stoichiometry of which is still being studied, a high conversion to $Cp_3Rh_3(CO)_2(CR)^+$ appears to occur without formation of $CpRh(CO)_2$.[9]

Stirring a solution of $Cp_3Rh_3(CO)_2(CH)^+PF_6^-$ under an atmosphere of ^{13}CO at 40 °C leads to ^{13}CO enrichment without significant decomposition over ca. 2 days. More elevated temperatures, however, do lead to decomposition. Significantly, treatment of $Cp_3Rh_3(CO)_2(CH)^+$ with $LiHBEt_3$ under a CO atmosphere regenerates the $Cp_2Rh_2(CO)_2(CH_2)$, apparently by hydride attack at the methylidyne carbon; $CpRh(CO)_2$ and C_{3v} $Cp_3Rh_3(CO)_3$ are formed also. Additional reactions of these cationic alkylidyne complexes are being examined.

Acknowledgment. This work has been supported by a grant from the National Science Foundation and by fellowships from the A. P. Sloan Foundation and the Camille and Henry Dreyfus Foundation to J.R.S.

Supplementary Material Available: Tables listing atomic coordinates (Table A), bond lengths (Table B), bond angles (Table C), thermal parameters (Table D), and structure factors (Table E) (30 pages). Ordering information is given on any current masthead page.

(9) As this work was being prepared for publication, we learned that Professor Herrmann and co-workers (Regensburg) also have observed the transformation of $Cp_2Rh_2(CO)_2(CH_2)$ into $[Cp_3Rh_3(CO)_2(CH)]^+$. Furthermore, they have obtained evidence that the reaction is initiated by protonation of the Rh-Rh bond, forming a hydrido-methylene complex, which then rearranges to a methyl species: Herrmann, W. A., et al. *Angew. Chem., Int. Ed. Engl.*, in press. Herrmann, W. A.; Plank, J.; Ziegler, M.; Balbach, B. *J. Am. Chem. Soc.* **1980**, *102*, 5908.

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Reaction of Diaryldiazomethanes with a Metal-Metal Triple Bond: Synthesis, Structural Characterizations, and Reactivity of Novel Bridging Diazoalkane and Alkylidene Complexes

Sir:

The reactions of diazoalkanes with organotransition-metal complexes, an area of growing interest,[1] have involved primarily mononuclear reactants. To our knowledge there have been no reported studies of diazoalkane reactions with metal-metal multiply bonded compounds. We wish to report the first example of such a reaction, in which the product has a new and unusual mode of diazoalkane coordination, and the clean conversion of the product to a novel species containing a bridging alkylidene[2] ligand. Although alkylidene complexes have been isolated previously from the reaction of diazoalkanes with metal complexes, our work also represents the first instance in which an intermediate diazoalkane complex has been characterized.

The reaction of $Cp_2Mo_2(CO)_4$[3] (**1**; Cp = η-C_5H_5) with either diphenyl- or bis(*p*-tolyl)diazomethane in CH_2Cl_2 at room temperature afforded in >90% yield the green diazoalkane adducts **2** (eq 1) which are air stable in the solid state. Analogous com-

$$Cp_2Mo_2(CO)_4 + R_2CN_2 \rightarrow \underset{\mathbf{1}}{} Cp_2Mo_2(CO)_4(N_2CR_2) \quad (1)$$
$$\mathbf{2a},\ R = Ph$$
$$\mathbf{2b},\ R = p\text{-}MeC_6H_4$$

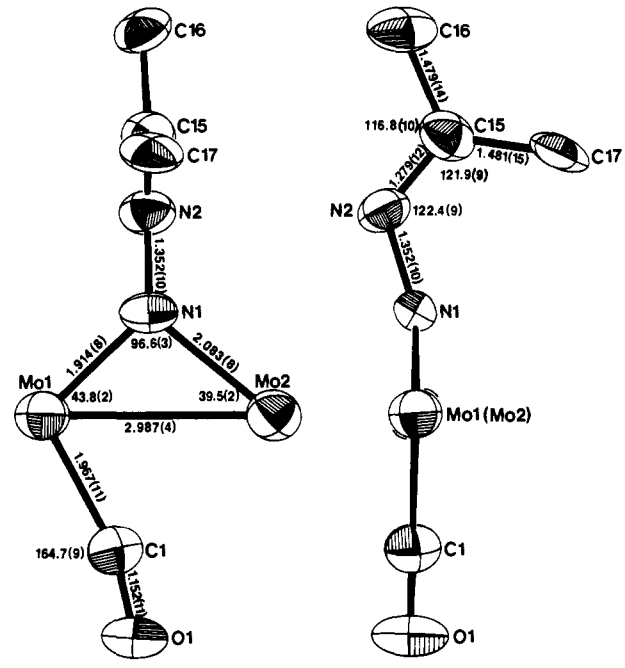

Figure 1. Two views of the molecular core of **2a**. The second view is obtained by rotating the first ~90° such that Mo1 is directly in front of Mo2. The Cp groups, the three remaining carbonyl groups, and all phenyl carbons except C_{ipso} are omitted for clarity. Bond distances are in angstroms, bond angles in degrees.

