dependent upon the number of aliphatic nitrogen donors, the above indicates that the presence of μ-ligand bridges results in less stable O_2 adducts. This postulate is further supported in comparison with oxygenation constants of μ-peroxo-μ-hydroxo-dicobalt(III) cations. McLendon and Martell[20] have shown that there exists a good direct correlation between the sum of the ligand protonation constants and the oxygenation constant (eq 4). The values for $[Co_2(en)_5(O_2)^{4+}]$ and

$$2CoL_x^{y+} + O_2 \rightleftharpoons [Co_2L_{2x}(O_2)^{2y+}] \qquad (4)$$

$[Co_2(trien)_2(en)(O_2)^{4+}]$ fall below the line for that of the μ-peroxo-μ-hydroxo-dicobalt(III) cations, indicating that the presence of μ-en bridges results in a somewhat less stable O_2 adduct.

Finally, the μ-ligand-μ-peroxo-dicobalt(III) complexes are reversible (release O_2) upon the addition of acid both in the acid buffer region and at high pH. Dilute solutions of the μ-en-bridged cations were stable for at least weeks to further reaction to yield mononuclear Co(III) species which are similar to most other (polyalkylenepolyamine)-μ-peroxo-dicobalt(III) cations.[14] However, concentrated solutions of the cations (required for NMR studies) seemed to be less stable, suggesting that further reaction to mononuclear Co(III) cations may occur via an intermolecular pathway or that perhaps the presence of small amounts of unoxygenated Co(II) cations can lead to accelerated rates.

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Reactions of Coordinated Dinitrogen. 6.[1] Displacement of Coordinated Dinitrogen by Dihydrogen in Low-Valent Molybdenum Complexes

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Bis(dinitrogen) complexes of molybdenum, $Mo(N_2)_2L_2$, where L is a bidentate tertiary phosphine ligand react rapidly in solution with dihydrogen to form MoH_4L_2. Conformation of a tetrahydride rather than a dihydride comes from both ^1H and ^{31}P NMR spectral studies using the specially prepared highly soluble bidentate phosphine ligands $(tol)_2PCH_2CH_2P(tol)_2$, where tol is *m*-tolyl (dmtpe) or *p*-tolyl (dptpe).

Introduction

Early reactions of transition-metal complexes of dinitrogen with dihydrogen resulted in displacement of N_2 by H_2 (e.g., eq 1).[3] These reactions are often reversible. Dihydrogen was

$$CoH(N_2)(PPh_3)_3 + H_2 \rightleftarrows N_2 + CoH_3(PPh_3)_3 \qquad (1)$$

reported by Hidai et al. to react with $Mo(N_2)_2(dppe)_2$, **1**, where dppe is $Ph_2PCH_2CH_2PPh_2$, to form a dihydride, $MoH_2(dppe)_2$, **2**, in benzene solution over a 3-day period.[4] On the other hand, in toluene solution a binuclear hydride, $[trans\text{-}MoH_2(dppe)]_2(\mu\text{-}dppe)$, **3**, was reported to be formed. In benzene solution **3** was converted into **2**, while in toluene solution **2** was converted into **3**. It was stated that these compounds were insufficiently soluble in organic solvents to allow their molecular structure to be determined by NMR spectroscopy. Aresta and Sacco also reported the formation of $MoH_2(dppe)_2$ from the reaction of **1** with dihydrogen.[5] The ^1H NMR spectrum of **2** in C_6D_6 at 60 °C gave a 1:4:6:4:1 quintet centered at τ 13.64 (J_{P-H} = 30 Hz) showing four equivalent phosphorus atoms. No spectrum could be obtained below 60 °C because of the poor solubility of **2** in suitable solvents. The stereochemistry of **2** depended upon the solvents, trans being formed in benzene solution and cis in tetrahydrofuran (THF) solution. Compound **2** was also formed by the reduction of $MoCl_4(dppe)$ in benzene solution with sodium and under a dihydrogen atmosphere.[5] Previously, Turio et al. had reported the preparation of **2** by the reduction of molybdenum(III) acetylacetonate with triisobutylaluminum in benzene solution containing dppe.[6] The poor solubility of **2** again precluded NMR studies. In the meantime, Pennella reported the preparation of $MoH_4(dppe)_2$, **4**, by the reduction of $MoCl_4(dppe)$ with an ethanol solution of sodium borohydride containing added dppe.[7] Characterization of **4** relied upon a comparison of the ν(Mo–H) frequency with those of $MoH_4(PMePh_2)_4$ and $MoH_4(PEtPh_2)_4$. The latter two compounds had been characterized by ^1H NMR spectroscopy in which the integration agreed with a formulation of four rather than two hydridic hydrogen atoms per molecule. Once again the poor solubility of the dppe hydride prevented an NMR spectral study.

