Table I. Calculated Ground- and Excited-State Bond Lengths for $Rh_2b_4^{2+}$

vib	ν_{gnd}, cm^{-1}	ν_{exc}, cm^{-1}	r_{gnd}, Å	r_{exc}, Å	Δr, Å
ν(Rh—Rh)	79[a] (0.189)[b]	144[a] (0.629)	3.175	2.965	−0.210
ν(Rh—C)	467[a] (1.381)	484[a] (1.483)	2.253	2.231	−0.022
ν(C≡N)	2193 (18.31)	2177 (18.04)	1.132	1.134	+0.002

[a] Values taken from ref 2. [b] Values in parentheses are force constants in mdyn/Å.

centration-dependent differences in the TRIR results were observed.

The absorbance increase at 2177 cm^{-1} is assigned to the excited-state C≡N stretch. The shift to lower energy in the excited-state vibration may be rationalized in terms of the excited-state electronic structure. The optically excited transition is the $^1A_{1g} \rightarrow {}^1A_{2u}$ ($d\sigma^* \rightarrow p\sigma$) transition.[6] The $^1A_{2u}$ state rapidly decays to the TRIR-observed $^3A_{2u}$ ($p\sigma$) state.[4] The change from antibonding ($d\sigma^*$) to bonding ($p\sigma$) character along the Rh–Rh axis is consistent with the observed increase in the ground-state metal–metal frequency from 79 cm^{-1} to that in the excited-state of 144 cm^{-1}, indicating an increase in the Rh–Rh bond order.[2] The ($p\sigma$) metal–metal orbital that is populated in the excited state has the correct symmetry for overlap with the π systems of the C≡N group of the isocyanide ligands. This overlap is, to a first approximation, lacking for the ($d\sigma^*$) metal–metal orbital, which is the HOMO of the ground state. Thus, increased backbonding from metal to isocyanide is expected in the excited state. This increased backbonding is manifested in the previously observed increase in the ground-state Rh–C stretching frequency from 467 cm^{-1} to the excited-state frequency of 484 cm^{-1}.[2] The additional electron density donated through π-backbonding is expected to be accommodated in an antibonding $2p\pi^*$ orbital of the C≡N group, resulting in a somewhat reduced C≡N force constant.[7] It is therefore expected that, upon photoexcitation, the C≡N stretching frequency will decrease. In fact, we observe a decrease from 2193 to 2177 cm^{-1}.

Knowledge of the excited-state C≡N frequency completes the vibrational information required for determining the excited-state bond displacements occurring in $Rh_2b_4^{2+}$. From systems with known force constants for vibrations between two atoms with known bond distances, Woodruff and co-workers[8] have developed a set of empirical rules that allows the calculation of unknown bond distances from measured force constants. The empirical results take the form of $r = A + B[\exp(-k/C)]$, where r is the bond distance in angstroms, k is the force constant in mdyn/Å, and A, B, and C are best fit coefficients determined for diatomic interactions between atoms of different rows.[9] The results for the Rh—Rh, Rh—C, and C≡N vibrations are summarized in Table I.

The calculations indicate that the largest displacements occur along the Rh—Rh bond (−0.210 Å) and between the Rh—C bond (−0.022 Å), while the C≡N bond undergoes very little displacement (+0.002 Å). This is consistent with expectations in that little metal-to-ligand charge-transfer character is expected for this transition. However, no quantitative estimate of this displacement has been reported previously. Analysis of vibrational progressions resolved in low-temperature UV/vis spectra of the dimer suggested only an upper limit of 0.01 Å for the displacement of the C≡N coordinate.[6] A comparison of our results and the vibronic analysis underscores the importance of the ability to obtain excited-state vibrational spectra using TRIR spectroscopy in that much smaller displacement values may be estimated.

