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REACTIONS OF METAL-METAL MULTIPLE BONDS. 8. FORMING MO-MO QUADR--ETC(U)

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REACTIONS OF METAL-METAL MULTIPLE BONDS. 8.

FORMING Mo-Mo QUADRUPLE BONDS BY

REDUCTIVE ELIMINATION (ALKYL GROUP DISPROPORTIONATION)

IN THE REACTIONS OF $1,2\text{-Mo}_2\text{R}_2(\text{NMe}_2)_4$ COMPOUNDS (M.M.)

WITH CARBON DIOXIDE AND 1,3-DIARYLTRIAZENES

by

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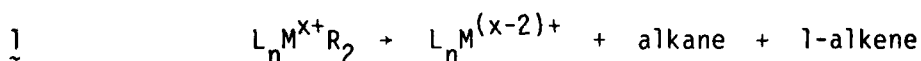
the elimination of the hydrocarbons is intramolecular and that the β -hydrogen atom of one ethyl group is transferred to the α -carbon atom of the other. These reactions proceed through intermediates of the form $\text{Mo}_2\text{R}_2(\text{NMe}_2)_2\text{L}_2$, where $\text{L} = \text{O}_2\text{CNMe}_2$ or ArN_3Ar . The compound $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$, where $\text{C}_7\text{H}_8 = \text{p-tolyl}$, has been isolated and structurally characterized: space group P21/a , $a = 16.503(4) \text{ \AA}$, $b = 23.116(7) \text{ \AA}$, $c = 11.318(3) \text{ \AA}$, $\beta = 88.15(1)^\circ$ and $Z = 4$. The molecule has virtual, but not crystallographically imposed C_2 symmetry. The $\text{Mo}\equiv\text{Mo}$ bond, $2.171(1) \text{ \AA}$, is bridged by a cis-pair of triazenido ligands which afford sufficient flexibility to allow a non-eclipsed geometry. Each molybdenum atom is coordinated to three nitrogen atoms and a carbon atom which lie roughly in a plane. Pertinent averaged bond distances are $\text{Mo}-\text{C} = 2.21(1) \text{ \AA}$, $\text{Mo}-\text{N}$ (dimethylamide) = $1.95(1) \text{ \AA}$ and $\text{Mo}-\text{N}$ (triazenido) = $2.17(1)$ and $2.25(1) \text{ \AA}$ with the longer bond being trans to the $\text{Mo}-\text{NMe}_2$ group. Plausible mechanisms for the reductive elimination sequence are presented and discussed in the light of related reactions.

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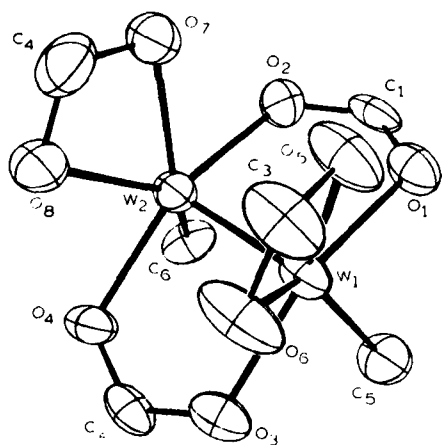
Introduction

The preparation² of an extensive series of 1,2-M₂R₂(NMe₂)₄ compounds (M≡M), where M = Mo and W, and R = Me, Et, i-Pr, n-Bu, sec-Bu, tert-Bu, CH₂CMe₃ and CH₂SiMe₃, provides the opportunity of studying the reactivity patterns of σ-alkyl groups³ bonded to dimetal centers. Of particular note in the above series are the thermally stable compounds which contain σ-alkyl groups that have β-hydrogen atoms. A common thermal decomposition pathway in mononuclear transition metal complexes containing such ligands involves the so-called β-hydrogen elimination reaction: M-alkyl → M-H + alkene. For dialkylmetal complexes, this is often the step which precedes reductive elimination by carbon-hydrogen bond formation, leading to the overall reaction shown in equation 1 below. This may be called reductive alkyl group disproportionation and has been observed and well studied in the thermal decomposition of di-n-butyl compounds of platinum(II),⁴ cobalt(III)⁵ and titanium(IV).⁶ It seemed reasonable to suppose that reductive alkyl group disproportionation might also occur for 1,2-dialkyldimetal compounds.



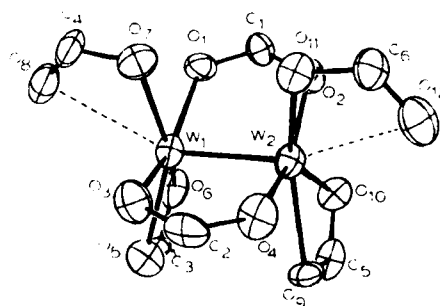
Previously, we suggested that the thermal stability of compounds of the type 1,2-Mo₂(i-Pr)₂(NMe₂)₄ arises from two factors: (i) β-hydrogen elimination is not favored because metal atomic orbitals are not available (or suited) to form C-H-Mo 3 center bonds⁷ and (ii) reductive elimination would generate a coordinatively unsaturated and presumably highly energetic metallic species⁸ (Me₂N)₂Mo≡Mo(NMe₂)₂. Both of these factors are controllable, in principle, by suitable reactions involving the dimethylamido ligands and previous work had shown that W₂Me₂(NEt₂)₄ and W₂(NMe₂)₆ react with carbon

dioxide to give $W_2Me_2(O_2CNEt_2)_4$ and $W_2(O_2CNMe_2)_6$, respectively.⁹ The structures of these two molecules, which are shown in I and II below, led us to believe that the ethyl compound $Mo_2Et_2(O_2CNMe_2)_4$ would be labile to reductive elimination. (1) The carbamato compound $Mo_2(O_2CNMe_2)_4$, formed upon reductive elimination, would be analogous to scores of well characterized $Mo_2(O_2CR)_4$ ($M \equiv M$) compounds.¹⁰ (2) The β -hydrogen elimination step would satisfy the 16-18 electron rule¹¹ and formation of the Mo-H bond along the Mo-Mo axis would be stereochemically analogous to formation of the axial W-O bonds found in $W_2(O_2CNMe_2)_6$. We report here the details of our studies which were prompted by these lines of reasoning. A preliminary report of some aspect of this work has appeared.¹²



Central skeleton of the $W_2Me_2(O_2CNEt_2)_4$ molecule.