Meakin and co-workers reported the first room-temperature ^1H NMR spectrum of **4**, in chlorobenzene.[8] They observed that the hydride region of the ^{1}H NMR spectrum consisted of a binomial quintet at all temperatures attainable. Confidence for the tetrahydride formulation was also based upon a single-crystal X-ray analysis of $MoH_4(PMePh_2)_4$.[8,9]

We had previously obtained the tetrahydride $MoH_4(dppe)_2$ by reacting **1** with dihydrogen in benzene solution for short periods of time.[10] In order to further characterize the product from the reaction of **1** with dihydrogen, we synthesized two new bidentate ligands to improve the solubility properties of the resulting hydrides formed. These ligands were $(m\text{-tol})_2PCH_2CH_2P(\text{tol-}m)_2$ (dmtpe) and $(p\text{-tol})_2PCH_2CH_2P(\text{tol-}p)_2$ (dptpe). Part of the work reported in this paper has been communicated.[11,12]

Experimental Section

All preparations and crystallizations were carried out in either argon, dihydrogen, or dinitrogen atmosphere by using standard vacuum-line, drybox (Vacuum/Atmospheres Co.), or glovebag techniques.

All solvents were reagent grade and were dried and distilled under an inert atmosphere from the drying agents given in parentheses: THF ($LiAlH_4$); cyclohexane, benzene, heptane, pentane, and dichloromethane (CaH_2); methanol (magnesium methoxide); acetonitrile (P_4O_{10}). Solvents were bubbled with either argon, dihydrogen, or dinitrogen for 0.3 h before use.

Molybdenum(V) chloride was purchased from Alfa Inorganics, Inc., tri-*m*-tolylphosphine and tri-*p*-tolylphosphine from Eastman Organic Chemicals, and dppe from ROC/RIC Inorganic Chemicals, and all were used as received. $MoCl_3(THF)_3$ was prepared by a published procedure.[13]

Infrared spectra were recorded with a Perkin-Elmer 621 spectrophotometer. Solid samples were obtained as cesium iodide pellets, mulls were taken between cesium iodide plates, and solutions were obtained in cyclohexane by using 1-mm sodium chloride solution cells. NMR spectra were recorded on Varian A-60D and XL-100 spectrometers and ^{31}P NMR spectra were referenced to 85% H_3PO_4 (89 950 Hz). Magnetic susceptibility measurements were taken at 20 °C on solid samples by the Gouy method with $FeSO_4 \cdot 7H_2O$ as calibrant. All susceptibilitites were corrected for diamagnetism by using values given by Pascal.[14] Melting points and decomposition points were recorded in sealed capiliaries and are uncorrected.

All analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, NY.

Preparation of Phosphines dmtpe and dptpe. These phosphines were prepared according to the published procedure for the syntheses of dppe from PPh_3 by using lithium metal in THF solution.[15] Typically, a mixture of either $(m\text{-tol})_3P$ or $(p\text{-tol})_3P$ (10.0 g, 32.8 mmol) and lithium metal pieces (1.0 g) was refluxed for 3 h in THF (75 mL) with magnetic stirring. After the mixture was cooled to 0 °C, the remaining pieces of metal were removed from the reaction mixture with tweezers. A solution of 1,2-dichloroethane (10 mL) in THF (50 mL) was added dropwise. After workup with methanol (25 mL) and water (200 mL), the volume of the resulting cloudy suspension was reduced to ca. 50 mL and extracted with hexane. The hexane solution was dried ($MgSO_4$) and filtered, and solvent was removed in vacuo to give a cream-colored semisolid oil. This material was heated to 170 °C in a sublimer to remove impurities. The resulting oil (>90% yield) was used without further purification in reactions.[16] Both dmtpe and dptpe were converted to their disulfides for characterization. The sulfides were prepared by heating benzene solutions of the phosphines with excess elemental sulfur at reflux. The disulfides were recrystallized from benzene–pentane solutions in low yields.

