Table II. Band Positions in μm^{-1} for $M_2(CH_3)_8{}^{n-}$ Complexes[a,b]

Complex	Energy	Assignment
$[Li(Et_2O)]_4Cr_2(CH_3)_8$	2.20[c]	$\delta \rightarrow \delta^*$ ($^1A_{2u} \leftarrow {}^1A_{1g}$)
$[Li(Et_2O)]_4Mo_2(CH_3)_8$	1.95[c]	$\delta \rightarrow \delta^*$ ($^1A_{2u} \leftarrow {}^1A_{1g}$)
	2.50	
	3.08	
$[Li(Et_2O)]_2Re_2(CH_3)_8$	1.86[c]	$\delta \rightarrow \delta^*$ ($^1A_{2u} \leftarrow {}^1A_{1g}$)
	2.29	
	2.76	

[a] Electronic spectra were recorded using a Cary 14 spectrometer. [b] All spectra in this table were obtained from diethyl ether solutions of the complexes in 1-cm cells. Diethyl ether was used as a reference. [c] Because of the difficulty of accurately measuring extinction coefficients on these extremely air-sensitive complexes, only upper limits of ϵ have been obtained for $\delta \rightarrow \delta^*$. These are: chromium ~700 M^{-1} cm^{-1}; molybdenum and rhenium ~1500 M^{-1} cm^{-1}.

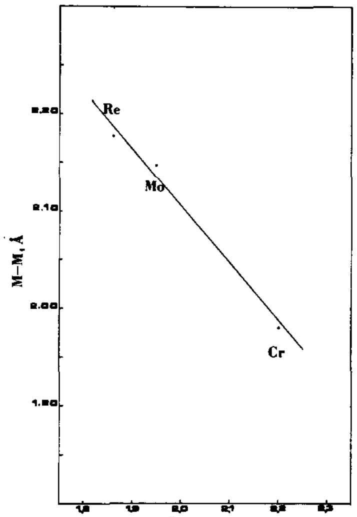

Figure 2. Plot of metal–metal bond distance (Å) vs. energy (μm^{-1}) of the $\delta \rightarrow \delta^*$ ($^1A_{2u} \leftarrow {}^1A_{1g}$) transition for the octamethyldimetalates of chromium(II), molybdenum(II), and rhenium(III).

the structural data on $[Li(THF)]_4Mo_2(CH_3)_8$[10] and $(NH_4)_4Mo_2Cl_8 \cdot NH_4Cl \cdot H_2O$.[15] The M–M bond lengths for these complexes are 2.148 (2) and 2.150 (5) Å, respectively. Furthermore, when benzene solutions of $Mo_2(CH_3)_8{}^{4-}$ are irradiated with 488.0 or 514.5 nm Ar^+ laser excitation, an intense Raman band is observed at 336 ± 1 cm^{-1} which we assign as the ν_1 (MoMo)a_{1g} stretching frequency. This band is to be compared with the similarly assigned band[7] found in the ammonium salt of $mo_2Cl_8{}^{4-}$ at 338 cm^{-1}.

As noted in Table II, two other regions of absorption are apparent in the solution spectrum of $Mo_2(CH_3)_8{}^{4-}$. Since both of these bands are weaker than $\delta \rightarrow \delta^*$, it is unlikely that they are dipole allowed transitions. However, a complete assignment of the spectra of $Mo_2(CH_3)_8{}^{n-}$ species must await further work.