I



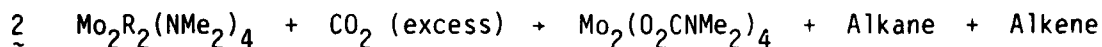
Central skeleton of the $W_2(O_2CNMe_2)_6$ molecule. Dotted lines indicate the long, quasi-axial W-O bonds.

II

Results and Discussion

Reactions with Carbon Dioxide. $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_4$ reacts with CO_2 in hydrocarbon solvents to give a finely divided yellow crystalline solid which is only sparingly soluble in hydrocarbon solvents. The compound shows a molecular ion, $\text{Mo}_2\text{Me}_2(\text{O}_2\text{CNMe}_2)_4^+$, in the mass spectrometer which, together with other spectroscopic and analytical data, lead us to believe it is analogous to the tungsten compound $\text{W}_2\text{Me}_2(\text{O}_2\text{CNEt}_2)_4$, which was structurally characterized and shown to have a metal-to-metal triple bond.⁹

In contrast to the above, the 1,2-dialkyl dimolybdenum compounds $\text{Mo}_2\text{R}_2(\text{NMe}_2)_4$, where the alkyl group contains β -hydrogen atoms react according to equation 2 below.



The compound $\text{Mo}_2(\text{O}_2\text{CNMe}_2)_4$ is formed as a finely divided, yellow, microcrystalline solid which is virtually insoluble in hydrocarbon solvents and only very sparingly soluble in polar solvents, such as pyridine. The ^1H nmr spectrum shows only one resonance and the infrared spectrum shows bands associated with the O_2CNMe_2 ligands.¹³ In the mass spectrometer, the most intense ion was the ion of highest mass, $\text{Mo}_2(\text{O}_2\text{CNMe}_2)_4^+$; there was also an intense doubly charged ion corresponding to $\text{Mo}_2(\text{O}_2\text{CNMe}_2)_4^{2+}$. There is therefore little doubt that $\text{Mo}_2(\text{O}_2\text{CNMe}_2)_4$ is closely related to scores of other well characterized compounds of general formula $\text{Mo}_2(\text{O}_2\text{CR})_4$, which contain metal-to-metal quadruple bonds.¹⁰

Studies of reaction 2 were carried out in sealed nmr tubes and the reaction course was followed by nmr spectroscopy. Within the limits of detection by the nmr method, reaction 2 is stoichiometric: equal molar quantities of alkane and alkene are liberated. At room temperatures, the reaction is

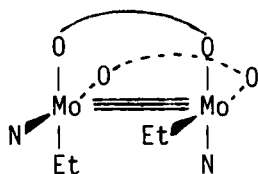
rapid and essentially complete within 30 min. Reactions involving $R = n\text{-Bu}$ gave 1-butene and n-butane (no 2-butenes were detected); for $R = \text{sec-Bu}$, n-butane, 1-butene and a trace of 2-butenes were evolved, while for $R = \text{tert-Bu}$, the eliminated hydrocarbons were iso-butylene and iso-butane. These observations are consistent with an irreversible β -hydrogen atom to metal transfer reaction. This was further supported by the studies of the reactions between CO_2 and each of the labelled ethyl compounds $\text{Mo}_2(\text{CH}_2\text{CD}_3)_2(\text{NMe}_2)_4$ and $\text{Mo}_2(\text{CD}_2\text{CH}_3)_2(\text{NMe}_2)_4$. In both of these reactions, the liberated ethylene was $\text{CH}_2=\text{CD}_2$.

The labelling pattern in the three $\text{C}_2\text{H}_2\text{D}_2$ isomers can be deduced by ^2H nmr spectroscopy. See Figure 1. Each of the isomers of dideuteroethylene show a central three-line pattern with the only apparent distinguishing characteristic being the separation between the outer lines, which is 4.8 Hz for $\text{CH}_2=\text{CD}_2$, 3.3 Hz for cis-CHD=CHD and 2.2 Hz for trans-CHD=CHD.

The ethane liberated in these reactions is either CH_2DCD_3 , when $R = \text{CH}_2\text{CD}_3$ or CHD_2CH_3 , when $R = \text{CD}_2\text{CH}_3$. The ^2H nmr spectra of the evolved ethanes are compared with the spectra obtained for commercially available samples of the specifically labelled ethanes in Figure 2. In a separate experiment, an equimolar mixture of $\text{Mo}_2(\text{CH}_2\text{CH}_3)_2(\text{NMe}_2)_4$ and $\text{Mo}_2(\text{CH}_2\text{CD}_3)_2(\text{NMe}_2)_4$ was allowed to react with CO_2 in an nmr tube. The ^2H nmr spectrum of the liberated ethane was indistinguishable from that shown in Figure 2 for CH_2DCD_3 . We conclude that, within the limits of detection by nmr spectroscopy, the elimination of ethane occurs by an intramolecular process: an intermolecular process would generate $\text{CH}_3\text{CH}_2\text{D}$ and CH_3CD_3 along with CH_2DCD_3 and C_2H_6 .

The reaction between $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_4$ and CO_2 in toluene- d_8 was followed by ^1H nmr spectroscopy at -30°C . At this temperature, the reaction is quite

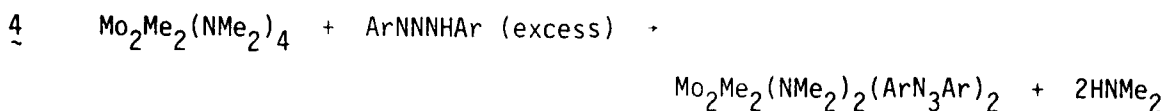
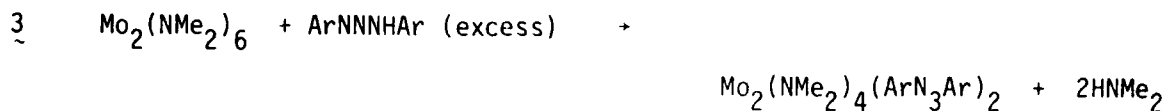
slow, taking several hours to go to completion. Certain significant observations arose from these studies. (1) The ratio of ethylene and ethane remained constant throughout the course of the reaction: ethylene and ethane are formed at apparently the same rate and it is not possible to determine which is eliminated first. (2) No signal, which might have been assigned to a metal-hydride, was detected in the range -20 to +30 ppm. [None would be expected, given (1) above]. (3) The reaction rate varied slightly from sample to sample, even when concentrations of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_4$ and CO_2 were kept virtually constant. This is similar to previous observations of CO_2 insertion reactions involving $\text{W}(\text{NMe}_2)_6$ and $\text{W}_2\text{Me}_2(\text{NEt}_2)_4$, which were shown to require the presence of trace amounts of free amine.¹⁴ (4) In all reactions, the build up in concentration of a reactive intermediate was seen. The ^1H nmr spectrum of this intermediate is shown in Figure 3 and is consistent with that expected for $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{O}_2\text{CNMe}_2)_2$, providing that the molecule has virtual C_2 symmetry. A plausible structure is shown schematically in III below. This proposed structure is similar to that found¹⁵ for $\text{Mo}_2(\text{OBu}^t)_4(\text{O}_2\text{COBu}^t)_2$ and that reported in this paper for $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$, where $\text{C}_7\text{H}_8 = p\text{-tolyl}$.