Preparation of the Dichlorides $MoCl_2(dppe)_2$, $MoCl_2(dmtpe)_2$, and $MoCl_2(dptpe)_2$. To a three-necked round-bottom flask containing THF (50 mL), 50 g (excess) of freshly prepared 0.5% magnesium amalgam, and dppe (3.8 g, 9.54 mmol) was added $MoCl_3(THF)_3$ (2.0 g, 4.78 mmol). An argon atmosphere was maintained during 4 h of mechanical stirring. The mixture was decanted from the amalgam and the solid filtered off. The yellow solid was washed twice with THF and then crystallized from a benzene–methanol solution. Yellow crystalline $MoCl_2(dppe)_2$ was dried in vacuo to yield 3.99 g (87%); dec pt >260 °C.[18]

The preparation of $MoCl_2(dmtpe)_2$ and $MoCl_2(dptpe)_2$ was carried out analogously. Both were crystallized from benzene–methanol solutions in 29% (dec pt >219 °C) and 34% (dec pt >240 °C) yields, respectively.

Preparation of the Tetrahydrides $MoH_4(dppe)_2$, $MoH_4(dmtpe)_2$, and $MoH_4(dptpe)_2$. A mixture of $MoCl_2(dmtpe)_2$ (1.0 g, 0.93 mmol) in THF (100 mL) and 40 g (excess) of 1% sodium amalgam was stirred magnetically for 5 h while dihydrogen was bubbled through the solution. The resulting solution was decanted and filtered. Solvent was removed from the solution to yield a dark yellow solid. Crystallization from a benzene–methanol solution produced yellow crystals of $MoH_4(dmtpe)_2$ (0.77 g, 82%); dec pt 110 °C. $MoH_4(dppe)_2$ and $MoH_4(dptpe)_2$ were prepared analogously in 78% and 66% (dec pt 165 °C) yields, respectively.

Preparation of $Mo(N_2)_2(dppe)_2$, $Mo(N_2)_2(dmtpe)_2$ and $Mo(N_2)_2(dptpe)_2$. A mixture of $MoCl_2(dmtpe)_2$ (0.5 g, 0.46 mmol) in THF (100 mL) and 30 g (excess) of 1% sodium amalgam was stirred magnetically for 5 h while dinitrogen was bubbled through the solution. This solution was decanted and filtered, and solvent removed to yield a dark yellow solid. Yellow crystalline $Mo(N_2)_2(dmtpe)_2$ (0.41 g, 84%), dec pt 140 °C, was obtained from a benzene–methanol solution.

$Mo(N_2)_2(dppe)_2$ and $Mo(N_2)_2(dptpe)_2$ were obtained analogously in 78% (dec pt 165 °C) and 77% (dec pt 145 °C) yields, respectively.

Reaction of Dihydrogen with $Mo(N_2)_2(dppe)_2$, $Mo(N_2)_2(dmtpe)_2$, and $Mo(N_2)_2(dptpe)_2$. Dihydrogen was bubbled through a magnetically stirred solution of the bis(dinitrogen) complex (0.09 mmol) in cyclohexane (75 mL)[20] at ambient temperature. Aliquots were removed periodically and the disappearance of $\nu(NN)$ and appearance of $\nu(MoH)$ monitored. After 2.5 h the $\nu(NN)$ band had completely disappeared, and a weak broad absorption band due to $\nu(MoH)$ was observable. The reaction was stopped after 4 h and solvent removed in vacuo to yield a yellow solid that was shown by the infrared and ^{1}H NMR spectra to be the corresponding tetrahydride.

Reaction of Dinitrogen with $MoH_4(dppe)_2$, $MoH_4(dmtpe)_2$, and $MoH_4(dptpe)_2$. Dinitrogen was bubbled through a magnetically stirred solution of the tetrahydride complex (0.09 mmol) in cyclohexane (75 mL)[20] at ambient temperature. Solution infrared spectra were taken during the course of the reaction to monitor loss of $\nu(MoH)$ and the appearance of $\nu(NN)$. The reaction was monitored for 40 h after which time solvent was removed in vacuo to give a yellow solid whose infrared and ^{1}H NMR spectra showed it to be a mixture of the bis(dinitrogen) (ca. 90%) and the tetrahydride (ca. 10%) complexes. In the case of $MoH_4(dppe)_2$ the mixture after 24 h was ca. 1:1 tetrahydride to bis(dinitrogen) complex.

