Synthesis and reactions of the first heterodinuclear methoxycarbyne complex (.eta.5-C5H5)(CO)Fe(.mu.-COCH3)(.mu.-CO)Mn(CO)(.eta.5-CH3C5H4)

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facile scrambling of the CO_2 and CO oxygen atoms in $Li[Fp(CO_2)]$,[11] the appearance of $C^{18}O$ in $[Fp_2]$ supports the intermediacy of $Li[Fp(CO^{18}O)]$. This is most reasonably formed by oxide transfer from $Li_2[W(CO)_5(C^{18}O_2)]$, and alternative interpretations involving CO_2 transfer were ruled out by the results of the ^{13}C experiment, which gave $[Fp_2]$ which was 99% unlabeled.

The $[W(CO_6]$ samples formed in the ^{18}O and ^{13}C labeling experiments were determined to be 61% and 68% monolabeled, respectively, while a further 3% of the material from each experiment contained two labels,[16] suggesting that ca. 30% of the observed $[W(CO)_6]$ had not been formed from $Li_2[W(CO)_5(CO_2)]$, but was a byproduct of the sequence used to prepare $Li_2[W(CO)_5(CO_2)]$. This further implies that the yield of $[W(CO)_6]$ from the reaction with $[Fp(CO)]BF_4$ was ca. 30% less than the spectroscopic yield, bringing the yield into reasonable agreement with the stoichiometry predicted for the formation of $[Fp_2]$ according to eq 1 and 2 after allowing for the persistence of some $Li[Fp(CO_2)]$.

We conclude that the reaction of $Li_2[W(CO)_5(CO_2)]$ with $[Fp(CO)]BF_4$ to give $[Fp_2]$ probably involves initial oxide ion transfer, and this suggests that oxide ion transfer to electrophilic substrates may indeed be a characteristic reaction of carbon dioxide when activated by coordination to an anionic transition-metal complex.

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Registry No. $Li_2[W(CO)_5(CO_2)]$, 96807-09-9; $[Fp(CO)]BF_4$, 12244-69-8; $[W(CO)_6]$, 14040-11-0; $[Fp_2]$, 38117-54-3; $Li_2[W_2(CO)_{10}]$, 96807-10-2; $Li[Fp(CO_2)]$, 96807-11-3; oxide, 16833-27-5.

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[16] The presence of a small quantity of *bis*-labeled complex is indicative of some scrambling within the labeled $Li_2[W(CO)_5(CO_2)]$.[2]

Synthesis and Reactions of the First Heterodinuclear Methoxycarbyne Complex $(\eta^5\text{-}C_5H_5)(CO)Fe(\mu\text{-}COCH_3)(\mu\text{-}CO)Mn(CO)(\eta^5\text{-}CH_3C_5H_4)$

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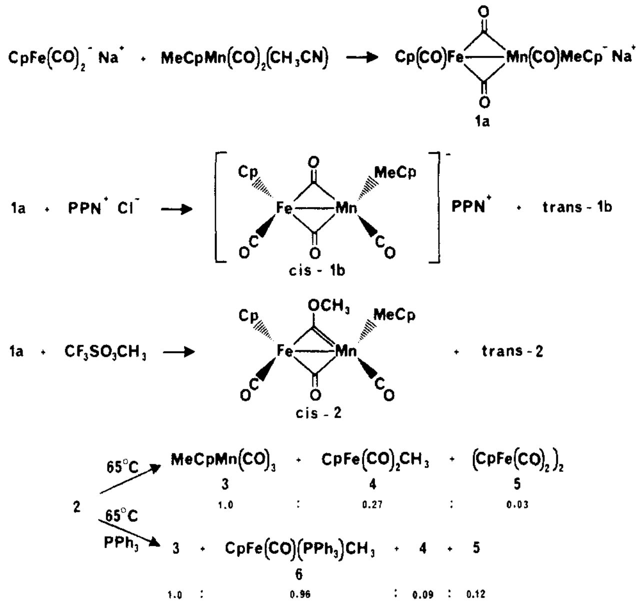
Summary: Alkylation of the new anion $Cp(CO)Fe(\mu\text{-}CO)_2Mn(CO)MeCp^-$ (**1**) with methyl triflate gives the novel heterodinuclear methoxycarbyne complex $Cp(CO)Fe(\mu\text{-}COMe)(\mu\text{-}CO)Mn(CO)MeCp$ (**2**). Thermal decomposition of **2** in the presence of triphenylphosphine gives $MeCpMn(CO)_3$ and $CpFe(CO)(PPh_3)CH_3$. Kinetic and isotopic labeling studies of this methyl migration are described.