III

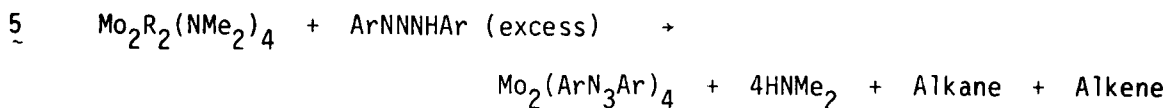
The rate determining step must involve a reaction of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{O}_2\text{CNMe}_2)_2$, but we have not been able to determine the nature of this step. It seemed reasonable to suppose that other reactions, in which dimethylamido ligands were replaced by bidentate ligands, would show similar facility in inducing reductive elimination of alkane and alkene.

Reactions with 1,3-Diaryltriazenes. Previously, we have shown that $\text{Mo}_2(\text{NMe}_2)_6$ and $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_4$ react with 1,3-diaryltriazenes according to equations 3¹⁶ and 4,¹⁷ respectively.



Reactions 3 and 4 appear quantitative and irreversible. Even in the presence of excess triazene, only two dimethylamido groups are substituted. Single crystal X-ray studies revealed that, in both compounds, the triazenido ligands are bidentate: in $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$,¹⁷ they bridge the Mo≡Mo bond, while in $\text{Mo}_2(\text{NMe}_2)_4(\text{C}_6\text{H}_5\text{N}_3\text{C}_6\text{H}_5)_2$,¹⁶ there is an unbridged Mo≡Mo bond.

In contrast to the above, 1,3-diaryltriazenes react with $\text{Mo}_2\text{R}_2(\text{NMe}_2)_4$ compounds, where R = Et and i-Pr, in hydrocarbon solvents according to equation 5.



The compound $\text{Mo}_2(\text{C}_6\text{H}_5\text{N}_3\text{C}_6\text{H}_5)_4$ has been synthesized previously and shown by Cotton and coworkers¹⁸ to have a Mo-to-Mo quadruple bond.

We were unable to obtain satisfactory elemental analyses for our compounds: indeed, analytical data varied in a seemingly erratic manner. Thus, crystals of the compound $\text{Mo}_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_4$, grown from toluene/hexane solution, were studied by X-ray diffraction. This yielded a solution¹⁹ having a structure essentially identical to that reported by Cotton, *et al.*,¹⁸ for the

$\text{Mo}_2(\text{PhN}_3\text{Ph})_4$. The problem associated with obtaining good elemental analytical results probably arises from the partial solvation observed in the crystals. The blades of the aryl groups are packed in such a way as to generate channels and holes. In the crystal which we examined, toluene molecules were located and refined in the channels, but a large hole, ca. 12 Å in diameter, was apparently partially occupied by some molecule of inclusion. Full structural details are given in the supplementary data. The characterization of $\text{Mo}_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_4$ is not pertinent to the thrust of this paper beyond establishing that, in reaction 5, the metal-to-metal bond order changes from three to four.

By the careful addition of 2 equivalents of 1,3-di-p-tolyltriazene to a hydrocarbon solution of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_4$, we have been able to isolate $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ as an orange crystalline compound. In the solid state, it is quite stable and like $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{ArN}_3\text{Ar})_2$ and $\text{Mo}_2(\text{NMe}_2)_4(\text{ArN}_3\text{Ar})_2$ compounds may be exposed to the atmosphere for short periods of time without significant decomposition. [This contrasts with the high reactivity of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_4$, which smolders when exposed to the atmosphere]. On the other hand, $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ slowly decomposes in solution at room temperature and eliminates ethane (no ethylene is evolved). Significantly, the addition of either $\text{C}_7\text{H}_8\text{NNHC}_7\text{H}_8$ or CO_2 causes a more rapid evolution of ethane and ethylene. Addition of $\text{C}_7\text{H}_8\text{NNHC}_7\text{H}_8$ yields $\text{Mo}_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_4$; reaction with CO_2 presumably generates $\text{Mo}_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2(\text{O}_2\text{CNMe}_2)_2$ - but this remains to be established.

Solid State Structure of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$. An ORTEP view of the molecule giving the atom numbering scheme is shown in Figure 4. A view down the Mo-Mo bond is shown in Figure 5. Final atomic coordinates and

thermal parameters are given in Table 1 and 2, respectively. Complete listings of bond distances and bond angles are given in Tables 3 and 4, respectively.

The molecule has virtual, but not crystallographically imposed, C_2 symmetry and is, in this and most other respects, analogous to that observed in $Mo_2Me_2(NMe_2)_2(C_7H_8N_3C_7H_8)_2$. The Mo-C bond distance, 2.21(1) Å (average), is longer than that in the methyl analogue, 2.193(4) Å and that in the parent compound, $Mo_2Et_2(NMe_2)_4$, which is 2.16(1) Å (averaged).² Also, unlike in $Mo_2Et_2(NMe_2)_4$, the ethyl groups are directed over the Mo=Mo bond. As we predicted for this situation, the Mo-BH distances across the Mo=Mo bond are shorter than are those to the molybdenum atoms to which the ethyl group is directly bonded. See Table 5. However, in the present case, no significant C-H---Mo intraction is seen in the ground state. The orientation of the ethyl group over the Mo=Mo moiety can readily be understood in terms of steric considerations. The tolyl groups effectively block the axial positioning of the methyl groups and direct them inwards. The obtuse Mo-C-C angles = 126.1(5) and 125.7(5)°, which may be compared with Mo-C-C = 112.0(3)° and 113.0(3)° found in $Mo_2Et_2(NMe_2)_4$, reflect the internal crowding of the molecule. This may also be the cause of the lengthening of the Mo-C (ethyl) bond distances.²⁰