Results and Discussion

Syntheses and Characterization. Two new bidentate tertiary phosphines, dmtpe and dptpe,[16] were prepared as analogues of dppe in order to improve the solubility properties of transition-metal complexes containing the dppe ligand but without dramatically altering the electronic properties. This work was begun to try and solve the problem of the identity of the hydride formed when dihydrogen displaces dinitrogen from $Mo(N_2)_2(dppe)_2$, eq 2. Work reported in the literature[4,5]

$$Mo(N_2)_2(dppe)_2 + 2H_2 \rightarrow MoH_4(dppe)_2 + 2N_2 \quad (2)$$

was confusing, and the physical properties of the putative $MoH_2(dppe)_2$ were very similar to $MoH_4(dppe)_2$ prepared from $MoCl_4(dppe)$.[7,8] dmtpe and dptpe proved eminently suitable to help answer the question.[11]

dmtpe and dptpe were prepared in high yield according to a published procedure for dppe[15] and were not purified exhaustively before use in preparing the resulting molybdenum complexes. The methyl substituents on the phenyl groups not only provided the enhanced solubility that had been anticipated (when complexed) but also provided a sharp singlet in the ^{1}H NMR spectrum at ca. τ 7.7 (see Table I) that was very useful for integrating the spectra of the hydrides to establish the ratio of hydridic hydrogen atoms per phosphine ligand in the molecule.

It was decided to use the molybdenum(II) chloride complexes as precursors for the preparation of the bis(dinitrogen) and hydride complexes because of their good relative stability and the high yields of conversion to final products. The dichlorides were prepared by the magnesium amalgam reduction of $MoCl_3(THF)_3$ in the presence of 2 equiv of phosphine ligand under an argon atmosphere (e.g., eq 3).[13,21] Only $MoCl_2$-

Table I. Analytical, Spectral, and Magnetic Moment Data

	Anal.												
	% C		% H		other			^{1}H NMR, τ (rel area)^b					
compd	calcd	found	calcd	found	calcd	found	IR (ν(AB)), cm^{-1} ^{a}	CH$_3$	CH$_2$(P)	phenyl	H(Mo)	solvent	μ_{eff}, μ_B^{c}
dmtpe^d	69.45	69.45	6.22	6.36	12.37^e	12.36^e		8.01 (3) s^f	7.67 (1) t^f			C_6H_6	
dptpe^g	69.45	66.67	6.22	6.35	12.37^e	10.29^e		7.95 (3) s^h	7.70 (1) t^h			C_6H_6	
$MoCl_2(dppe)_2$	64.81	64.18	5.02	5.37	7.36^i	7.18^i	313 m^j						2.99
$MoCl_2(dmtpe)_2$	66.98	67.21	5.99	6.17	6.59^i	6.29^i	313 m^j						2.89
$MoCl_2(dptpe)_2$	66.98	66.65	5.99	6.28	6.59^i	7.72^i	309 m^j						2.86
$MoH_4(dmtpe)_2$^{k}	71.42	70.51	6.79	7.29			1750 m, br^l 761w^m	7.97 (6) s	7.65 (2) t	2.7 (8) m	13.59 (1) q^n	C_6D_6	
$MoH_4(dptpe)_2$	71.42	71.12	6.79	6.83			1750 m, br^l 761w^m	7.94 s	7.60 t	2.8 m	13.55 q^o	C_6D_6	
$Mo(N_2)_2(dmtpe)_2$	67.92	67.38	6.08	6.14	5.28^p	5.10^p	2017 vw, 1960 s	7.97 s	7.63 t	2.8 m		C_6D_6	
$Mo(N_2)_2(dptpe)_2$	67.92	67.89	6.08	5.89	5.28^p	5.12^p	2019 vw, 1970 s	7.94 s	7.50 t	2.9 m		C_6D_6	

[a] vs = very strong, s = strong, m = medium, w = weak, vw = very weak. [b] Relative to $Si(CH_3)_4$; s = singlet, d = doublet, t = triplet, q = quintet, m = multiplet, br = broad. [c] Solid state at 20 °C. [d] Analyzed as the disulfide, mp 220 °C. [e] Sulfur analysis. [f] For the disulfide: 8.12 s (CH_3), 6.89 m (CH_2). [g] Analyzed as the disulfide, mp 60 °C. [h] For the disulfide: 8.05 s (CH_3), 6.95 m (CH_2). [i] Chlorine analysis. [j] ν(Mo–Cl). [k] ^{31}P NMR (C_6D_6): −84 ppm (q, J_{PH} = 30 Hz). [l] ν(Mo–H). [m] δ(Mo–H). [n] J_{PH} = 30 Hz. [o] J_{PH} = 31 Hz. [p] Nitrogen analysis.