The accuracy of the displacement calculations and, thus, the picture of the excited-state geometry that they reveal are limited by the assumptions inherent in the application of the simple empirical rules. Application of these rules to our data implies that each vibration is solely a local stretching mode, rather than a normal-coordinate displacement involving several bond length and bond angle changes. It is, however, reasonable to expect that, upon shortening of the Rh—Rh bond in the excited state, the Rh—C≡N bond angle might change. Thus, some Rh-CN bending character could be mixed into the vibrational modes assumed to be of only local stretching character. Future work will be directed at extending the nanosecond TRIR studies to a series of Rh–Rh ligand-bridged systems in which the chain length of the diisocyanide bridge is varied to see how the steric constraints imposed by the bridge affect excited-state behavior in these systems.

Acknowledgment. We wish to thank Dr. David C. Smith for his donation of a sample of $Rh_2b_4^{2+}$ for these studies. This work was supported by Los Alamos National Laboratory under LDRD Grant X15D. K.C.G. gratefully acknowledges support by a LANL director's postdoctoral fellowship. This work was performed at LANL under the auspices of the U.S. Department of Energy.

---

(6) Rice, S. F.; Miskowski, V. M.; Gray, H. B. *Inorg. Chem.* **1988**, *27*, 4704.

(7) Nakamoto, K. *Infrared and Raman Spectra of Inorganic and Coordination Compounds*, 4th ed.; Wiley: New York, 1986; p 272.

(8) Woodruff, W. H. Unpublished results. See also: Miskowski, V. M.; Dallinger, R. F.; Christoph, G. G.; Morris, D. E.; Spies, G. H.; Woodruff, W. H. *Inorg. Chem.* **1987**, *26*, 2127.

(9) A, B, and C are 1.077, 0.629, and 7.539, respectively, for two first-row elements (i.e. C≡N); 1.816, 1.461, and 2.610 for two interacting fourth-row elements (Rh—Rh); and 1.617, 1.040, and 2.812 for interactions between a first- and a fourth-row element (Rh—C).

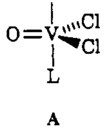
Contribution from the Department of Chemistry
and Ames Laboratory, Iowa State University,
Ames, Iowa 50011-3111

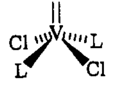
Unexpected Formation of a Novel Divanadium(IV) Monophosphine Complex

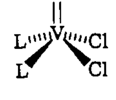
A. K. Fazlur-Rahman, V. G. Young, Jr., and J. G. Verkade*

Received September 5, 1991

While complexes of the type $L_nV(O)Cl_2$, wherein L is a monodentate (n = 1, 2, or 3) or a bidentate neutral ligand, have been known for many years,[1] only a few such complexes containing phosphine ligands have been reported and no reports of stable arsine or stibine adducts exist. The only reported compounds containing phosphines include $(dppe)(H_2O)V(O)X_2$ (X = Cl, Br),[2] $(dppm)(H_2O)V(O)Br_2$,[2] $(Ph_3P)_2V(O)Cl_2$,[3] $(Ph_2MeP)_2V(O)Cl_2$,[3] and $(dppm)V(O)Cl_2$.[3] $L_2V(O)Cl_2$ complexes, wherein L is NMe_3[4] and $OC(NMe_2)_2$,[5] have been shown by X-ray diffraction means to display a trigonal bipyramidal (A) and a square pyramidal (B) geometry, respectively. However, no reports of X-ray structures of the phosphine complexes were found. From IR data, the geometries of $(Ph_3P)_2V(O)Cl_2$ and $(Ph_2MeP)_2V(O)Cl_2$ have been


A


B


C

---

(1) (a) Clark, R. J. H. In *Comprehensive Inorganic Chemistry*; Bailar, J. C., Emeleus, H. J., Nyholm, R. S., Eds.; Trotman-Dickinson, A. F.; Pergamon Press: Oxford, 1973; Vol. 3. (b) Boas, L. V.; Pessoa, J. C. In *Comprehensive Coordination Chemistry*; Wilkinson, G., Gillard, R. D., McCleveny, J. A., Eds.; Pergamon Press: Oxford, 1987; Vol. 3.