Concluding Remarks. Addition of either CO_2 or 1,3-diaryltriazines to 1,2- $Mo_2R_2(NMe_2)_4$ compounds promotes reductive elimination from the dimetal center by alkyl group disproportionation with a concomittant metal-metal bond order change from three to four when R = an alkyl group having β -hydrogen atoms. Both reactions proceed through intermediates of the form 1,2- Mo_2R_2 -

$(\text{NMe}_2)_2(\text{chelate})_2$. The elimination of alkane and alkene is intramolecular and has a formal analogy with that found in mononuclear chemistry, namely β -hydrogen elimination followed by reductive elimination by carbon-hydrogen bond formation. The detailed pathway leading to this is, however, uncertain. The presence of the dimetal center introduces additional possibilities. For example, the H-Mo bond could be formed across the Mo=Mo bond. This would be akin to a cyclometallation reaction involving a 5-membered ring²¹ and the structure of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ clearly shows the geometric plausibility of this reaction pathway. The observation that excess $\text{C}_7\text{H}_8\text{NNHC}_7\text{H}_8$ promotes a reaction with $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$, but not with $\text{Mo}_2\text{R}_2(\text{NMe}_2)_2$ compounds, where $\text{R} = \text{CH}_3$ or CH_2CMe_3 , could be correlated with a reversible protonation of a Mo-NMe₂ nitrogen lone pair. In the $\text{Mo}_2\text{R}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ compounds, the NMe₂ ligands are clearly strong π -donors to one of the five in-plane molybdenum atomic orbitals (s , p_x , p_y , d_{xy} , $d_{x^2-y^2}$). Upon protonation of the amide lone pair, one of these orbitals would be freed to form a CH---Mo 3-center, 2-electron bond. The structure of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ suggests that the β -hydrogens of the ethyl ligands are poised for such an opportunity. Alternatively, a 1,2- to 1,1-Mo₂Et₂ isomerization reaction, involving an alkyl group transfer across the Mo=Mo bond, may be required, and promoted in the association reaction between 1,2-Mo₂Et₂(NMe₂)₂(C₇H₈N₃C₇H₈)₂ and excess triazine, prior to β -hydrogen elimination. For a 1,1-Mo₂Et₂(NMe₂)₂(C₇H₈N₃C₇H₈)₂ molecule, one molybdenum atom would have one in-plane atomic orbital available for β -hydrogen elimination in the more conventional sense of this terminology (i.e. mononuclear). Within this context, it should be noted that (i) there is a high kinetic barrier to 1,1- and 1,2-isomerization of $\text{Mo}_2(\text{NMe}_2)_2(\text{CH}_2\text{SiMe}_3)_4$ compounds²² and (ii) Bergman, *et*

al.,²³ have proposed, with good experimental evidence, that reductive elimination of acetone from $1,2\text{-Cp}_2\text{Co}_2\text{Me}_2(\text{CO})_2$ proceeds via an initial methyl group transfer from one cobalt atom to the other. Finally, it should be recognized that it is not necessary to invoke the formation of an intermediate metal-hydride in these reactions. The β -hydrogen atom of an ethyl ligand could be transferred to the α -carbon of the other ethyl ligand through the agency of one of the nitrogen atoms, which could be either a triazenido or dimethyl-amido ligand. In a formal sense this would have analogy with E_2 reactions of substituted alkanes. These, and other possibilities, provide us with the stimulus for further studies of reductive eliminations of hydrocarbons from dinuclear metal centers.

Experimental Section

General procedures, including the preparations of 1,2-Mo₂R₂(NMe₂)₄ compounds, have been described previously.² Note the use of dry and oxygen-free solvents and atmospheres.

¹H and ²H nmr spectra were recorded on Varian HR220 MHz spectrometer equipped with a variable temperature probe. Infrared spectra were recorded on a PE-283 spectrophotometer. Mass spectra were obtained on a Varian MS-902 by the method of direct insertion, courtesy of Mr. Peter Cook, Queen Mary College, London.

Preparation of Mo₂(O₂CNMe₂)₄. To a 25 mL flask equipped with a stir-bar and N₂ inlet, was added 0.100 g (0.23 mmoles) of Mo₂Et₂(NMe₂)₄ in 15 mL of toluene. The flask was attached to a calibrated manifold, evacuated at liquid N₂ temperature, and 6 equivalents of CO₂ were added. As the flask gradually warmed to room temperature, the color changed from a golden yellow to orange with the precipitation of a pale yellow powder. The solution above the powder was virtually colorless at the completion of the reaction. Infrared spectra of the pale yellow powder using Nujol mull and CsI plates gave a strong absorption at 1560 cm⁻¹ (ν NCO₂ of bridging carbamate). The yellow powder was extremely insoluble in most solvents; however, a sufficient quantity was soluble in pyridine-d⁵ to record its nmr spectrum, which showed a singlet at 2.93 ppm relative to TMS corresponding to O₂CNMe₂. Elemental analyses: Found (calculated) - C, 33.55 (33.79); H, 7.89 (8.04); N, 12.99 (13.15).

Preparation of Mo₂(C₇H₈N₃C₇H₈)₄(toluene)_x. A 25 mL round-bottomed flask was charged with 0.25 g (0.58 mmoles) of Mo₂Et₂(NMe₂)₄ and 0.78 g (3.5 mmoles) of di-p-tolyliriazene. Approximately 15 mL of toluene were added at

room temperature in a Vacuum Atmospheres Dri Lab. The color was intensely red-orange. Hexanes (5 mL) were added to the flask before placing it in the cool storage refrigerator of the Dri Lab at -35°C . Dark orange crystals of $\text{Mo}_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_4$ were prepared in this manner. Satisfactory analysis could not be obtained for this complex as was the case for many of the p-tolyltriazene derivatives of analogous complexes.^{16,17} In all respects, the complex showed properties similar to the unsubstituted aromatic derivative prepared by Cotton, et al.¹⁸

Analyses of Gases Liberated. The gases liberated upon reaction of CO_2 with $\text{Mo}_2(\text{C}_2\text{H}_5)_2(\text{NMe}_2)_4$, $\text{Mo}_2(\text{n-C}_4\text{H}_9)_2(\text{NMe}_2)_4$, $\text{Mo}_2(\text{sec-C}_4\text{H}_9)_2(\text{NMe}_2)_4$, $\text{Mo}_2(\text{i-C}_3\text{H}_7)_2(\text{NMe}_2)_4$, and $\text{Mo}_2(\text{t-C}_4\text{H}_9)_2(\text{NMe}_2)_4$ in toluene- d_8 were all compared with authentic samples of the respective alkenes and alkanes by nmr spectroscopy.