For $Re_2(CH_3)_8{}^{2-}$ we assign the lowest energy band at 1.86 μm^{-1} as $\delta \rightarrow \delta^*$. This band is responsible for the red-purple color of the complex. The position of this transition represents a blue shift of >4000 cm^{-1} relative to the lowest energy band in $Re_2Cl_8{}^{2-}$. Such a significant shift in energy must reflect a large increase in δ overlap. This shift is consistent with the greater M–L π interaction calculated for $Re_2Cl_8{}^{2-}$ although it is much larger than expected. The structures of $[(C_4H_9)_4N]_2Re_2Cl_8$[5] and $[Li(Et_2O)]_2Re_2(CH_3)_8$[12] indicate significant M–M bond differences, however. In the latter complex the metal–metal bond distance (2.178 (1) Å) is almost 0.05 Å shorter than its chloro analogue (2.224 (1) Å), indicating an increase in *both* M–M π and δ bonding. Two other bands of lower intensity than $\delta \rightarrow \delta^*$ were uncovered in the spectrum of $Re_2(CH_3)_8{}^{2-}$ (Table II) and are presumably dipole forbidden.[16]

In the spectrum of octamethyldichromate(II) ion, only one band was observed in the visible region of the spectrum at 2.20 μm^{-1}. It is responsible for the golden yellow color of the compound. It is logical to assign this band to $\delta \rightarrow \delta^*$ inasmuch as the M–M separation is only 1.980 (5) Å,[11] one of the shortest metal-to-metal bonds known. Such a close approach of metal atoms apparently results in favorable δ overlap, even exceeding that found in the molybdenum analogue.

In Figure 2 we have plotted M–M bond length vs. the position of the $\delta \rightarrow \delta^*$ transition. This plot suggests a reasonable correlation. As the metal–metal separation decreases, the overlap of metal orbitals presumably increases, resulting in the shift of $\delta \rightarrow \delta^*$. The spectrum[13] of the recently discovered $W_2(CH_3)_8{}^{4-}$ anion strengthens this correlation. Raman spectral measurements of the methyl dimers are in progress.

Acknowledgments. The support of the National Science Foundation, MPS74-05998, is gratefully recognized for this study. We thank Professors Zerner, Wilkinson, and Cotton for sending us information about their studies prior to publication. We acknowledge with thanks the funding from the General Electric Foundation used for the purchase of the inert atmosphere equipment used in this study.

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(16) It is puzzling that the band near 2.29 μm^{-1} in the $Re_2(CH_3)_8{}^{2-}$ is only a factor of two less intense than the band at 1.95 μm^{-1}. We examined the possibility that oxidation was involved but have consistently reproduced the spectrum presented. Introduction of oxygen produces no new band in this region but causes bands to develop near 300 and 600 nm. Recrystallization produced no change in the character of the spectrum. Further spectroscopic studies clearly are desirable; however, the extreme air sensitivity of these materials makes such work exceedingly difficult.

A. P. Sattelberger, J. P. Fackler*
Department of Chemistry, Case Western Reserve University
Cleveland, Ohio 44106

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Crystallographic Proof of the Stability of a Quadruple Bond between Tungsten Atoms

Sir:

Since the recognition that a quadruple bond exists in the $Re_2Cl_8{}^{2-}$ ion,[1,2] such bonds have been found in other dirhenium(III) species,[2,3-6] in many dimolybdenum(II) compounds,[2,7-12] including the anions $M_2(CH_3)_8{}^{n-}$ for Cr,[2,13]

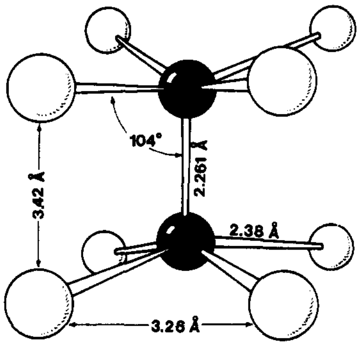

Figure 1. A model for the $W_2X_8{}^{4-}$ ion refined with an appropriate average of CH_3 and Cl ligands.

Mo,[2,13] and Re.[6,13] The only report of quadruple W-W bonds concerns $W_2(O_2CR)_4$ (R = C_6H_5, p-$CH_3C_6H_4$, C_6F_5, C_3H_7, C_3F_7) compounds, none of which has been characterized crystallographically.[14] A scattered wave-Xα calculation[15] for $W_2Cl_8{}^{4-}$ indicates stability but attempts to isolate a salt have so far failed. In efforts to prepare compounds[16,17] containing $W_2(CH_3)_8{}^{4-}$ we have obtained and characterized crystallographically substances of composition $Li_4W_2(CH_3)_{8-x}Cl_x \cdot 4THF$.