plexes can be prepared from $(\eta\text{-}C_5H_4Me)_2Mo_2(CO)_4$. Elemental analysis, ^1H and ^{13}C NMR spectroscopy, and infrared spectroscopy[4] were consistent with this adduct formulation and suggested a structure, confirmed by single-crystal X-ray diffraction, with a diazoalkane bridge between the two molybdenum atoms. The principal features of the structure,[5] shown in Figure 1, are (1) a novel N(terminal) diazoalkane bridge with approximately planar coordination about N1, (2) asymmetry in the metal-bridge distances which suggests multiple bonding character in the Mo1-N1 bond, and (3) a semibridging[6] carbonyl group, trans to the diazoalkane bridge, which reduces charge asymmetry between the two singly bonded molybdenum atoms. N1 acts presumably as a four-electron donor,[7] with two electrons involved in a double bond to Mo1 and two electrons in a dative bond to Mo2; other canonical forms may contribute to the ground-state electronic structure. The adducts **2** are fluxional in solution on the ^1H and ^{13}C NMR time scales at room temperature, with a $\Delta G^{\ddagger}$ for cyclopentadienyl group equilibration in **2a** at the coalescence temperature (241 K) of 6.3 ± 0.1 kcal mol^{-1} as determined by variable-temperature ^1H NMR studies.

These adducts can be thermally decomposed in solution (C_6H_6, 60 °C) with loss of dinitrogen to afford the stable red μ-diarylmethylene complexes **3** (eq 2) in high yield. Their formulation

$$Cp_2Mo_2(CO)_4(N_2CR_2) \xrightarrow{\Delta} Cp_2Mo_2(CO)_4(CR_2) + N_2 \quad (2)$$
$$\mathbf{3a},\ R = Ph$$
$$\mathbf{3b},\ R = p\text{-}MeC_6H_4$$

(1) Herrmann, W. A. *Angew. Chem., Int. Ed. Engl.* **1978**, *17*, 800-812 and references therein.

(2) "International Union of Pure and Applied Chemistry, Nomenclature of Organic Compounds, Sections A, B, and C"; Butterworths: London, 1971; rule A.4.

(3) Klingler, R. J.; Butler, W. M.; Curtis, M. D. *J. Am. Chem. Soc.* **1978**, *100*, 5034-5039.

(4) Anal. Calcd for $Mo_2C_{27}H_{20}O_4N_2$: C, 51.60; H, 3.21; N, 4.46. Found: C, 50.41; H, 3.19; N, 4.16. ^1H NMR (δ, $C_6D_5CD_3$, -70 °C, 89.56 MHz) 4.69 (s, 5, Cp), 4.94(s, 5, Cp), 6.37-6.45(br d, 4, H_o), 6.85-7.78 (m, H_m and H_p); ^{13}C NMR (δ, $CDCl_3$, -38 °C, 22.5 MHz, $\{^1H\}$) 93.7 (s, CN_2), 95.5 and 97.3 (s, Cp and Cp'), 127.8, 128.5, 129.1, 131.5, 135.8, and 136.2 (s, Ph and Ph'), and 247 and 241 (br s, CO and CO'); IR (cm^{-1}, KBr) 1970 m, 1925 s, 1845 s, 1815 m (ν_{CO}), 1535 w $\nu_{C=N}$).

(5) **2a** crystallizes from a cooled CH_2Cl_2/C_6H_{14} solution in the monoclinic space group $P2_1/n$ (No. 13) with lattice constants a = 11.847 (4) Å, b = 10.407 (3) Å, c = 20.572 (6) Å, β = 97.81 (2)°, Z = 4, and V = 2513 (1) Å^3; ρ_{calcd} = 1.64 g cm^{-3}, ρ_{obsd} = 1.64 g cm^{-3} (flotation). The structure was refined to anisotropic convergence by using 2306 reflections with $I > 3\sigma(I)$; the final R value was 0.048 and the weighted R value was 0.059.