Recent work on the Fischer–Tropsch reaction[1] has led to wide acceptance[2] of a carbon–carbon bond-forming mechanism involving surface-bound methylene polymerization. The key proposed intermediate might most simply be modelled by a dinuclear η^1-alkyl, μ-methylene complex. While many dinuclear μ-methylene compounds are known,[3] only a single type of system is known that also contains an additional η^1-alkyl group.[4,5] A stepwise approach to the synthesis of such compounds might reasonably involve alkylation of a dinuclear anion. We report here the synthesis of a new heterodinuclear anion, its alkylation to give not an alkyl complex but rather a novel heterodinuclear methoxycarbyne complex, and its decomposition to give a mononuclear alkyl complex. This final

Scheme I



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[1] (a) Brady, R. C., III; Pettit, R. *J. Am. Chem. Soc.* **1980**, *102*, 6181–6182. (b) Brady, R. C., III; Pettit, R. *Ibid.* **1981**, *103*, 1287–1289.

[2] (a) Herrmann, W. A. *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 117–130. (b) Parshall, G. W.; Thorn, D. L.; Tulip, T. H. *CHEMTECH* **1982**, 571–576. (c) Other mechanisms have also been proposed, but these have less organometallic precedent: see ref 2a.

[3] For a recent review, see: Herrmann, W. A. *Adv. Organomet. Chem.* **1982**, *20*, 159–263.

[4] (a) Isobe, K.; Andrews, D. G.; Mann, B. E.; Maitlis, P. M. *J. Chem. Soc., Chem. Commun.* **1981**, 809–810. (b) Isobe, K.; Bailey, P. M.; Schofield, P.; Gauntlett, J. T.; Nutton, A.; Maitlis, P. M. *Ibid.* **1982**, 425–426. (c) Vazquez de Miguel, A.; Isobe, K.; Taylor, B. F.; Nutton, A.; Maitlis, P. M. *Ibid.* **1982**, 758–759. (d) Nutton, A.; Vazquez de Miguel, A. Isobe, K.; Maitlis, P. M. *Ibid.* **1983**, 166–167. (e) Isobe, K.; Vazquez de Miguel, A.; Bailey, P. M.; Okeya, S.; Maitlis, P. M. *J. Chem. Soc., Dalton Trans.* **1983**, 1441–1447.