Our method of preparation[18] consists in adding CH_3Li (prepared in Et_2O from CH_3Cl and Li) slowly at −78 °C to a slurry of WCl_4 with a mole ratio CH_3Li/WCl_4 of 6. The reaction mixture is warmed to 0 °C for 10 min and then cooled to and maintained at −20 °C for 1 h and filtered and the filtrate cooled to −78 °C, yielding red-purple, pyrophoric crystals, thermally stable only to about −20 °C. When this product is redissolved in the minimum volume of Et_2O at −20 °C, 1 ml of carefully purified THF added, and the solution cooled to −78 °C, there is nearly quantitative recovery of red crystals. A toluene-d_8 solution of this product at −40 °C gave a carbon-13 NMR spectrum with a sharp quartet at 2.16 ppm, besides the signals for toluene-d_8 and THF.

We first assumed that the red-purple and red crystals were $Li_4W_2Me_8 \cdot 4Et_2O$ and $Li_4W_2Me_8 \cdot 4THF$, respectively. Many crystals of the red-purple product were examined on a diffractometer, having been maintained throughout at temperatures between −78 and −20 °C. Despite their excellent visual appearance they diffracted very poorly. Attention was then turned to the red crystals, which also looked good and seemed perhaps slightly more stable thermally.

A red crystal was sealed in a capillary containing mineral oil. This transfer was conducted at −20 °C and the capillary immediately placed on a Syntex four-circle, automatic diffractometer equipped with a cryostat set to maintain the crystal at −55 °C. Preliminary examination showed the crystal to be of high quality, and intensity data were recorded for 657 unique reflections in the range $0 < 2\theta < 47°$, of which 436 with $I > 3\sigma(I)$, corrected for Lorentz and polarization effects, were used to solve the structure.[19] Refinement in the space group $P42_12$[20] assuming the formula $Li_4W_2Me_8 \cdot 4THF$ converged to $R_1 = 0.052$ but the thermal parameters for the assumed methyl carbon atoms suggested the presence of a random mixture of CH_3 and Cl ligands in roughly equal numbers. Chemical analyses were then carried out and they confirmed this.[21]

Refinement was then undertaken using a model in which CH_3 and Cl ligands were assigned fractional weights and assumed to occupy each ligand site simultaneously. The occupation numbers were constrained as ρ for CH_3 and 1-ρ for Cl. The initial values of W-C, W-Cl, and ρ were set at 2.10 Å, 2.40 Å, and 0.60. Full-matrix least-squares refinement in which tungsten atoms were assigned anisotropic thermal vibration parameters while all others were treated isotropically converged with unweighted (R_1) and weighted (R_2) discrepancy indices of 0.041 and 0.045. The ratio of data to parameters is 8.9 and structure factors calculated from the refined structure satisfactorily confirmed the observation[20] that for $hk0$, $h + k = 2n + 1$, the intensities are very weak though this is not an extinction condition for $P42_12$.

The $W_2X_8{}^{4-}$ unit is required crystallographically to have C_4 symmetry. Thus the ligands are symmetry related by a fourfold axis within each WX_4 moiety, but the two WX_4 moieties are crystallographically independent. In fact, the $W_2X_8{}^{4-}$ unit approximates very closely to D_{4h} symmetry. When the ligands were modeled as single atoms having suitably averaged scattering factors, the two WX_4 moieties differed from a perfectly eclipsed rotational relationship about the common C_4 axis by only about 0.6°. It is this model that is represented in Figure 1, with parameter values being averages according to D_{4h} symmetry.