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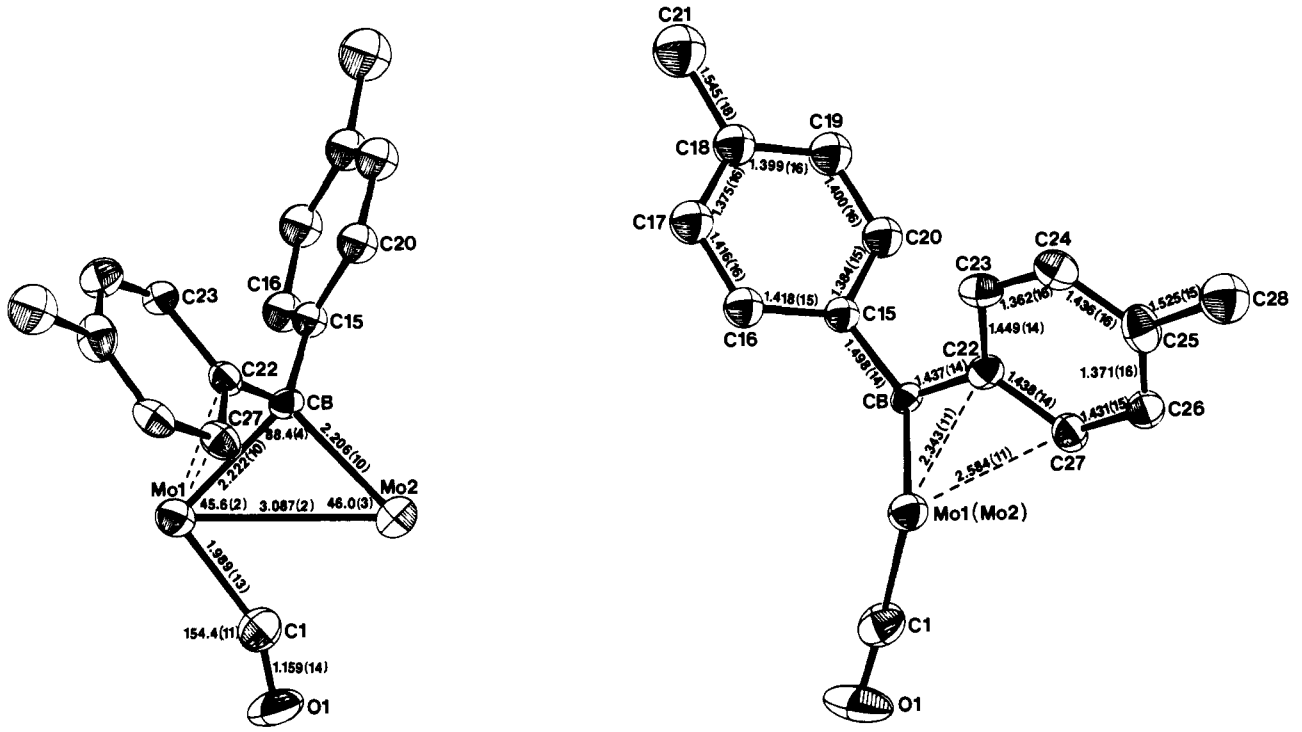
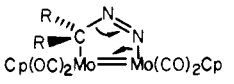


Figure 2. Two views of the molecular core of **3b**. The second view is obtained by rotating the first ~90° such that Mo1 is directly in front of Mo2. The Cp groups and the three remaining carbonyl groups are omitted for clarity.

as bridging alkylidene ligands is supported by elemental analysis,[8] which showed the absence of nitrogen, ^1H and ^{13}C NMR spectroscopy,[8] and single-crystal X-ray diffraction.[9] These complexes are also fluxional in solution, as shown by ^1H and ^{13}C NMR spectroscopy. At −66 °C the cyclopentadienyl group singlet in the 25 °C ^1H NMR spectrum of **3a** split into a doublet consistent with inequivalent Cp groups. The diphenylmethylene C_α signal at δ 176.0 in the ^{13}C NMR spectrum of **3a** was found in the same range as that reported for μ-diphenylmethylene ligands in several dinuclear rhodium complexes;[10] in addition, the slow exchange limit ^{13}C NMR spectrum of **3b** showed 4 carbonyl resonances, 12 resonances for the inequivalent aryl groups, and 2 aryl methyl resonances. Key features from the crystal structure determination (Figure 2) of **3b** are (1) a Mo1–Mo2 distance of 3.087 (2) Å consistent with a single bond,[11] (2) a semibridging[6] carbonyl group approximately trans to the μ-methylene carbon CB, and (3) a novel distortion of the bridging methylene group which brings the edge of one of the *p*-tolyl groups into bonding distance to Mo1 and results in a bond distance alternation in the interacting aryl group. The asymmetry about CB and the π interaction of the aryl group with Mo1 suggest an η^3-allyl type structure where the terminal allylic atom (CB) is also η^1-bonded to Mo2. Indeed, there are remarkable bond distance similarities between the aryl CB/Mo1 interaction in **3b** and the η^3-benzyl complex, $CpMo(CO)_2(CH_2C_6H_4$-*p*-$Me)$.[12]