$$MoCl_3(THF)_3 + 2dmtpe \xrightarrow[THF]{Mg/Hg;\ Ar} MoCl_2(dmtpe)_2 + {}^1/{}_2MgCl_2 \quad (3)$$

$(dppe)_2$ has been reported previously.[5,6,13,21] All three dihalides exhibit one ν(Mo–Cl) band ca. 311 cm^{-1}, suggesting a trans configuration, and have a magnetic moment at 20 °C (see Table I) very close to the spin-only value calculated[23] for a D_{4h} mononuclear six-coordinate molybdenum(II) (d^4) complex. Similar six-coordinated complexes of molybdenum of lower symmetry are often diamagnetic, e.g., $Mo(SR)_2(dppe)_2$ and $MoCl(SPh)(dppe)_2$.[24]

The bis(dinitrogen) complexes were all prepared in high yield by the sodium amalgam reduction of the dichloride in THF under an atmosphere of dinitrogen, e.g., eq 4. $Mo(N_2)_2(dmtpe)_2$ and $Mo(N_2)_2(dptpe)_2$ were yellow. The infrared spectra showed ν(NN) bands at ca. 2018 (vw) and ca. 1970 (s) cm^{-1} both in cesium iodide pellets (see Table I) and in solution consistent with a trans configuration.[19]

$$MoCl_2(dmtpe)_2 + 2N_2 \xrightarrow[THF]{Na/Hg} Mo(N_2)_2(dmtpe)_2 + 2NaCl \quad (4)$$

The hydrides were similarly prepared from the dichlorides except under a dihydrogen atmosphere. The infrared spectrum of $MoH_4(dppe)_2$ gave a broad unresolved band at 1750 cm^{-1} due to ν(Mo–H)[25] and a weak band at 761 cm^{-1} due to δ(Mo–H) in a cesium iodide pellet. $MoH_4(dmtpe)_2$ and $MoH_4(dptpe)_2$ both show ν(Mo–H) and δ(Mo–H) in the same regions as noted for $MoH_4(dppe)_2$ (see Table I). When $MoH_4(dppe)_2$ that had been formed in benzene (from $MoCl_2(dppe)_2$) was recrystallized from a toluene–hexane solution, ν(Mo–H) moved to 1735 cm^{-1}. However, upon recrystallization of this sample from a benzene–hexane solution, ν(Mo–H) shifted back to 1750 cm^{-1}. These observations agree with those reported by Hidai,[4] but there is no evidence for a complex equilibrium suggested between **2** and **3**, and as will be shown below there is no evidence of a dihydride under our working conditions either by reduction of the dichloride under dihydrogen or by displacement of dinitrogen with dihydrogen.

In the ^{1}H NMR spectra of $MoH_4(dmpte)_2$ and $MoH_4(dptpe)_2$ at ambient temperature, the hydridic hydrogens appeared as a 1:4:6:4:1 quintet at τ 13.59 (J_{PH} = 30 Hz) and 13.55 (J_{PH} = 31 Hz), respectively, and the tolyl methyl protons at τ 7.97 and 7.94, respectively. The integrated areas under the peaks indicated four hydride hydrogen atoms per two phosphine ligands, i.e., a tetrahydride. When phosphorus was decoupled, the quintet collapsed to a sharp singlet, and integration again supported the formulation as a tetrahydride. However, the most conclusive piece of evidence came from the selectively proton-decoupled[26] ^{31}P NMR spectrum of $MoH_4(dmtpe)_2$ obtained at 40.5 MHz. The spectrum showed a 1:4:6:4:1 quintet arising from the coupling of four equivalent hydridic hydrogens with the four equivalent phosphorus atoms. A trans dihydride would be expected to produce a triplet but not a binomial quintet. Complete proton decoupling collapsed the quintet to a singlet.

The variable-temperature ^{1}H NMR spectrum of $MoH_4(dmtpe)_2$ was run at 100.1 MHz in both toluene and methylene chloride solutions between 0 and −70 °C and between 0 and −50 °C, respectively. These spectra are shown in Figure 1. In toluene solution, all lines in the quintet begin to broaden and merge as the temperature is lowered in a very similar manner to the behavior of $MoH_4(dppe)_2$ in chlorobenzene solution.[8] On the other hand, in dichloromethane solution the two outer lines remain sharp while a broad central region develops as the temperature decreases. This latter spectrum