The deuterated gases $\text{CD}_3\text{CH}_2\text{D}$, $1,1\text{-CD}_2\text{CH}_2$, and $\text{CH}_3\text{CD}_2\text{H}$ were purchased from Merck, Sharp, and Dohme and compared identically with those generated in the reactions given in Figure 2 at 33.77 MHz in benzene. Calculations using ^1H - ^1H coupling constants given in R.M. Lynden-Bell and N. Sheppard, Proc. R. Soc. A., 269, 1385 (1965) for ethylene and a comparison of the magnetogyric ratios $\gamma(^1\text{H}) = 2.675 \times 10^4$ radians/(s·G), $\gamma_{^2\text{H}} = 4.107 \times 10^3$ radians/(s·G) allowed the simulation of the ^2H nmr at 33.77 MHz of the various dideuteroethylenes shown in Figure 1. The calculated splittings were obtained by using the program entitled NMRCAL, a part of the Nicolet 80 computer system. Having purchased the various dideuteroethylenes, the line shapes and splittings observed for the commercial samples were found to be identical to those predicted by simulation.

Synthesis of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$. In a 50 mL round-bottomed flask were placed 0.50 g (1.17 mmol) of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_4$ and 0.53 g (2.35 mmol) of p-tolyltriazene. To this flask 30 mL of toluene were added in the Dri Lab. The color of the solution was intensely red. After 3 days in the refrigerator of the Dri Lab at -35°C , red crystals precipitated. Addition of 10 mL of hexane to the reaction flask induced crystallization. A crystal of appropriate dimensions was chosen for X-ray analysis.

X-Ray Structural Determination of $\text{Mo}_2(\text{NMe}_2)_2\text{Et}_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$. A crystal of dimensions 0.20 x 0.20 x 0.40 mm was mounted in a nitrogen-filled glove-bag and transferred to the liquid-nitrogen boil-off system of the diffractometer.

The cell dimensions obtained from 46 reflections at -175°C with $\text{MoK}\alpha$ ($\lambda = 0.71069 \text{ \AA}$) were $a = 16.503(4) \text{ \AA}$, $b = 23.116(7) \text{ \AA}$, $c = 11.318(3) \text{ \AA}$, $\beta = 88.15(1)^\circ$, $V = 4315.6(5) \text{ \AA}^3$, $Z = 4$, $d_{\text{calcd}} = 1.353 \text{ g cm}^{-3}$ and space group $P21/a$.

A total number of 6970 reflections were collected using standard moving-crystal, moving-detector techniques with the following values: scan speed = 4.0 deg min^{-1} , scan width = $1.8 + \text{dispersion}$, single background time at extremes of scan = 5 sec, aperture size = $3.0 \times 4.0 \text{ mm}$. Of the 6970 reflections collected, the number with unique amplitudes was 5667 and the number with $F > 2.33 \text{ sigma}(F)$ was 4622. The limits of data collection were $6^\circ < 2\theta < 45^\circ$.

The structure was solved using a combination of direct methods and Fourier techniques. A molecule of solvent, toluene, was located. All non-hydrogen atoms were refined anisotropically. All hydrogens were located in

the difference map, but were not refined due to space limitations in the least-squares program. The final residuals were $R(F) = 0.057$ and $R_w(F) = 0.0795$. The goodness of fit for the last cycle, 1.795 and the maximum delta/sigma, 0.01.

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Table 1. Fractional Coordinates for the $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ Molecule.

Note 1: The isotropic thermal parameter listed for those atoms refined anisotropically are the isotropic equivalent.

Note 2: Number in parentheses in this and all following tables refer to the error in the least significant digits.

Atom	$10^4 x$	$10^4 y$	$10^4 z$	$10 B_{iso}$
Mo(1)	4878.8(4)	2293.1(3)	8199(1)	14
Mo(2)	5997.0(4)	2268.3(3)	9156(1)	14
N(3)	4518(4)	1480(3)	9174(5)	15
N(4)	4876(4)	1306(3)	10097(5)	17
N(5)	5510(3)	1619(2)	10368(5)	14
N(6)	6281(3)	1435(3)	8195(5)	15
N(7)	5909(3)	1278(3)	7267(5)	15
N(8)	5306(3)	1622(3)	7006(5)	15
N(9)	4848(3)	3014(3)	7310(5)	16
C(10)	5389(5)	3512(3)	7144(7)	23
C(11)	4063(4)	3132(3)	6769(7)	22
C(12)	3959(4)	2579(3)	9529(6)	20
C(13)	3990(5)	2485(4)	10891(7)	25
N(14)	6089(3)	3001(3)	9998(5)	18
C(15)	5582(5)	3523(3)	10109(7)	23
C(16)	6894(5)	3108(4)	10505(7)	24
C(17)	6941(4)	2516(4)	7807(6)	19
C(18)	6902(5)	2406(4)	6469(7)	29
C(19)	3826(4)	1141(3)	8886(6)	15
C(20)	3400(4)	1312(3)	7913(7)	20
C(21)	2714(5)	992(3)	7564(7)	24
C(22)	2466(5)	510(3)	8205(7)	23
C(23)	1741(5)	147(4)	7812(8)	29
C(24)	2906(5)	340(4)	9197(7)	27
C(25)	3588(5)	653(3)	9529(7)	22
C(26)	5880(4)	1409(3)	11391(6)	18
C(27)	5872(4)	828(3)	11714(7)	22
C(28)	6243(5)	653(4)	12750(7)	27
C(29)	6649(5)	1046(4)	13468(7)	28
C(30)	7027(5)	840(4)	14583(7)	34
C(31)	6665(5)	1619(4)	13130(7)	30
C(32)	6294(5)	1811(4)	12084(7)	25
C(33)	6974(4)	1086(3)	8417(6)	16
C(34)	7456(5)	1245(3)	9353(7)	21
C(35)	8185(5)	947(3)	9580(7)	20
C(36)	7456(5)	1245(3)	9353(7)	21
C(35)	8185(5)	947(3)	9580(7)	20
C(36)	8417(5)	493(3)	8864(7)	22
C(37)	9208(5)	175(4)	9082(8)	28
C(38)	7911(5)	315(4)	7949(7)	25
C(39)	7193(5)	607(3)	7731(7)	21
C(40)	4911(4)	1435(3)	5961(6)	17
C(41)	4659(5)	1860(4)	5177(7)	24
C(42)	4269(5)	1701(4)	4138(7)	28
C(43)	4102(5)	1119(4)	3910(7)	26
C(44)	3692(5)	944(4)	2789(7)	31
C(45)	4350(5)	698(4)	4714(7)	26
C(46)	4748(4)	858(4)	5738(7)	22
C(47)	-753(8)	1307(7)	3265(10)	74
C(48)	85(7)	1215(5)	3654(8)	45
C(49)	625(7)	1672(5)	3635(8)	52
C(50)	1425(8)	1588(8)	3991(9)	78
C(51)	1681(8)	1022(9)	4366(10)	83
C(52)	1108(11)	588(8)	4355(11)	98
C(53)	322(9)	683(5)	4015(9)	65

Table 2. Anisotropic Thermal Parameters for the $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ Molecule. The form of the exponent is: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}kleb^*c^*)]$.