All present experimental evidence is consistent with intramolecular loss of dinitrogen from the diazoalkane adduct. Observation by spectrophotometry of an isosbestic point at 554 nm during conversion of **2a** to **3a** demonstrated the absence of any appreciable concentration of an intermediate. Heating a benzene solution containing a mixture of either **2a** and $(C_5H_4Me)_2Mo_2(CO)_4$ or **2a** and $(C_5H_4Me)_2Mo_2(CO)_4[N_2C(C_6H_4$-*p*-$Me)_2]$ yielded only the direct products; no cross products ($(C_5H_4Me)_2Mo_2(CO)_4(CPh_2)$ in the former, $Cp_2Mo_2(CO)_4[C(C_6H_4$-*p*-$Me)_2]$ and $(C_5H_4Me)_2Mo_2(CO)_4(CPh_2)$ in the latter) were observed in either case. These results, coupled with the observation that $(p\text{-}MeC_6H_4)_2CN_2$ itself showed no evidence of decomposition under the thermolysis conditions, ruled out a route involving prior dissociation of the diazoalkane from **2**, decomposition of the diazoalkane to form a free carbene, and trapping of the carbene by **1**. We currently favor a cyclic transition state (I) from which dinitrogen is evolved; similar 1,3-dipolar additions



I

are observed in reactions of diazoalkanes with carbon–carbon unsaturated systems.[13]

The μ-alkylidene ligand in **3** has been found to be a reactive species toward several small molecules. The addition of dihydrogen (45 psi, 50 °C) to **3a** resulted in cleavage of the alkylidene from the metals; diphenylmethane and **1** were obtained in high yield. Interestingly, the use of a D_2/H_2 mixture in the hydrogenolysis reaction yielded Ph_2CHD in addition to Ph_2CH_2 and Ph_2CD_2. This labeling experiment demonstrates that the cleavage of R_2CH_2 is not concerted, but it is premature to speculate on the mechanism. Carbon monoxide also cleaved off the diphenylmethylene moiety under the same mild conditions to give $Cp_2Mo_2(CO)_6$, the product from carbonylation of **1**, and diphenylketene.

(8) **3a**: Anal. Calcd for $Mo_2C_{27}H_{20}O_4$: C, 54.01, H, 3.36; N, 0.00. Found: C, 55.23; H, 3.63; N, <0.1. ^1H NMR (δ, C_6D_6, 25 °C, 360 MHz) 4.270 (s, 10, Cp), 6.545 (v br s, 4, H_o), 6.688 (t, 2, J_{HH} = 8 Hz, H_p), and 6.898 (t, 4, J_{HH} = 8 Hz, H_m); ^{13}C NMR (δ, CD_2Cl_2 −60 °C, 90.56 MHz, {^1H}) 88.8, 110.8–157.4 (Ph and Ph'), 94.9 and 98.2 (Cp and Cp'), 174.9 (C_α), and 241.7, 244.1, 251.1, and 251.7 (CO).

(9) **3b** crystallizes (from toluene/methylcyclohexane as the toluene solvate) in the triclinic space group $P\bar{1}$ (No. 2) with lattice constants a = 18.481 (8) Å, b = 19.095 (5) Å, c = 10.065 (3) Å, α = 90.01 (2)°, β = 105.76 (3)°, γ = 117.01 (3)°, Z = 4, and V = 3013.6 (1.8) Å^3; ρ_{calcd} = 1.55 g cm^{-3}, ρ_{obsd} = 1.56 g cm^{-3} (flotation). The structure was refined to anisotropic convergence on 17 atoms (isotropic on all others) by using 6085 reflections with $I > 3\sigma(I)$; final R value was 0.057 and the weighted R value was 0.078.

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Transition-metal complexes have been used extensively to catalyze the transfer of carbene moieties from diazoalkanes to olefins.[1,14] It is often assumed that the metal center attacks the unique *carbon* of the diazoalkane, displacing N_2. The results reported here suggest that this view may be naive; the catalytically active species may be N bonded or polynuclear.