(2) Selbin, J.; Vigee, G. *J. Inorg. Nucl. Chem.* **1968**, *30*, 1644.

(3) Cave, J.; Dixon, P. R.; Seddon, K. R. *Inorg. Chim. Acta* **1978**, *30*, L349.

(4) Drake, J. E.; Vekris, J.; Wood, J. S. *J. Chem. Soc. A* **1968**, 1000.

(5) Coetzer, J. *Acta Crystallogr.* **1970**, *B26*, 872.

(6) Kern, R. J. *J. Inorg. Nucl. Chem.* **1962**, *24*, 1105.

0020-1669/92/1331-2285$03.00/0

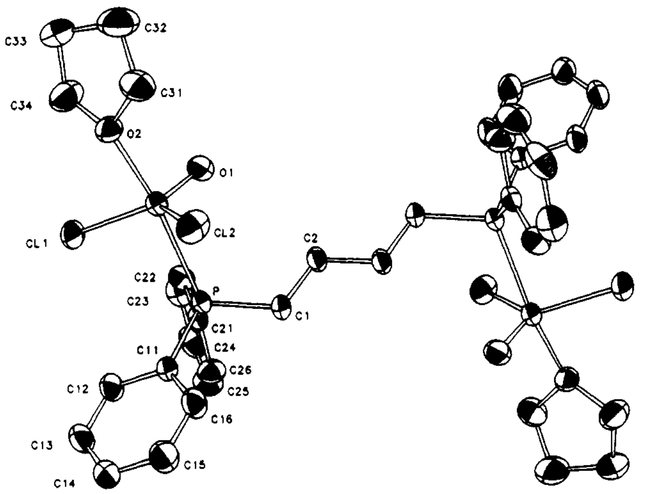


Figure 1. ORTEP drawing of **2** with elipsoids drawn at the 50% probability level.

deduced to be of type A, while that of (dppm)V(O)Cl_2 was inferred from such an analysis to possess structure C.[3] The reported synthesis of the phosphine adducts all involve $V(O)X_2$ as a starting material.

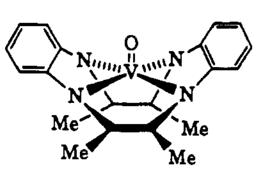
During the course of attempts to recrystallize a compound which may have been **1** (dppb = 1,4-bis(diphenylphosphino)butane), we

$$(THF)_3VCl_3 + \underset{\text{dppb}}{Ph_2P(CH_2)_4PPh_2} \rightarrow \underset{\mathbf{1}}{(THF)(dppb)VCl_3} + 2THF \quad (1)$$

apparently inadvertently introduced an adventitious source of oxygen and moisture causing the formation of the novel oxovanadium(IV) phosphine complex **2**. The mixture obtained from

$$8\mathbf{1} + 4H_2O + 2O_2 + 2THF \rightarrow dppb + \underset{\mathbf{2}}{(THF)Cl_2(O)VPPh_2(CH_2)_4PPh_2V(O)Cl_2(THF)} + 8HCl \quad (2)$$

reaction 1 was a brown suspension before it was filtered under inert atmosphere through Celite. However, the filtrate and its evaporated residue from which light blue-green **2** was recrystallized were blue-green, suggesting that the Celite was probably at least partially responsible for introducing moisture and oxygen, although THF as a source of oxygen cannot be ruled out. Taken by themselves, however, the colors exhibited in our reaction and work-up are inconclusive regarding the oxidation state of the metal since (dippe)VX_3(THF) (X = Cl, Br) is orange,[7] $(Et_3P)_2VCl_3$ is red,[8] (dmpe)(THF)VCl_3 is green,[8] and (dppm)(H_2O)V(O)Br_2 is also green,[2] as is (dppe)(H_2O)V(O)X_2 (X = Cl,Br).[2] Very recently, Cotton and co-workers[9] reported the structure of **3** which