For the disordered model in which C and Cl atoms are permitted to have individual position, thermal, and occupancy parameters, the results, again averaged, are: W-W = 2.263 (2) Å, W-Cl = 2.48 (4) Å, W-C = 2.20 (9) Å, ∠W-W-Cl = 102.5 (8)°, ∠W-W-C = 109 (2)°, and ρ = 0.59 (3). There is no crystallographic indication that a disordering of $W_2Cl_8{}^{4-}$ and $W_2Me_8{}^{4-}$ units rather than internal disordering of Cl and CH_3 ligands within each $W_2X_8{}^{4-}$ unit is to be preferred; since such an array of the two limiting species appears chemically quite unlikely we reject that model in favor of the internally disordered one.

The W-C and W-Cl distances obtained from refinement of the internally disordered model are plausible when compared to Mo-C (2.29 Å), Re-C (2.20 Å), and Mo-Cl (2.45 Å) in previous $M_2X_8{}^{n-}$ structures.[2,6]

We do not consider it a misfortune not to have obtained the structure of pure $Li_4W_2Me_8 \cdot 4THF$. On the contrary, we feel confident that the structure of the octamethyl compound will eventually be determined, while the presence of a mixture of CH_3 and Cl ligands suggests in a positive way that compounds of the $W_2Cl_8{}^{4-}$ ion may also be isolable. In any event, the question of whether a quadruple bond between tungsten atoms is capable of existence has now been conclusively answered in the affirmative.[22]

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(16) We have been kept fully informed of work by K. Mertis and G. Wilkinson, Imperial College, London, directed toward the same end. They have isolated, inter alia, a substance they believe to be $Li_4W_2Me_8 \cdot 4Et_2O$.
(17) We have also been successful in isolating pure $W_2Me_8{}^{4-}$ compounds. By increasing the CH_3Li/WCl_4 mole ratio to 7.5 and following reaction procedures similar to those outlined here, the complexes $Li_4W_2(CH_3)_8 \cdot 4Et_2O$ and $Li_4W_2(CH_3)_8 \cdot 4THF$ have been isolated; these complexes are similar in appearance and properties to the $W_2(CH_3)_{8-x}(Cl)_x{}^{4-}$ complexes. Their formulation as pure $W_2(CH_3)_8{}^{4-}$ species is supported by the demonstrated absence of Cl^- and by CH_3/W mole ratios of 4/1. The electronic spectrum of the $W_2(CH_3)_8{}^{4-}$ ion in a methyltetrahydrofuran glass at 77 K is qualita-

tively similar to that[13] of the $Mo(CH_3)_8{}^{4-}$; the band corresponding to the δ–δ^* transition occurs at 16.9 cm^{-1}, and this figure together with a W–W distance of 2.26 Å gives a point very close to Sattelberger and Fackler's[13] line.

(18) We have also confirmed that WCl_5, the reagent used by Mertls and Wilkinson,[16] is a suitable source of tungsten. When we used a CH_3Li/WCl_5 mole ratio of 8/1 and maintained the temperature $< -20°$, we obtained $W_2Me_{8-x}4_x{}^{4-}$ compounds; Mertis and Wilkinson use an 8/1 ratio.

(19) Setting angles for 15 strong reflections ($2\theta \sim 20°$) were used to calculate the lattice parameters (Mo $K\alpha$, λ 0.710 73 Å; −55 (3) °C) $a = 11.403$ (8), and $c = 11.969$ (5) Å of the tetragonal cell. Based on a crystal composition of $Li_4W_2Cl_4(CH_3)_4 \cdot 4C_4H_8O$, a density of 1.89 g cm^{-3} is calculated when $Z = 2$.

Intensity data were collected using monochromatized Mo $K\alpha$ radiation and θ–2θ scans of variable rate (3 to 24°/min depending on reflection intensity) and range. The range was varied to account for spectral dispersion, and background measurements were made at both limits of each scan. An initial examination of the data established the Laue group to be 4/*mmm*. Because the crystal was under mineral oil we were unable to measure the crystal faces accurately and no absorption correction was applied. Three standard reflections monitored throughout the diffraction experiment were stable.