Complexes containing μ-alkylidene ligands are a small but growing class of compounds of considerable chemical[15] and theoretical[16] interest, and their reaction chemistry is still largely unexplored.[17] Pettit et al. have recently stressed the role of bridging alkylidenes in Fischer–Tropsch reductions of carbon monoxide to alkanes.[18]

Further chemistry of complexes of types **2** and **3** bearing on these points will be reported in the near future.

Acknowledgment. We thank the donors of the Petroleum Research Fund, administered by the Americal Chemical Society, and the National Science Foundation (Grant CHE-7907748) for support of this research. We thank Professor R. Hoffmann for a preprint describing calculations on bridging methylene complexes.

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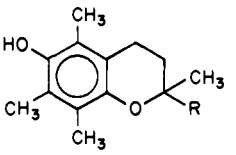
Received July 25, 1980

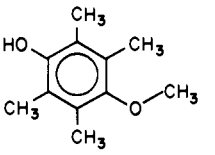
Antioxidant Activity of Vitamin E and Related Phenols. Importance of Stereoelectronic Factors[1]

Sir:

There is now a rather general agreement that α-tocopherol (**1**), the major component[2] of vitamin E, functions as an efficient inhibitor of lipid peroxidation in vivo,[3] but there is widespread confusion regarding its absolute antioxidant effectiveness in vitro. Comparisons of **1** with other natural and synthetic phenols have usually led to the conclusion that it has only a rather modest



1 R = $[CH_2CH_2CH_2CH(CH_3)]_3CH_3$



2

3 R = CH_3

antioxidant activity in vitro.[4] The apparent "discrepancy" between the high in vivo vitamin E activity of **1** and its apparently low in vitro antioxidant activity has generally been accepted uncritically. This is surprising because **1** has just those structural features in its phenolic moiety which would lead one to predict that it would be a highly efficient chain-breaking (peroxyl radical trapping) antioxidant.[5,6] That is, inhibition by phenols involves reactions 1 and 2,[5] and the magnitude of the rate constant for the rate

$$ROO\cdot + ArOH \xrightarrow{k_{inh}} ROOH + ArO\cdot \quad (1)$$

$$ROO\cdot + ArO\cdot \xrightarrow{fast} \text{nonradical products} \quad (2)$$

controlling step, k_{inh}, has been shown, in a comprehensive survey of the effect of ring substituents on the rate of reaction (1),[6] to be increased by a 4-methoxy group and by methyl groups in the 2, 3, 5, and 6 positions.[7] Furthermore, chain transfer via ArO· (which reduces the effectiveness of ArOH) is retarded when the phenoxyl oxygen is sterically protected by alkyl groups in the 2 and 6 position.[5b,5c] Chain transfer is also retarded by the electron-donating 4-methoxy group.[5b]

In an attempt to reconcile the structure of **1** with its purported low in vitro antioxidant activity, we have measured k_{inh} for **1** in the well-proven autoxidation system of styrene under 760 torr of O_2, thermally initiated with azobis(isobutyronitrile).[6,8] The standard induction period method[9] showed that **1**, like the majority of phenols,[5,6,8a,10] reacts with two peroxyls, i.e., the stoichiometric factor (n) is 2.0, as would be expected for reactions 1 and 2. For **1** at 30 °C, $k_{inh} = (23._5 \pm 5._0) \times 10^5\ M^{-1}\ s^{-1}$, which indicates that it is an extremely efficient phenolic chain-breaking antioxidant,[5,6,8a,11] just as we anticipated. Deuteration of the phenolic hydrogen in the usual way[6,8a] reduces the antioxidant activity of α-tocopherol, $k_{inh}^H/k_{inh}^D = 4.0 \pm 0.5$, showing that H-atom abstraction (reaction 1) is rate controlling, as with other phenols.

We expected that 4-methoxy-2,3,5,6-tetramethylphenol, **2**, would be equally reactive. This phenol has $n \sim 2.0$ and $k_{inh}^H/k_{inh}^D = 10.6 \pm 3.7$, but to our great surprise it has $k_{inh} = (2._1 \pm 0._2) \times 10^5\ M^{-1}\ s^{-1}$ at 30 °C. To discover whether the "vital force" (magic) of **1** resides in the phytyl side chain (R in **1**) or in the

(1) Issued as N.R.C.C. 18858.

(2) Generally ~85%; the other three components (β-, γ-, and δ-tocopherol) are close structural relatives to **1**.

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