non-hydrogen atoms with anisotropic thermal parameters and hydrogen atoms with isotropic thermal motion was effected to give R = 0.023 and R_w = 0.031. The maximum parameter shift for the last cycle of refinement was 0.007σ; residual electron density in the final difference Fourier map ranged from −0.6 to +0.3 e $Å^{-3}$.

(7) Legzdins, P.; Martin, J. T.; Einstein, F. W. B.; Jones, R. H. *Organometallics* **1987**, *6*, 1826.

(8) Legzdins, P.; Rettig, S. J.; Sanchez, L. *Organometallics* **1988**, *7*, 2394.

(9) Brookhart, M.; Green, M. L. H. *J. Organomet. Chem.* **1983**, *250*, 395.

(10) For comparison, the Mo–C formal single-bond length in the Mo–CH_2SiMe_3 portion of the molecule is 2.218 (2) Å.

(11) Becker, E. D. *High Resolution NMR*, 2nd ed.; Academic Press: New York, 1980; pp 73–74.

The spectroscopic properties of Cp*Mo(NO)(CH_2SiMe_3)(η^2-CH_2Ph)[5] indicate that it retains the molecular structure shown in Figure 1 in solution. A notable feature of its ^1H NMR spectrum in C_6D_6 is the high-field position of the AB doublet due to the diastereotopic methylene protons of the CH_2SiMe_3 ligand. This feature is undoubtedly a manifestation of the shielding provided by the still-extant ring current of the benzyl's phenyl ring.[11]

The propensity to attach benzyl ligands in a η^2 manner appears to be a general property of this class of compounds since a variety of Cp′M(NO)X(η^2-CH_2Ph) complexes (Cp′ = Cp, Cp*; M = Mo, W; X = alkyl, halide) display this structural feature.[4,12] Indeed, a closer examination of similar structures exhibited by d^0 and d^0f^n metal benzyl complexes[13] may well reveal that their M-CH_2Ph interactions should also be formulated as in B.

Representation B above reflects primarily a valence-bond description of the Mo-CH_2Ph bonding in Cp*Mo(NO)(CH_2SiMe_3)(η^2-CH_2Ph). It does not, however, provide a rationale for why the benzyl ligand in this compound does not coordinate in a η^3 fashion to the metal center[14] and undergo the characteristic σ–π electronic distortion found for the η^3-allyl ligands in the related CpMo(NO)(η^3-allyl)X compounds.[15] Perhaps a detailed molecular orbital description of the bonding in Cp*Mo(NO)(CH_2SiMe_3)(η^2-CH_2Ph) or a related complex can provide the requisite insights.

Acknowledgment. We are grateful to the Natural Sciences and Engineering Research Council of Canada for support of this work in the form of grants to P.L. and J.T. and a postgraduate scholarship to N.H.D.

Supplementary Material Available: Tables of fractional coordinates and isotropic and anisotropic thermal parameters for Cp*Mo(NO)(CH_2SiMe_3)(η^2-CH_2Ph) (3 pages); a table of structure factors (22 pages). Ordering information is given on any current masthead page.

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(12) Legzdins, P.; Phillips, E. C. Manuscript in preparation.

(13) That benzyl ligands can attach to metal centers in a η^2 fashion has been noted previously by other workers (cf.: Jordan, R. F.; LaPointe, R. E.; Bajgur, C. S.; Echols, S. F.; Willett, R. *J. Am. Chem. Soc.* **1987**, *109*, 4111 and references cited therein). However, we are the first to invoke that these ligands then provide three electrons to the metal centers in the manner represented by resonance hybrid B.

(14) There does not appear to be a steric encumbrance to such a coordination mode.

(15) Greenhough, T. J.; Legzdins, P.; Martin, D. T.; Trotter, J. *Inorg. Chem.* **1979**, *18*, 3268 and references cited therein.

Dimetallostannylene-Transition-Metal Complexes

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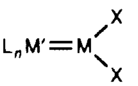
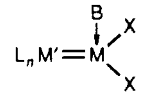
Summary: The crystalline complex [{Fe(CO)$_3$(μ-CO)}(SnFp$_2$)]-(*O*,*Sn*) (**1**), [{Cr(CO)$_4$(μ-CO)}(SnFp$_2$)]$_2$-(*O*,*Sn*) (**2**), and [Fe(CO)$_4${SnFp$_2$(Py)}] (**3**) [Fp = Fe(η-C_5H_5)(CO)$_2$] have been prepared; X-ray data on **1** and **2** show them to be centrosymmetric dimers by virtue of two isocarbonyl-tin bonds.

Heavy group 14 metal carbene analogues MX_2 or related neutral Lewis base (B) adducts MX_2(B) have an extensive transition-metal (M′ or M″) coordination chemistry as terminal ligands, forming complex I or II (L_n, L_m = the sum of all the ligands at M′ or M″).[1] In I and II, X has generally been a monoanionic C-, N-, O-, P-, or S-centered ligand or a halide. Rarely has X been a transition-metal-centered group, and then the derived complexes invariably had structure III, in which the main-group metal M is part of an $\overline{M'MM'}$ ring: [W(CO)$_5${M(W$_2$(CO)$_{10}$)}] (M = Ge, Sn),[2a] [W(CO)$_5${Sn(THF)(W$_2$(CO)$_{10}$)}],[2b] or [MnCp(CO)$_2${M(MnCp(CO)$_2$)$_2$}] (Cp = η-C_5H_5, η-C_5H_4Me, M = Ge;[2c,d] Cp = η-C_5H_4Me, M = Sn;[2e] Cp = η-C_5H_5, M = Pb[2f]).