(20) The systematic absence of significant intensity for reflections of the type $hk0$, $h + k = 2n + 1$, suggested (uniquely) the space group, $P4/nmm$. The Patterson map allowed placement of two independent tungsten atoms in special positions 2(c); refinement of their positions with assigned isotropic thermal parameters of 2.1 $Å^2$ led to $R_1 = 0.22$. So long as space group $P4/nmm$ was used, difference Fourier maps were uninterpretable. Nevertheless, by employing a disordered model which turned out to a superposition of half-weighted mirror-images of the correct idealized $W_2X_8{}^{4-}$ unit, it was possible to obtain by refinement a model for which $R_1 = 0.10$. These difficulties vanished when one image was placed in the correct space group, $P42_12$, and a Li atom and a tetrahydrofuran molecule were found in a difference map.

(21) Thermal instability precludes combustion analysis. Determinations of CH_3 (by CH_4 evolution), Cl (as AgCl), and W (as WO_3), performed on the crystalline substances, satisfied the relation $(CH_3 + Cl)/W = 4$ with the CH_3/Cl ratio varying from one preparation to another within the limits 2.8 to 4.6.

(22) This work was supported financially by the National Science Foundation under Grant No. GP33142X. We thank Drs. John Kolb and Larry W. Shive for their interest and assistance in the earlier stages of the work.

Douglas M. Collins, F. Albert Cotton,* Stephen Koch
Michelle Millar, Carlos A. Murillo
Department of Chemistry, Texas A&M University
College Station, Texas 77843

Received October 4, 1976

μ-Allene-bis(cyclopentadienyl)tetracarbonyldimolybdenum; a Bridging Allene Ligand

Sir:

There is a current belief that transition metal cluster chemistry could lead to a new era in homogeneous hydrocarbon catalysis.[1] The reactivity of metal-to-metal bonds and the synthesis of new cluster compounds are clearly two topics requiring study if such aspirations are to be realized. Bimetallic compounds containing metal-to-metal multiple bonds[2] will surely occupy a unique position in this field of chemistry. They are the smallest examples of unsaturated metal cluster compounds and provide building blocks for the systematic synthesis of new cluster compounds. We have previously shown that an extensive coordination chemistry surrounds metal-to-metal triple bonds in the chemistry of molybdenum[3-7] and tungsten.[8-16] Others have shown[17] that acetylenes, but *not* olefins,[18] add across metal-to-metal triple bonds, although the same products can be obtained directly by reaction of the acetylenes with $Cp_2Mo_2(CO)_6$ without isolating the presumed $Cp_2Mo_2(CO)_4$ intermediates.[19] The structure of a representative product, $Cp_2Mo_2(CO)_4(\mu$-EtCCEt), has been determined; the length of the Mo–Mo single bond is 2.977 (1) Å.[20] We now report the preparation and characterization of $Cp_2Mo_2(CO)_4$(allene) from the reaction between allene and the M–M triple-bonded compound $Cp_2Mo_2(CO)_4$,[17] thus providing the first instance in which an allene molecule, acting as a four-electron donor, bridges two directly bonded metal atoms.[21]

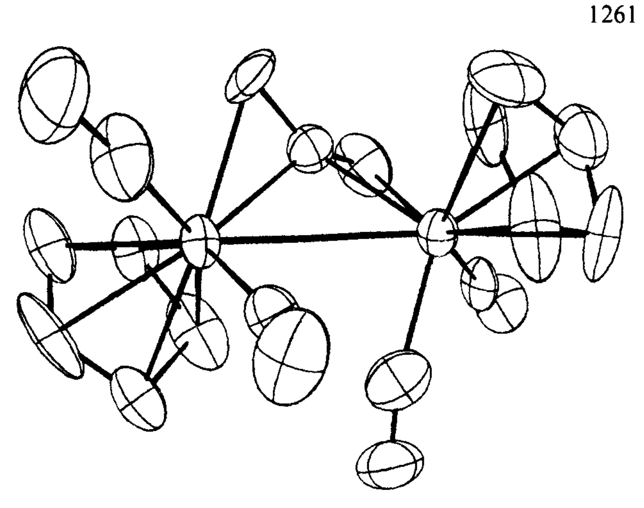

Figure 1. The molecular structure of $Cp_2Mo_2(CO)_4$(allene). Atoms are represented by thermal ellipsoids enclosing 50% of their electron density.