Atom	U11	U22	U33	U12	U13	U23
Mo(1)	15.6(4)	15.7(4)	20.9(4)	0.8(3)	0.4(3)	1.1(3)
Mo(2)	15.4(4)	16.2(4)	21.3(4)	0.2(3)	-0.1(3)	-0.6(3)
N(3)	20(4)	18(4)	20(4)	-1(3)	3(3)	1(3)
N(4)	20(4)	22(4)	22(4)	3(3)	3(3)	-2(3)
N(5)	21(4)	15(3)	18(3)	1(3)	2(3)	2(3)
N(6)	16(3)	19(4)	23(4)	3(3)	1(3)	0(3)
N(7)	21(4)	19(4)	17(3)	-2(3)	0(3)	-0(3)
N(8)	18(3)	19(4)	21(4)	1(3)	-1(3)	-1(3)
N(9)	18(4)	26(4)	17(3)	0(3)	4(3)	5(3)
C(10)	28(5)	14(4)	44(5)	-7(4)	2(4)	0(4)
C(11)	24(4)	30(5)	28(5)	4(4)	-6(4)	6(4)
C(12)	15(4)	37(5)	23(4)	2(4)	7(3)	-3(4)
C(13)	30(5)	39(5)	25(5)	4(4)	4(4)	-0(4)
N(14)	18(4)	22(4)	27(4)	-2(3)	9(3)	-3(3)
C(15)	31(5)	20(5)	36(5)	7(4)	4(4)	-5(4)
C(16)	22(4)	37(5)	32(5)	-11(4)	-4(4)	-1(4)
C(17)	13(4)	39(5)	19(4)	-3(4)	6(3)	6(4)
C(18)	29(5)	50(6)	31(5)	2(4)	6(4)	8(5)
C(19)	19(4)	15(4)	21(4)	3(3)	3(3)	-1(3)
C(20)	11(4)	26(5)	37(5)	1(3)	6(4)	-1(4)
C(21)	23(5)	27(5)	42(5)	6(4)	2(4)	-5(4)
C(22)	27(5)	26(5)	33(5)	3(4)	4(4)	-4(4)
C(23)	26(5)	41(6)	44(6)	-19(4)	-3(4)	-4(4)
C(24)	39(5)	30(5)	32(5)	0(4)	4(4)	-5(4)
C(25)	27(5)	24(5)	32(5)	-3(4)	8(4)	-4(4)
C(26)	20(4)	28(5)	19(4)	3(4)	3(3)	0(4)
C(27)	17(4)	33(5)	35(5)	7(4)	3(4)	9(4)
C(28)	25(5)	49(6)	29(5)	10(4)	11(4)	7(4)
C(29)	23(5)	54(7)	28(5)	9(4)	2(4)	3(5)
C(30)	43(6)	63(7)	24(5)	14(5)	-1(4)	11(5)
C(31)	40(6)	51(7)	23(5)	1(5)	0(4)	3(4)
C(32)	25(5)	41(5)	27(5)	4(4)	4(4)	3(4)
C(33)	17(4)	19(4)	25(4)	-1(3)	1(3)	4(4)
C(34)	30(5)	17(4)	33(5)	-3(4)	1(4)	12(4)
C(35)	24(5)	20(5)	32(5)	3(4)	5(4)	12(4)
C(36)	25(5)	22(5)	36(5)	-0(4)	1(4)	12(4)
C(37)	27(5)	28(5)	53(6)	14(4)	-3(4)	5(4)
C(38)	31(5)	28(5)	35(5)	2(4)	5(4)	7(4)
C(39)	26(5)	27(5)	26(5)	1(4)	3(4)	3(4)
C(40)	11(4)	30(5)	24(4)	-2(3)	3(3)	1(4)
C(41)	29(5)	32(5)	28(4)	-4(4)	3(4)	3(4)
C(42)	40(5)	30(5)	37(5)	-7(4)	-6(4)	-5(4)
C(43)	31(5)	45(6)	22(4)	-3(4)	6(4)	5(4)
C(44)	35(5)	56(6)	25(5)	-8(4)	-6(4)	-8(4)
C(45)	25(5)	40(5)	32(5)	0(4)	3(4)	-15(4)
C(46)	18(4)	32(5)	34(5)	2(4)	-3(4)	-7(4)
C(47)	88(10)	139(13)	55(8)	-30(9)	-7(7)	6(8)
C(48)	77(8)	63(8)	30(6)	-18(7)	7(5)	-3(5)
C(49)	87(9)	84(9)	27(6)	-27(7)	15(6)	-15(5)
C(50)	79(10)	187(18)	28(7)	-26(11)	24(7)	-25(9)
C(51)	68(10)	215(21)	30(7)	29(12)	9(7)	-15(10)
C(52)	143(17)	181(19)	45(8)	57(15)	21(11)	-33(10)
C(53)	147(14)	68(10)	29(6)	13(9)	15(7)	-6(6)

All U's are x10**3

Table 3. Bond Distances, in Angstroms,
for the $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ Molecule.