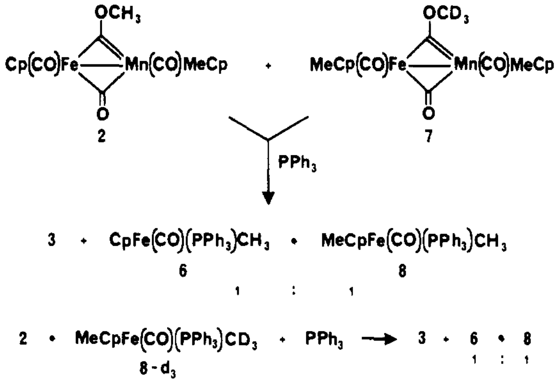
[5] For examples of compounds containing a bridging alkylidene and an additional (non-alkyl) σ-bound group, specifically η^1-acetylide (a), μ_3-Ph_2C_2 (b), μ-alkylidene (c–g), and μ-H (h), see: (a) Afzal, D.; Lenhert, P. G.; Lukehart, C. M. *J. Am. Chem. Soc.* **1984**, *106*, 3050–3052. (b) Clauss, A. P.; Shapley, J. R.; Wilson, S. R. *J. Am. Chem. Soc.* **1981**, *103*, 7387–7388. (c) Yamamoto, T.; Garber, A. R.; Wilkinson, J. R.; Boss, C. B.; Streib, W. E.; Todd, L. J. *J. Chem. Soc., Chem. Commun.* **1974**, 354–356. (d) Fischer, E. O.; Lindner, T. L.; Fischer, H.; Huttner, G.; Friedrich, P.; Kreissl, F. R. *Z. Naturforsch., B: Anorg. Chem., Org. Chem.* **1977**, *32B*, 648–652. (e) Hursthouse, M. B.; Jones, R. A.; Malik, K. M. A.; Wilkinson, G. *J. Am. Chem. Soc.* **1970**, *101*, 4128–4139. (f) Schmidt, G. F.; Muetterties, E. L.; Beno, M. A.; Williams, J. M. *Proc. Natl. Acad. Sci. U.S.A.* **1981**, *78*, 1318–1320. (g) Cooke, M.; Davies, D. L.; Guerchais, J. E.; Knox, S. A. R.; Mead, K. A.; Roue, J.; Woodward, P. *J. Chem. Soc., Chem. Commun.* **1981**, 862–864. (h) Calvert, R. B.; Shapley, J. R. *J. Am. Chem. Soc.* **1978**, *100*, 7726–7727.

step, in which an unprecedented oxygen to metal methyl migration occurs, suggests the possibility that the observed methoxycarbyne moiety and the desired η^1-alkyl, μ-carbonyl moiety could lie on the same reaction coordinate.

Refluxing a 1:1 mixture of $MeCpMn(CO)_2(CH_3CN)$ ($MeCp = \eta^5\text{-}CH_3C_5H_4$) and $CpFe(CO)_2^-Na^+$ ($Cp = \eta^5\text{-}C_5H_5$) in THF overnight gave in 87% yield a red solid identified as the sodium salt of the dinuclear anion $Cp(CO)Fe(\mu\text{-}CO)_2Mn(CO)MeCp^-$ (**1a**)[6] (Scheme I). Metathesis with PPN^+Cl^- (PPN^+ = bis(triphenylphosphine)nitrogen(1+)) gave the PPN^+ salt **1b**[6] as a dark purple solid in 76% yield. The anion is isoelectronic with the well-known iron dimer $[CpFe(CO)_2]_2$, and spectroscopically it appears to be isostructural as well,[7] both with respect to carbonyl geometry and the presence of cis and trans isomers. Heating **1b** in the NMR probe to 320 K resulted in coalescence of the two *Me*Cp signals, consistent with rapid cis–trans isomerization; line-shape analysis[8] gave $\Delta G^{\ddagger}(320\text{ K}) = 16.2 \pm 0.8$ kcal/mol. By contrast, the barrier to cis–trans isomerization in $[CpFe(CO)_2]_2$ is only 10.4 kcal/mol at 220 K.[7a]