3

they observed to crystallize from the filtrate of an orange-brown solution filtered through Celite. These authors do not comment on whether the green crystals formed a green or an orange filtrate, and attribute the source of the oxygen to tetrahydrofuran. Although we have not verified the stoichiometry shown in reaction 2, previous workers have demonstrated the involvement of oxygen and moisture in the formation of $(THF)_2V(O)Cl_2$ from $(THF)_3VCl_3$.[6]

Table I. Crystallographic Data for **2**

$V_2Cl_4P_2O_4C_{36}H_4$	formula weight 846.38
$a = 8.728$ (3) Å	space group $P\bar{1}$
$b = 9.676$ (4) Å	$T = -50$ (1) °C
$c = 11.859$ (3) Å	$\lambda = 0.71073$ Å
$\alpha = 93.90°$	
$\beta = 100.65$ (3)°	$\rho_{calcd} = 1.431$ g/cm^3
$\gamma = 90.41$ (3)°	$\mu = 8.9$ cm^{-1}
$V = 982.7$ Å^3	transmission coeff = 0.999–0.884
$Z = 1$	$R^a = 0.036$
	$R_w^{b,c} = 0.048$

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$; $w = 1/\sigma^2(|F_o|)$. [c] Quality-of-fit $= [\sum w(|F_o| - |F_c|)^2/(N_{obs} - N_{parameters})]^{1/2}$.

The molecular structure of the green complex **2** was shown by X-ray diffraction experiments to feature a trigonal bipyramidal geometry around each vanadium, in which a phosphorus is trans to the oxygen of a THF molecule in the centrosymmetric dppb-bridged structure (Figure 1). Apparently the four methylene carbons in the dppb ligand inhibit chelation (via potential displacement of the coordinated THF) which would give a structure of type B or C. This coupled with the bulky nature of each phosphine ligating site in dppb permitted formation of **2**, the first recorded example of a monophosphine oxovanadium(IV) complex.

The V–P bond length of 2.5100 (8) Å in **2** is somewhat longer compared with that in $CpV(CO)_2$(dppe) (2.442 Å)[10] and with the V–PPh_2 distance in $CpV(CO)[PhP(CH_2CH_2PPh_2)_2]$ (2.435 Å),[11] complexes in which the vanadium is monovalent. The V–P distance in the V(IV) complex **2** is, however, virtually the same as that found in the trivalent metal complex $CpV(PMe_3)_2Cl_2$ (2.51 Å).[8] The V–Cl distances in **2** (av 2.2751 (9) Å) are shorter than in $CpV(PMe_3)_2Cl_2$ (av 2.403 (1) Å)[8] but longer than in VCl_4 (2.138 Å)[12] and $(Me_3N)_2VCl_3$ (2.239 (4) Å).[13] The V–O1 distance of 1.585 (2) Å is typical of that in oxovanadium complexes.[13] The larger bond angle for Cl1–V–Cl2 in **2** (129.28 (4)°) compared with Cl1–V–O1 (114.20 (8)°) and Cl2–V–O1 (116.17 (8)°) in conjunction with the larger-than-90° O1–V–O2 (98.45 (9)°) and O1–V–P (93.49 (7)°) bond angles reflects a Berry pseudorotational distortion of the trigonal bipyramidal vanadium geometry toward a square pyramidal structure having an apical V=O oxygen.

The electron paramagnetic resonance (EPR) spectrum of **2** at room temperature in $CHCl_3$ solution revealed a resonance at 294 mT with the eight peaks of equal width at half height ($A_{iso} = 104$ G, $g = 1.96$) expected for the interaction of an unpaired electron with the nuclear quadrupolar spin of ^{51}V ($I = {}^7/_2$). No hyperfine splitting from ^{31}P ($I = {}^1/_2$) in the phosphine ligand was observed. This was also the case for the EPR spectrum of a powdered sample of $(Ph_3P)_2V(O)Cl_2$ taken at room temperature.[3]

Experimental Section

All manipulations were carried out under an inert atmosphere of dry nitrogen, using standard vacuum line and Schlenk techniques. THF was freshly distilled from sodium benzophenone ketyl. CH_2Cl_2 was distilled from CaH_2 and stored over molecular sieves. VCl_3·3THF was prepared according to a literature procedure.[14] VCl_3 and dppb were obtained from Strem Chemicals. The EPR spectrum of **2** was recorded on a Bruker ER 200G-FRC spectrometer operating at 9.46 GHz (X-band) in $CHCl_3$ solution at room temperature in a quartz tube.