A	B	Distance
Mo(1)	Mo(2)	2.171(1)
Mo(1)	N(3)	2.251(6)
Mo(1)	N(8)	2.160(6)
Mo(1)	N(9)	1.949(6)
Mo(1)	C(12)	2.205(7)
Mo(2)	N(5)	2.171(6)
Mo(2)	N(6)	2.253(6)
Mo(2)	N(14)	1.952(6)
Mo(2)	C(17)	2.221(7)
N(3)	N(4)	1.281(8)
N(3)	C(19)	1.431(9)
N(4)	N(5)	1.316(8)
N(5)	C(26)	1.412(9)
N(6)	N(7)	1.286(8)
N(6)	C(33)	1.428(9)
N(7)	N(8)	1.314(8)
N(8)	C(40)	1.437(9)
N(9)	C(10)	1.464(9)
N(9)	C(11)	1.476(9)
N(14)	C(15)	1.471(9)
N(14)	C(16)	1.484(9)
C(12)	C(13)	1.559(10)
C(17)	C(18)	1.539(11)
C(19)	C(20)	1.384(10)
C(19)	C(25)	1.393(10)
C(20)	C(21)	1.420(11)
C(21)	C(22)	1.384(11)
C(22)	C(23)	1.540(11)
C(22)	C(24)	1.411(11)
C(24)	C(25)	1.400(11)
C(26)	C(27)	1.393(11)
C(26)	C(32)	1.406(11)
C(27)	C(28)	1.400(11)
C(28)	C(29)	1.403(12)
C(29)	C(30)	1.502(11)
C(29)	C(31)	1.380(12)
C(31)	C(32)	1.421(11)
C(33)	C(34)	1.395(10)
C(33)	C(39)	1.394(10)
C(34)	C(35)	1.417(10)
C(35)	C(36)	1.372(11)
C(36)	C(37)	1.526(10)
C(36)	C(38)	1.413(11)
C(38)	C(39)	1.391(11)
C(40)	C(41)	1.397(10)
C(40)	C(46)	1.384(11)
C(41)	C(42)	1.407(11)

Table 3. Continued.

A	B	Distance
C(42)	C(43)	1.400(12)
C(43)	C(44)	1.511(11)
C(43)	C(45)	1.402(11)
C(45)	C(46)	1.400(11)
C(47)	C(48)	1.479(16)
C(48)	C(49)	1.382(14)
C(48)	C(53)	1.358(16)
C(49)	C(50)	1.407(16)
C(50)	C(51)	1.445(21)
C(51)	C(52)	1.379(22)
C(52)	C(53)	1.383(19)

Table 4. Bond Angles, in Degrees,
for the $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_5)_2$ Molecule.

A	B	C	Angle
Mo(2)	Mo(1)	N(3)	87.1(2)
Mo(2)	Mo(1)	N(8)	91.6(2)
Mo(2)	Mo(1)	N(9)	108.5(2)
Mo(2)	Mo(1)	C(12)	104.2(2)
N(3)	Mo(1)	N(8)	77.4(2)
N(3)	Mo(1)	N(9)	163.1(2)
N(3)	Mo(1)	C(12)	75.5(3)
N(8)	Mo(1)	N(9)	107.8(2)
N(8)	Mo(1)	C(12)	147.8(3)
N(9)	Mo(1)	C(12)	93.9(3)
Mo(1)	Mo(2)	N(5)	91.7(2)
Mo(1)	Mo(2)	N(6)	87.1(1)
Mo(1)	Mo(2)	N(14)	107.5(2)
Mo(1)	Mo(2)	C(17)	103.8(2)
N(5)	Mo(2)	N(6)	77.4(2)
N(5)	Mo(2)	N(14)	109.0(2)
N(5)	Mo(2)	C(17)	148.2(3)
N(6)	Mo(2)	N(14)	163.5(2)
N(6)	Mo(2)	C(17)	76.0(3)
N(14)	Mo(2)	C(17)	92.8(3)
Mo(1)	N(3)	N(4)	122.7(5)
Mo(1)	N(3)	C(19)	123.1(5)
N(4)	N(3)	C(19)	114.0(6)
N(3)	N(4)	N(5)	114.1(6)
Mo(2)	N(5)	N(4)	120.9(4)
Mo(2)	N(5)	C(26)	126.4(5)
N(4)	N(5)	C(26)	111.8(6)
Mo(2)	N(6)	N(7)	122.6(4)
Mo(2)	N(6)	C(33)	123.6(5)
N(7)	N(6)	C(33)	113.0(6)
N(6)	N(7)	N(8)	113.4(6)
Mo(1)	N(8)	N(7)	121.9(4)
Mo(1)	N(8)	C(40)	125.7(5)
N(7)	N(8)	C(40)	111.7(6)
Mo(1)	N(9)	C(10)	135.4(5)
Mo(1)	N(9)	C(11)	114.2(4)
C(10)	N(9)	C(11)	110.0(6)
Mo(2)	N(14)	C(15)	134.5(5)
Mo(2)	N(14)	C(16)	114.7(5)
C(15)	N(14)	C(16)	110.1(6)
Mo(1)	C(12)	C(13)	126.1(5)
Mo(2)	C(17)	C(18)	125.7(5)
N(3)	C(19)	C(20)	117.0(6)
N(3)	C(19)	C(25)	122.6(7)

Table 4. Continued.

A	B	C	Angle
C(20)	C(19)	C(25)	120.4(7)
C(19)	C(20)	C(21)	120.1(7)
C(20)	C(21)	C(22)	119.9(8)
C(21)	C(22)	C(23)	120.4(7)
C(21)	C(22)	C(24)	119.4(8)
C(23)	C(22)	C(24)	120.1(8)
C(22)	C(24)	C(25)	120.6(8)
C(19)	C(25)	C(24)	119.6(8)
N(5)	C(26)	C(27)	123.0(7)
N(5)	C(26)	C(32)	117.5(7)
C(27)	C(26)	C(32)	119.4(7)
C(26)	C(27)	C(28)	119.9(8)
C(27)	C(28)	C(29)	121.9(8)
C(28)	C(29)	C(30)	120.1(8)
C(28)	C(29)	C(31)	117.8(8)
C(30)	C(29)	C(31)	122.1(8)
C(29)	C(31)	C(32)	121.7(8)
C(26)	C(32)	C(31)	119.3(8)
N(6)	C(33)	C(34)	117.7(7)
N(6)	C(33)	C(39)	123.1(7)
C(34)	C(33)	C(39)	119.2(7)
C(33)	C(34)	C(35)	121.2(8)
C(34)	C(35)	C(36)	119.2(7)
C(35)	C(36)	C(37)	119.8(7)
C(35)	C(36)	C(38)	119.7(7)
C(37)	C(36)	C(38)	120.5(7)
C(36)	C(38)	C(39)	121.0(8)
C(33)	C(39)	C(38)	119.6(7)
N(8)	C(40)	C(41)	117.6(7)
N(8)	C(40)	C(46)	122.4(7)
C(41)	C(40)	C(46)	119.9(7)
C(40)	C(41)	C(42)	120.0(7)
C(41)	C(42)	C(43)	120.3(8)
C(42)	C(43)	C(44)	120.6(8)
C(42)	C(43)	C(45)	118.9(8)
C(44)	C(43)	C(45)	120.5(8)
C(43)	C(45)	C(46)	120.6(8)
C(40)	C(46)	C(45)	120.3(8)
C(47)	C(48)	C(49)	119.6(11)
C(47)	C(48)	C(53)	120.0(12)
C(49)	C(48)	C(53)	120.4(12)
C(48)	C(49)	C(50)	120.1(13)
C(49)	C(50)	C(51)	119.7(13)
C(50)	C(51)	C(52)	116.8(13)
C(51)	C(52)	C(53)	122.4(17)
C(48)	C(53)	C(52)	120.7(14)

Table 5. Distances, in Angstroms,
from β -Hydrogens of the Ethyl Ligands to the
Molybdenum Atoms in the $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$
Molecule, as Located in the Difference Map.