Alkylation of **1a** was accompanied by rapid addition of 1.1 equiv of $CF_3SO_3CH_3$ to a THF solution of **1a** at room temperature. Solvent removal and crystallization from pentane/ether gave a 60% yield of a red-purple solid that was identified as the dinuclear carbyne complex **2**[6] formed by apparent O-alkylation rather than as the anticipated η^1-alkyl complex that would have been formed by M-alkylation (Scheme I). To the best of our knowledge, **2** is the first example of a neutral heterodinuclear methoxycarbyne complex. The most closely related compound is Cutler's dinuclear cation $[CpFe]_2(\mu\text{-}COCH_3)(\mu\text{-}CO)(\mu\text{-}Ph_2PCH_2CH_2PPh_2)^+$;[9e] a set of neutral homopolynuclear iron, ruthenium, and osmium clusters which contain an edge-bridging methoxycarbyne ligand is also known.[9a–d,f] The key spectroscopic data in support of the carbyne formulation are the appearance of the new methyl signal in the ^1H NMR at δ 5.02 (acetone-d_6), confirmed by the use of $CF_3SO_3CD_3$ for the alkylation, and of the carbyne carbon signal in the ^{13}C NMR (acetone-d_6) at 390.7 ppm and the new methyl signal at 73.2 ppm. The methyl signal in both spectra is clearly characteristic of a methoxy group, while the downfield carbyne signal is characteristic of those in tolylidyne complexes[10] as well as in the related methoxycarbyne clusters mentioned above, in which the carbyne carbon was observed at 352–382 ppm.[9a–d,f] The proposed arrangement of carbonyl ligands is based on the infrared spectrum, which shows one bridging and two terminal carbonyl bands, while the double bond between the carbyne carbon and manganese is proposed based solely on the effective atomic number rule.

Scheme II



The characterization of **2** was complicated by the fact that it, like **1b**, apparently exists as a mixture of cis and trans isomers. At ambient temperature, isomer ratios of 100:0, 74:26, and 64:36 were observed in acetone, methylene chloride, and benzene solution, respectively, as judged by ^1H NMR. These results are in accord with the more abundant isomer being the cis isomer, since like $[CpFe(CO)_2]_2$ it would be expected to have a larger dipole moment and therefore be present in larger proportions in solvents of higher dielectric constant.[7] Typical coalescence behavior was observed upon heating a sample of **2** in benzene in the NMR; the activation barrier to cis–trans[11] isomerization at 320 K was determined[8] to be roughly 16.8 ± 1.8 kcal/mol, which is curiously similar to the barrier found for **1b**.

Heating a benzene solution of **2** to 65 °C resulted in slow decomposition (130 h) to $MeCpMn(CO)_3$ (**3**), $CpFe(CO)_2CH_3$ (**4**), and $[CpFe(CO)_2]_2$ (**5**). A somewhat cleaner decomposition was observed in the presence of 1.2 equiv of triphenylphosphine, giving $CpFe(CO)(PPh_3)CH_3$ (**6**) as the principal methyl complex (Scheme I).[12] Due to the novelty of this apparent methyl migration, this reaction was examined in some detail.

First-order decomposition of **2** was observed both thermally and in the presence of a fivefold excess of triphenylphosphine. The dependence of the rate on phosphine concentration revealed the presence of two decomposition pathways, one a phosphine-independent pathway with the same rate constant as that of simple thermal decomposition, and the other a first-order phosphine-dependent pathway. A crossover experiment (Scheme II) involving the decomposition of a mixture of **2** and doubly labeled **7** was carried out next, at a phosphine concentration (0.1 M) that would allow most of the reaction to proceed via the phosphine-independent kinetic pathway. After 3 h at 65 °C, a 1:1 mixture of **6** and **8** was observed by ^1H NMR, indicating that complete *intermolecular* exchange or migration of the methyl group had occurred. A control experiment in which a mixture of products **6** and **8**-d_3 was heated for 9 h at 65 °C showed only a small amount of methyl exchange. A reasonable mechanism for this process would involve slow (rate-determining) dissociation of phosphine to give $CpFe(CO)CH_3$ (**9**), which,