Preparation of 2. VCl_3·3THF (600 mg, 1.60 mmol) was allowed to reflux in THF (50 mL) with dppb (700 mg, 1.64 mmol) for 6 h. The color of the solution changed from violet to orange brown. The reaction mixture was cooled to room temperature and filtered through Celite. The green filtrate was dried under vacuum to a green solid. The green solid was dissolved in CH_2Cl_2 and layered with *n*-hexane to give light blue-

---

(7) Hermes, A. R.; Girolami, G. S. *Inorg. Chem.* **1990**, *29*, 313.

(8) Nieman, J.; Teuben, J. H.; Huffman, J. G.; Caulton, K. G. *J. Organomet. Chem.* **1983**, *255*, 193.

(9) Cotton, F. A.; Czuchajowska, J.; Feng, X. *Inorg. Chem.* **1991**, *30*, 349.

(10) Rehder, D.; Müller, I.; Kopf, J. *J. Inorg. Nucl. Chem.* **1978**, *40*, 1013.

(11) von Deuten, K.; Rehder, D.; Roose, W. *J. Organomet. Chem.* **1981**, *214*, 71.

(12) Morino, Y.; Uehara, U. *J. Chem. Phys.* **1966**, *45*, 4543.

(13) Greene, P. T.; Orioli, P. L. *J. Chem. Soc. A* **1969**, *1621*.

(14) Fackler, J. P., Ed. *Inorganic Synthesis*; Wiley: New York, 1982; Vol. 21, p 135.

green acicular crystals. Yield: 437 mg, 64%. Anal. Calcd (found) for $V_2Cl_4P_2O_4C_{36}H_{44}$: C, 51.06 (51.88); H, 5.20 (5.43); P, 7.32 (7.49). IR (Nujol cm^{-1}): 2995 (s), 2852 (s), 1616 (m), 1457 (s), 1376 (s), 1260 (m), 1118 (s), 998 (s) (probably ν(V═O), 906 (m), 736 (s), 680 (br), 491, 461.

Structure determination of 2. A blue-green crystal of dimensions 0.20 × 0.10 × 0.10 mm was mounted on an Enraf-Nonius CAD4 diffractometer, and accurate cell parameters were derived from 25 independent reflections. The crystal data parameters and other pertinent details are presented in Table I.

Intensities were measured with graphite-monochromated Mo Kα radiation. A correction based on the decay of standard reflections was applied to the data. Lorentz and polarization corrections were applied as was an empirical absorption correction based on a series of Ψ-scans.

The centrosymmetric space group $P\bar{1}$ was indicated by intensity statistics. The positions of the V, P, Cl, and O atoms were taken from the direct-methods solution.[15] All remaining non-hydrogen atoms were found in one difference Fourier map. All hydrogen atoms were placed at ideal positions 0.95 Å from the bonded carbon atom. Refinement calculations were performed on a Digital Equipment Corp. MicroVAX II computer using the CAD4-SDP programs.[16]

Acknowledgment. Ames Laboratory is operated for the U.S. Department of Energy by Iowa State University under Contract No. 2-7405-ENG-82. This work was supported, in part, by the Assistant Secretary for Fossil Energy through the Pittsburgh Energy Technology Center. Partial support through DOE Grant No. DE-FG2288PC88923 is also acknowledged.