Addition of allene (1 equiv) to hydrocarbon solutions of $Cp_2Mo_2(CO)_4$ leads to a deep red solution and the formation (~10% yield) of a microcrystalline orange precipitate.[23] Deep red crystals of $Cp_2Mo_2(CO)_4$(allene) were the only product isolated from the solution. Mass spectrum (120 °C): parent ion, $Cp_2Mo_2(CO)_4(C_3H_4)^+$, strong; $Cp_2Mo_2(CO)_2C_3H_4{}^+$; $Cp_2Mo_2C_3H_4{}^+$. IR (cm^{-1}): 1995, 1960, 1915, 1860, 1830; these bands must be due to ν_{CO} and $\nu_{C=C}$ but unequivocal assignment must await study of ^{13}C-labeled samples.

The crystal structure[24] was determined giving the molecular configuration shown in Figure 1. Some important dimensions are: Mo(1)–Mo(2), 3.117 (1) Å; Mo(1)–C(15), 2.23 (2) Å; Mo(1)–C(16), 2.11 (1) Å; Mo(2)–C(17), 2.23 (2) Å; Mo(2)–C(16), 2.13 (1) Å; C(15)–C(16), 1.44 (2) Å; C(16)–C(17), 1.41 (1) Å; ∠C(15)–C(16)–C(17), 146 (1)°, where C(15)–C(16)–(17) constitute the skeleton of the allene ligand. The M-to-C(terminal) distances are ca. 0.10 Å longer than M-to-C(central) distances as in other allene complexes.[22] The entire Mo(1)–Mo(2)–C(15)–C(16)–C(17) configuration is consistent with each of the two virtually orthogonal ethylenic portions of C_3H_4 interacting with one metal atom. The entire molecule has approximately C_2 symmetry, with the C_2 axis passing through C(16) and the mid-point of the Mo(1)–Mo(2) bond. The longer Mo–Mo bond here compared to $Cp_2Mo_2(CO)_4$(EtCCEt) is consistent with the use of a three-carbon chain rather than the two-carbon acetylene to bridge the metal atoms. The Mo–C–O chains are all a little bent, presumably due to intramolecular crowding (angles of 164° to 176°), but the longer Mo–Mo distance mitigates crowding and there is no semibridging CO ligand as in $Cp_2Mo_2(CO)_4$(EtCCEt).

NMR spectra (benzene-d_6, 40 °C, δ values in parts per million downfield from Me_4Si) are consistent with the same structure in solution. These spectra are unchanged by raising the temperature to 80 °C or by addition of allene to the solutions. The ^1H spectrum: 4.54, singlet, 10 H; 3.67, triplet, 2 H; 2.52, triplet, 2 H. The triplets are not of 1:2:1 relative intensities and can be assigned to a "deceptively simple" AA′BB′ multiplet,[25] consistent with the C_2 molecular symmetry. The $^{13}C[^1H]$ spectrum consists of five singlets: 237, 2 C, and 233, 2 C (carbonyl carbon atoms); 196, 1 C (central allenic C); 93, 10 C (Cp rings); 36, 2 C (terminal allenic C's). Without ^1H-decoupling the terminal allenic signal became four equally intense lines, assignable to the X portion of an $X_2AA'BB'$ spectrum, with $J_{AX} \approx J_{BX} \approx 160$ Hz, indicative[26] of approximately sp^2 hybridization at C.

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