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(6) Data for new compounds. **1a**: IR (THF) 1909 (s), 1850 (s), 1723 (m), 1648 (s) cm^{-1}; ^1H NMR (CD_3CN) δ 4.38 (s, 5 H), 4.10 (br s, 4 H), *Me*Cp obscured by solvent. **1b**: IR (CH_2Cl_2) 1899 (s), 1832 (m), 1671 (s) cm^{-1}; ^1H NMR (CD_2Cl_2) δ 4.38 (s, 5 H), 4.15–4.00 (br m, 4 H), 1.98, 1.84 (br s, 1:1, 3 H). Anal. Calcd for $C_{51}H_{42}NO_4P_2FeMn$: C, 67.64; H, 4.67; N, 1.55. Found: C, 67.41; H, 4.61; N, 1.55. **2**: IR (THF) 1954 (s), 1907 (s), 1774 (s) cm^{-1}; ^1H NMR (C_6D_6) δ 4.47, 4.26 (s, trans, cis *Cp*Fe), 4.19 (s, cis, trans *Me*O), 4.01, 3.92 (br m, Me*Cp*Mn), 1.84, 1.78 (s, cis, trans *Me*CpMn); ^{13}C NMR (C_6D_6) δ 394.6, 391.2 (trans, cis COMe), 270.8, 269.9 (cis, trans μ-*C*O), 228.1, 214.1 (*C*O), 104.7, 103.7 (trans, cis η-$CH_3CC_4H_4Mn$), 84.2–89.4 (*Cp*Fe, Me*Cp*Mn), 72.0 (cis, trans, *Me*O), 13.2, 12.8 (cis, trans, *Me*CpMn); MS (EI), *m/e* 382 (M^+), 354, 326, 311, 283, 255. Anal. Calcd for $C_{16}H_{15}O_4FeMn$: C, 50.30; H, 3.96. Found: C, 49.90; H, 3.67.

(7) (a) Bullitt, J. G.; Cotton, F. A.; Marks, T. J. *Inorg. Chem.* **1972**, *11*, 671–676 and references therein. (b) Gansow, O. A.; Burke, A. R.; Vernon, W. D. *J. Am. Chem. Soc.* **1972**, *94*, 2550–2552.

(8) Line-shape analyses were carried out by using a program written by F. A. L. Anet and modified by S. S. Miura.

(9) (a) Shriver, D. F.; Lehman, D.; Strope, D. *J. Am. Chem. Soc.* **1975**, *97*, 1594–1596. (b) Gavens, P. D.; Mays, M. J. *J. Organomet. Chem.* **1978**, *162*, 389–401. (c) Johnson, B. F. G.; Lewis, J.; Orpen, A. G.; Raithby, P. R.; Suss, G. *Ibid.* **1979**, *173*, 187–197. (d) Hodali, H. A.; Shriver, D. F. *Inorg. Chem.*, **1979**, *18*, 1236–1241. (e) LaCroce, S. J.; Menard, K. P.; Cutler, A. R. *J. Organomet. Chem.* **1980**, *190*, C79–C83. (f) Kolis, J. W.; Basolo, F.; Shriver, D. F. *J. Am. Chem. Soc.* **1982**, *104*, 5626–5630.

(10) Chetcuti, M. J.; Green, M.; Jeffery, J. C.; Stone, F. G. A.; Wilson, A. A. *J. Chem. Soc., Chem. Commun.* **1980**, 948–949.

(11) The trans–cis isomerization barrier is 16.5 ± 1.8 kcal–mol; work is in progress to refine these numbers, which depend on the temperature dependence of both the cis–trans ratios and the chemical shifts.

(12) In both cases, relative yields are shown in Scheme I, but quantitative NMR experiments at 75 °C indicate the absolute yields of $MeCpMn(CO)_3$ to be 80–97%.

having the free coordination site and low steric hindrance that are presumably necessary for methyl exchange, leads to fast scrambling of the methyl labels.[13] *We suggest that this same intermediate is responsible for methyl exchange in the thermal decomposition of 2.* Thus, **9** (and **3**) may reasonably form in (or following) the rate-determining step in the thermal decomposition of **2**, after which **9** can be trapped by triphenylphosphine to give **6**. However, **9** might also react with **8** (resulting in methyl exchange) *prior* to trapping by triphenylphosphine. If this hypothesis is correct, then reaction of **2** and **8**-d_3 (Scheme II) ought to result in a high degree of methyl exchange between the carbyne decomposition product **6** and **8**-d_3. In fact, nearly a 1:1 mixture of **6** and **8** was observed after 3 h at 65 °C. Thus while it will be impossible (at least in this particular system) to determine if methyl scrambling occurs during the migration from oxygen to iron, it clearly occurs among the products under the reaction conditions.