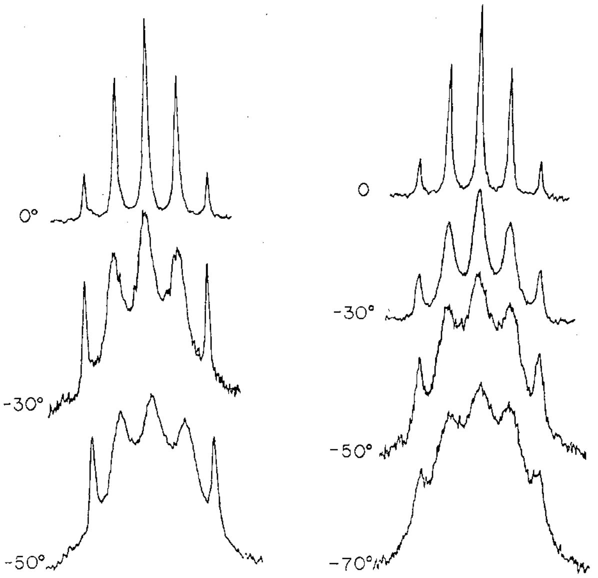

Figure 1. Temperature-dependent hydride region of the ^1H 100.1 MHz NMR spectrum of $MoH_4(dmtpe)_2$ in toluene (right) and dichloromethane (left) solutions, respectively.

is more like the limiting low-temperature spectra reported by Meakin et al.[8] for $MoH_4(PMe_2Ph)_4$, $MoH_4(PEtPh_2)_4$, and $MoH_4[PPh(OEt)_2]_4$. Clearly, $MoH_4(dmtpe)_2$ is stereochemically nonrigid and is undergoing an intramolecular mutual exchange process in which all the hydridic hydrogen atoms are equally coupled to all ^{31}P nuclei.[8]

Exchange Reactions. When dihydrogen was bubbled through cyclohexane solutions of $Mo(N_2)_2(dmtpe)_2$ and $Mo(N_2)_2(dptpe)_2$, dinitrogen was rapidly lost to form the corresponding tetrahydride, which was identified by ^1H and ^{31}P NMR ($MoH_4(dmtpe)_2$) and infrared spectroscopy. No evidence was found for any other species. When the same reaction was carried out with $Mo(N_2)_2(dppe)_2$ in benzene, the product was again identified as the tetrahydride by ^1H NMR and infrared spectroscopy, with no evidence for any other products.

In reactions of $MoH_4(dmtpe)_2$ and $MoH_4(dptpe)_2$ with dinitrogen, exchange occurred much more slowly than the dinitrogen exchange by dihydrogen (>16 times slower according to the rate of increase or decrease of $\nu(NN)$ in the infrared spectrum), and in fact after 40 h the exchange was not complete. Spectroscopic investigation of the product showed a mixture of the bis(dinitrogen) (ca. 90%) and tetrahydride (ca. 10%) complexes. $MoH_4(dppe)_2$ also slowly reacted with dinitrogen to form a mixture of the two products $MoH_4(dppe)_2$ and $Mo(N_2)_2(dppe)_2$.

In summary, in our hands the replacement of dinitrogen from complexes such as $Mo(N_2)_2(dmtpe)_2$ results in the formation of a tetrahydride, $MoH_4(dmtpe)_2$, that is identical to the tetrahydride formed by the reduction of either $MoCl_4(dmtpe)$ with sodium borohydride in ethanol with 1 equiv of dmtpe or $MoCl_2(dmtpe)_2$ with sodium amalgam under a dihydrogen atmosphere. We find no evidence for a dihydride such as **2** or a binuclear hydride such as **3**. The ^1H NMR spectrum of the putative dihydride **2** appears to be identical[5] in the hydridic hydrogen region with the tetrahydride.[8,11,27]

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Registry No. dmtpe, 70320-29-5; dptpe, 70320-30-8; $MoCl_2(dppe)_2$, 59122-11-1; $MoCl_2(dmtpe)_2$, 70354-82-4; $MoCl_2(dptpe)_2$, 70320-85-3; $MoH_4(dppe)_2$, 32109-09-4; $MoH_4(dmtpe)_2$, 42593-57-7; $MoH_4(dptpe)_2$, 70320-86-4; $Mo(N_2)_2(dppe)_2$, 25145-64-6; $Mo(N_2)_2(dmtpe)_2$, 70354-83-5; $Mo(N_2)_2(dptpe)_2$, 70320-87-5; $MoCl_3(THF)_3$, 31355-55-2; (*m*-tol)$_3$P, 6224-63-1; (*p*-tol)$_3$P, 1038-95-5; 1,2-dichloroethane, 107-06-2.

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