I



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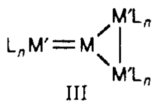
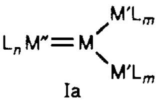
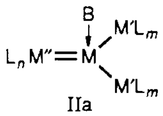


III

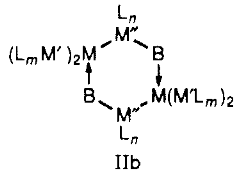
We now report two members of a new class of complex, the *acyclic* dimetallostannylenes (Ia): [Fe(CO)$_4$(SnFp$_2$)] (**1a**) and [Cr(CO)$_5$(SnFp$_2$)] (**2a**; Fp = [Fe(η-C_5H_5)(CO)$_2$]).



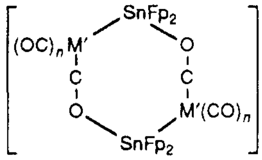
Ia



IIa



IIb



1: M′ = Fe, *n* = 3
2: M′ = Cr, *n* = 4

[M′(CO)$_{n+1}$(SnFp$_2$)]
1a: M′ = Fe, *n* = 3
2a: M′ = Cr, *n* = 4

[Fe(CO)$_4${SnFp$_2$(py)}]
3

Complexes **1a** and **2a** are dimers **1** and **2** in the crystalline state, by virtue of two isocarbonyl-tin bonds, M′CO$^+$–$^-$SnFp$_2$. Their thermal stability is surprising, as facile reductive elimination of Fp$_2$ was expected.[3] The complexes [Fe(CO)$_4${SnFp$_2$(Py)}] (**3**), [{Fe(CO)$_3$(μ-CO)}(SnFp$_2$)]$_2$-(*O*,*Sn*) (**1**) and [{Cr(CO)$_4$(μ-CO)}(SnFp$_2$)]$_2$-(*O*,*Sn*) (**2**) are representative of two further new classes IIa and IIb: **2** is the first mixed dimetallostannylene-metal complex (i.e., M′ ≠ M″).

The dimetallostannylene complexes **1** and **2** were obtained by alternative alkali-metal chloride eliminations, (i) [M′(CO)$_{n+1}$]$^{2-}$/SnCl$_2$Fp$_2$ or (ii) (for M′ = Cr) [Cr(CO)$_5${SnCl$_2$(THF)}]/2 KFp, yielding ca. 50% yields of red crystalline **1**[4] and **2**,[5] respectively (slow crystallization from

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(1) Petz, W. *Chem. Rev.* **1986**, *86*, 1019. Lappert, M. F. *Silicon, Germanium, Tin, Lead Compd.* **1986**, *9*, 129.

(2) (a) Huttner, G.; Weber, U.; Sigwarth, B.; Scheidsteger, O.; Lang, H.; Zsolnai, L. *J. Organomet. Chem.* **1985**, *282*, 331. (b) Scheidsteger, O.; Huttner, G.; Dehnicke, K.; Pebler, J. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 428. (c) Melzer, D.; Weiss, E. *J. Organomet. Chem.* **1984**, *263*, 67. (d) Gäde, W.; Weiss, E. *J. Organomet. Chem.* **1981**, *213*, 451. (e) Herrmann, W. *Angew. Chem., Int. Ed. Engl.* **1986**, *25*, 56. (f) Kneuper, H.-J.; Herdweck, E.; Herrmann, W. *J. Am. Chem. Soc.* **1987**, *109*, 2508.

(3) Low-coordinate lead(II) complexes M′X$_2$ featuring [MoCp(CO)$_3$]$^-$ σ-substituents (X) are very sensitive to elimination of the Mo–Mo-bonded dimer with concomitant deposition of metallic lead; cf.: Hitchcock, P. B.; Lappert, M. F.; Michalczyk, M. J. *J. Chem. Soc., Dalton Trans.* **1987**, 2635.

(4) [{Fe(CO)$_3$(μ-CO)}(SnFp$_2$)]$_2$-(*O*,*Sn*) (**1**). IR (Nujol, cm^{-1}): ν(CO) 2948, 2025, 2016, 2001, 1984, 1966, 1952, 1930, 1894; ν(CO→Sn) 1824. Anal. Calcd for $C_{36}H_{20}Fe_6O_{16}Sn_2$: C, 33.7; H, 1.58. Found: C, 33.6; H, 1.61. Crystal data for **1**: monoclinic, space group $P2_1/n$, a = 11.041 (5) Å, b = 12.588 (5) Å, c = 15.029 (8) Å, β = 99.91 (4)°, V = 2057.5 Å^3, Z = 2, D_c = 2.07 g cm^{-3}, $F(000)$ = 1240 (monochromated Mo Kα radiation, λ = 0.71069 Å, μ = 33.2 cm^{-1}). Refinement of 3069 reflections with $|F^2| > \sigma(F^2)$ converged with final residuals R = 0.032 and R' = 0.043.

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