Work is continuing in our laboratory to elucidate the details of the cis–trans isomerizations and the methyl exchange and carbyne decomposition reactions described above, as well as to introduce the additional μ-methylene moiety in place of the μ-carbonyl ligand.

Acknowledgment. Financial support for this work from Research Corp., the UCLA Committee on Research, and the Universitywide Energy Research Group is gratefully acknowledged.

Registry No. **1a**, 97011-38-6; **1b**, 97011-40-0; *cis*-**1b**, 97100-76-0; *trans*-**1b**, 97100-78-2; *cis*-**2**, 97011-41-1; *trans*-**2**, 97100-79-3; **3**, 12108-13-3; **4**, 12080-06-7; **5**, 12154-95-9; **6**, 12100-51-5; **7**, 97011-42-2; **8**, 97011-43-3; **8**-d_3, 97011-44-4; (MeCp)Mn(CO)$_2$(CH$_3$CN), 82648-13-3; CpFe(CO)$_2^-$Na$^+$, 12152-20-4.

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[13] Bryndza, H. E.; Evitt, E. R.; Bergman, R. G. *J. Am. Chem. Soc.* **1980**, *102*, 4948–4951.

New Types of Organometallic Oxo Complexes Containing Tungsten

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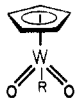
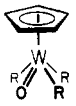
Summary: The preparation and isolation of the first examples of three new types of cyclopentadienyl oxo alkyl complexes are described. The complexes (η^5-C$_5$H$_5$)W(O)$_2$(CH$_2$SiMe$_3$), **1**, (η^5-C$_5$H$_5$)W(O)(CH$_2$SiMe$_3$)$_3$, **2**, and (η^5-C$_5$H$_5$)W(O)(CHSiMe$_3$)(CH$_2$SiMe$_3$), **3**, have been characterized by conventional spectroscopic methods, and the molecular structures of **1** and **2** in the solid state have been determined by single-crystal X-ray crystallography.

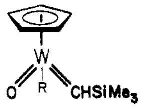
Interest in molecules which contain both organic and oxo groups attached to a metal atom derives from the expectation that their characteristic chemistry will provide some insight into how metal oxides heterogeneously catalyze various organic transformations.[1] Recent reports concerning the preparation and characterization of diverse organometallic oxo complexes such as (η^5-C$_5$R'$_5$)ReO$_3$ (R' = H[2] or Me[3]), (η^5-C$_5$H$_5$)Re(O)Me$_2$,[2b] Re(O)I(MeC≡CMe)$_2$,[4] and W$_2$O$_3$(CH$_2$CMe$_3$)$_6$[5] suggest that these types of compounds may be more accessible than was previously believed.[6] Indeed, during our investigations of the chemical properties of (η^5-C$_5$H$_5$)W(NO)(CH$_2$SiMe$_3$)$_2$,[7] we have encountered the first examples of three new types of cyclopentadienyl oxo alkyl complexes. Consequently, we now wish to report the isolation and characterization of these compounds **1**–**3** depicted below (R = CH$_2$SiMe$_3$).