Supplementary Material Available: Tables of crystallographic data, anisotropic thermal parameters, bond lengths and bond angles, and general displacement parameters (5 pages); a table of calculated and observed structure factors (15 pages). Ordering information is given on any current masthead page.

---

(15) Sheldrick, G. M. *SHELXS-86*; Institut für Anorganische Chemie der Universität: Göttingen, FRG.

(16) Enraf-Nonius Structure Determination Package; Enraf-Nonius: Delft, Holland. Neutral-atom scattering factors and anomalous scattering corrections were taken from *International Tables for X-ray Crystallography*; The Kynoch Press: Birmingham, England, 1974.

Contribution from the Department of Chemistry, 6-331,
Massachusetts Institute of Technology,
Cambridge, Massachusetts 02139

Simple, High-Yield Syntheses of Molybdenum(VI) Bis(imido) Complexes of the Type $Mo(NR)_2Cl_2$(1,2-dimethoxyethane)

Harold H. Fox, Kimo B. Yap, Jennifer Robbins, Shi'ang Cai, and Richard R. Schrock*

Received December 10, 1991

Introduction

Imido ligands have been widely used to stabilize high oxidation state metal centers and to provide steric protection about the metal center;[1] the latter is an important property of imido complexes that is not true of analogous oxo complexes. We have employed bis((2,6-diisopropylphenyl)imido) complexes of molybdenum[2,3] and tungsten[4,5] as part of a scheme in which metathetically active complexes of the type M(CH-*t*-Bu)(N-2,6-C_6H_3-*i*-Pr_2)$(OR)_2$ are prepared. We also have prepared a variety of rhenium,[6–13] osmium,[14,15] and ruthenium[16] (2,6-diisopropylphenyl)imido complexes. Several 2,6-disubstituted arylimido complexes have been reported from other laboratories recently.[17,18] *tert*-Butylimido ligands have been used much more commonly than 2,6-disubstituted arylimido ligands to stabilize high oxidation state transition metal complexes,[1] but in at least two cases species that contain 2,6-disubstituted arylimido ligands have been synthesized that have no *tert*-butylimido analogues.[13,15]

Certain imido complexes can be prepared most simply by adding a primary amine to a metal–oxo complex, water being the byproduct. For example, addition of *tert*-butylamine to a solution of osmium tetraoxide affords OsO_3(N-*t*-Bu), the first reported imido complex, in up to 90% yield.[19–21] Imido complexes also can be prepared by treating an oxo complex with an isocyanate[5,6,18,22,23] or by adding a (trimethylsilyl)amine to a metal oxide or chloride.[24] A method that combines several of these approaches was perfected in order to synthesize $Mo(NAr)_2Cl_2$(DME) (Ar = 2,6-diisopropylphenyl, DME = 1,2-dimethoxyethane), i.e., adding $ArNH_2$, Me_3SiCl, and 2,6-lutidine to a solution of $MoO_2Cl_2(THF)_2$.[2] 2,6-Lutidine and Me_3SiCl can be viewed most simply as agents that remove HCl and water generated during the reaction, although they may also have more complex roles. The analogous tungsten complex has been prepared in a similar manner from $WOCl_4$.[5] Molybdenum(VI) dioxide dichloride also has been used by other researchers to prepare bis(imido) complexes from isocyanates.[18,23] We report here new syntheses of molybdenum(VI) bis(imido) complexes that employ inexpensive, air-stable $(NH_4)_2Mo_2O_7$, that are easily carried out, and that are quantitative in several cases.

Results and Discussion

Treatment of $(NH_4)_2Mo_2O_7$ with 8 equiv of triethylamine, an excess of chlorotrimethylsilane (~17 equiv), and 4 equiv of a substituted aniline (eq 1) at ~65 °C for 6–10 h in DME under

---

(1) Nugent, W. A.; Mayer, J. M. *Metal-Ligand Multiple Bonds*; Wiley: New York, 1988.