The numbering scheme for the hydrogen
atoms is given in Figure 5.

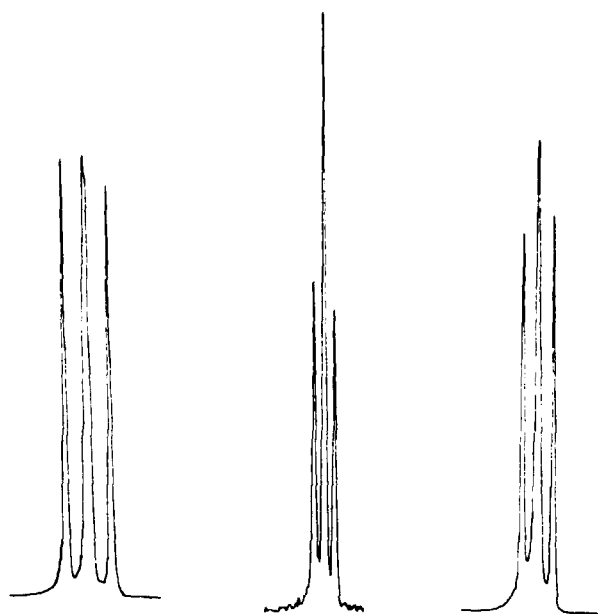
Atom	Atom	Distance ^a
Mo(1)	H(1)	3.5(1)
Mo(1)	H(2)	3.6(1)
Mo(1)	H(3)	4.2(1)
Mo(2)	H(4)	3.5(1)
Mo(2)	H(5)	3.7(1)
Mo(2)	H(6)	4.1(1)
Mo(1)	H(4)	3.4(1)
Mo(1)	H(5)	4.2(1)
Mo(1)	H(6)	4.7(1)
Mo(2)	H(1)	3.4(1)
Mo(2)	H(2)	4.0(1)
Mo(2)	H(3)	4.8(1)

^aErrors are estimated based on the internal
consistency of the C-H distances and angles, as
located in the difference synthesis.

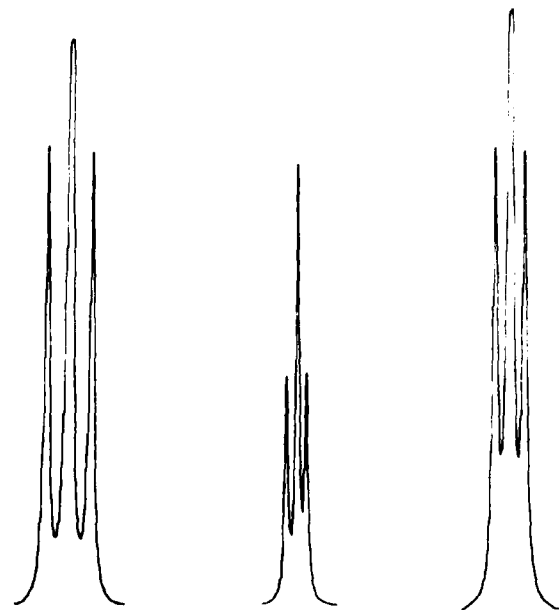
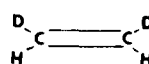
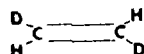
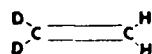
Captions to Figures

- Figure 1. ^2H nmr spectra for the isomers of $\text{C}_2\text{H}_2\text{D}_2$ recorded in benzene (top) are compared with the predicted spectra (bottom) based on ^1H - ^1H coupling constants for ethylene and (^1H) taken from R.M. Lynden-Bell and M. Sheppard, Proc. R. Soc. A. 269, 1385 (1965). (See Experimental Section).
- Figure 2. ^2H nmr spectra of the ethanes liberated in the reactions between $\text{Mo}_2(\text{CH}_2\text{CD}_3)_2(\text{NMe}_2)_4$ and CO_2 (A) and $\text{Mo}_2(\text{CD}_2\text{CH}_3)_2(\text{NMe}_2)_4$ and CO_2 (B) are compared with spectra associated with commercially supplied samples of $\text{CD}_3\text{CH}_2\text{D}$ and $\text{CH}_3\text{CD}_2\text{H}$, respectively.
- Figure 3. ^1H nmr spectra showing the formation of $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{O}_2\text{CNMe}_2)_2$ at 100 MHz in the reaction between $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_4$ and CO_2 in toluene- d_8 at -30°C . The signals denoted by * correspond to the protio impurities in toluene- d_8 . The signal denoted by ** corresponds to the methyl resonance of $\text{Mo}_2(\text{O}_2\text{CNMe}_2)_4$ which, because of its low solubility, is precipitated as it is formed.
- Figure 4. An ORTEP view of the $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ molecule showing the atom numbering scheme used in the tables.
- Figure 5. An ORTEP view of the central skeleton of the $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{-C}_7\text{H}_8)_2$ molecule viewed down the $\text{Mo}\equiv\text{Mo}$ bond showing the arrangement of the ethyl groups.

ISOMERS OF DIDEUTEROETHYLENE



EXPERIMENTAL



CALCULATED

←4.8→

←2.2→

←3.3→

(Hz)

A. $\text{Mo}_2(\text{CH}_2\text{CD}_3)_2(\text{NMe}_2)_4 + \text{CO}_2$

B. $\text{Mo}_2(\text{CD}_2\text{CH}_3)_2(\text{NMe}_2)_4 + \text{CO}_2$



A



$\text{CD}_3\text{CH}_2\text{D}$



B



$\text{CH}_3\text{CD}_2\text{H}$