1



2



3

Compound **1** may be conveniently synthesized by exposing a hexanes solution of (η^5-C$_5$H$_5$)W(NO)(CH$_2$SiMe$_3$)$_2$ to an atmosphere of O$_2$ overnight, i.e., eq 1, and it is

$$(\eta^5\text{-C}_5\text{H}_5)\text{W(NO)(CH}_2\text{SiMe}_3)_2 \xrightarrow[\text{hexanes, 20 °C}]{\text{O}_2\text{ (1atm)}} (\eta^5\text{-C}_5\text{H}_5)\text{W(O)}_2(\text{CH}_2\text{SiMe}_3) \quad (1)$$

isolable from the final reaction mixture as analytically pure, white crystals in 65% yield by fractional crystallization. Compound **2** is formed as a byproduct during the preparation of (η^5-C$_5$H$_5$)W(NO)(CH$_2$SiMe$_3$)$_2$ from [(η^5-C$_5$H$_5$)W(NO)I$_2$]$_2$[7] if an excess of Grignard reagent is employed. It is separable by chromatography of the dried reaction residue on Florisil with hexanes as eluant and is obtainable from the eluate as lemon yellow crystals in 3% yield (based on W) by subsequent crystallization. Compound **3** results from the thermal decomposition of solid (η^5-C$_5$H$_5$)W(NO)(CH$_2$SiMe$_3$)$_2$, i.e., eq 2, and is isolable as

$$(\eta^5\text{-C}_5\text{H}_5)\text{W(NO)(CH}_2\text{SiMe}_3)_2 \xrightarrow[\text{20 °C, 30 days}]{\text{N}_2\text{ (1atm)}} (\eta^5\text{-C}_5\text{H}_5)\text{W(O)(CHSiMe}_3)(\text{CH}_2\text{SiMe}_3) + \text{other products} \quad (2)$$

a crystalline, pale yellow solid in 40% yield by fractional crystallization of the final mixture from hexanes. Interestingly, the alkylidene compound **3** is also obtainable in virtually quantitative yields by the thermal decomposition of **2**, i.e., eq 3. This is the first instance of an oxo alkyl

$$(\eta^5\text{-C}_5\text{H}_5)\text{W(O)(CH}_2\text{SiMe}_3)_3 \xrightarrow[\text{50 °C, 2 days}]{\text{benzene}} (\eta^5\text{-C}_5\text{H}_5)\text{W(O)(CHSiMe}_3)(\text{CH}_2\text{SiMe}_3) + \text{Me}_4\text{Si} \quad (3)$$

complex converting to an isolable oxoalkylidene complex, a possibility previously envisaged by Pedersen and Schrock.[8]

Single-crystal X-ray crystallographic analyses of **1**[9] and **2**[10] have confirmed their monomeric natures and have

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[1] Sheldon, R. A.; Kochi, J. K. "Metal Catalyzed Oxidations of Organic Compounds"; Academic Press: New York, 1981.

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[3] Klahn-Oliva, A. H.; Sutton, D. *Organometallics* **1984**, *3*, 1313.

[4] Mayer, J. M.; Tulip, T. H. *J. Am. Chem. Soc.* **1984**, *106*, 3878.

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[7] Legzdins, P.; Rettig, S. J.; Sánchez, L.; Bursten, B. E.; Gatter, M. G. *J. Am. Chem. Soc.* **1985**, *107*, 1411.

[8] Pedersen, S. F.; Schrock, R. R. *J. Am. Chem. Soc.* **1982**, *104*, 7483 and references therein.

[9] Crystals of **1** are monoclinic of space group $P2_1/n$ with a = 6.3178 (7) Å, b = 19.9826 (9) Å, c = 9.6923 (11) Å, β = 93.320 (5)°, and Z = 4. The structure was solved by conventional heavy-atom methods and was refined by full-matrix least-squares procedures to R = 0.029 and R_w = 0.032 for 2220 absorption-corrected reflections with $I > 3\sigma(I)$ collected at 22 °C with Mo Kα radiation on an Enraf-Nonius CAD4-F diffractometer. Hydrogen atoms were fixed in idealized positions.

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