(2) Schrock, R. R.; Murdzek, J. S.; Bazan, G. C.; Robbins, J.; DiMare, M.; O'Regan, M. *J. Am. Chem. Soc.* **1990**, *112*, 3875.

(3) Robbins, J.; Bazan, G. C.; Murdzek, J. S.; O'Regan, M. B.; Schrock, R. R. *Organometallics* **1991**, *10*, 2902.

(4) Schrock, R. R.; DePue, R.; Feldman, J.; Schaverien, C. J.; Dewan, J. C.; Liu, A. H. *J. Am. Chem. Soc.* **1988**, *110*, 1423.

(5) Schrock, R. R.; DePue, R. T.; Feldman, J.; Yap, K. B.; Yang, D. C.; Davis, W. M.; Park, L. Y.; DiMare, M.; Schofield, M.; Anhaus, J.; Walborsky, E.; Evitt, E.; Krüger, C.; Betz, P. *Organometallics* **1990**, *9*, 2262.

(6) Horton, A. D.; Schrock, R. R. *Polyhedron* **1988**, *7*, 1841.

(7) Horton, A. D.; Schrock, R. R.; Freudenberger, J. H. *Organometallics* **1987**, *6*, 893.

(8) Toreki, R.; Schrock, R. R.; Davis, W. M. *J. Am. Chem. Soc.* **1992**, *114*, 3367.

(9) Edwards, D. S.; Biondi, L. V.; Ziller, J. W.; Churchill, M. R.; Schrock, R. R. *Organometallics* **1983**, *2*, 1505.

(10) Weinstock, I. A.; Schrock, R. R.; Davis, W. M. *J. Am. Chem. Soc.* **1991**, *112*, 135.

(11) Weinstock, I. A.; Schrock, R. R.; Williams, D. S.; Crowe, W. E. *Organometallics* **1991**, *9*, 1.

(12) Schofield, M. H.; Schrock, R. R.; Park, L. Y. *Organometallics* **1991**, *10*, 1844.

(13) Williams, D. S.; Schofield, M. H.; Schrock, R. R.; Davis, W. M. *J. Am. Chem. Soc.* **1991**, *113*, 5480.

(14) Anhaus, J. T.; Kee, T. P.; Schofield, M. H.; Schrock, R. R. *J. Am. Chem. Soc.* **1990**, *112*, 1642.

(15) Schofield, M. H.; Kee, T. P.; Anhaus, J. T.; Schrock, R. R. *Inorg. Chem.* **1991**, *30*, 3595.

(16) Kee, T. P.; Park, L. Y.; Robbins, J.; Schrock, R. R. *J. Chem. Soc., Chem. Commun.* **1991**, 121.

(17) Yuan-Wei, C.; Rogers, P. M.; Wigley, D. E.; Alexander, S. J.; Rheingold, A. L. *J. Am. Chem. Soc.* **1991**, *113*, 6326.

(18) Bryson, N.; Youinou, M.-T.; Osborn, J. A. *Organometallics* **1991**, *10*, 3389.

(19) Chong, A. O.; Oshima, K.; Sharpless, K. B. *J. Am. Chem. Soc.* **1977**, *99*, 3420.

(20) Sharpless, K. B.; Patrick, D. W.; Truesdale, L. K.; Biller, S. A. *J. Am. Chem. Soc.* **1975**, *97*, 2305.

(21) Clifford, A. F.; Kobayashi, C. S. *Inorg. Synth.* **1960**, *6*, 207.

(22) Kolomnikov, I. S.; Koreshkov, Yu. D.; Lobeeva, T. S.; Volpin, M. E. *J. Chem. Soc., Chem. Commun.* **1970**, 1432.

(23) Ehrenfeld, D.; Kress, J.; Moore, B. D.; Osborn, J. A.; Schoettel, G. *J. Chem. Soc., Chem. Commun.* **1987**, 129.

(24) Nugent, W. A. *Inorg. Chem.* **1983**, *22*, 965.

0020-1669/92/1331-2287$03.00/0