

plexes to serve as models for Tb(nucleic acid) complexes is evident in the similarity of their spectral properties. The ordering properties associated with guanine residues provide the unique environment that leads to the observation of a strong Tb(III) excitation feature between 290 and 310 nm, the exact energy of which depends on an inner filter effect that is related to base absorption. Other nucleotide bases do not order in the same fashion and hence do not provide the necessary environment required to intensify the  ${}^7F_6 \rightarrow {}^5H_4$  absorption.

A final point concerns the fact that in simple Tb(nucleotide) complexes one may observe conventional energy transfer from the aromatic residues to the Tb(III) ion,<sup>10</sup> while in the Tb-(EDTA)(nucleotide) compounds such transfer is not observed. Such a situation would arise if the stereochemical constraints of the ternary complex were such that Forster-type energy transfer could not occur. For example, a 90° rotation of the aromatic residue could result in an unfavorable geometry for which the energy-transfer process could not take place. We are probing such possibilities at the present time through chiroptical techniques. It is clear, however, that the nature of the intrinsic Tb(III) excitation bands does not depend on this latter geometrical constraint.

**Acknowledgment.** This work was supported by the Camille and Henry Dreyfus Foundation (through a Teacher-Scholar Award to H.G.B.) and by the Samuel Roberts Noble Foundation.

(23) On leave from the Department of Chemistry, University of Trieste, 34127 Trieste, Italy.

Department of Chemistry  
Seton Hall University  
South Orange, New Jersey 07079

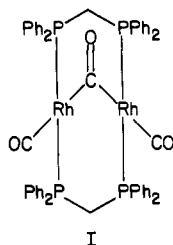
Allen Rudman  
Sergio Paoletti<sup>23</sup>  
Harry G. Brittain\*

Received October 8, 1984

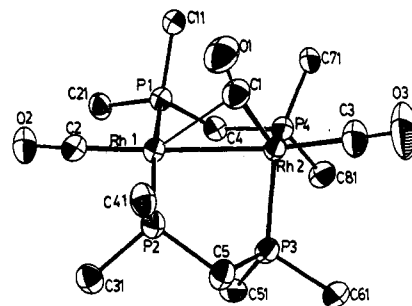
### Structural Characterization of the Non-A-frame Complex Tricarbonylbis(bis(diphenylphosphino)methane)dirhodium, $Rh_2(CO)_3(dppm)_2$

Sir:

The complex  $Rh_2(CO)_3(dppm)_2$  (**1**; dppm = bis(diphenylphosphino)methane), was first reported in 1980<sup>1</sup> and was assigned the A-frame structure I on the basis of spectroscopic data and

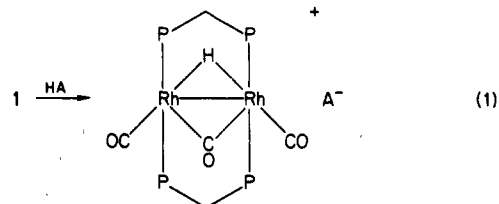


reaction chemistry. The spectroscopic data included carbonyl stretches at 1940, 1920, and 1835  $cm^{-1}$  and a  ${}^{31}P$  NMR spectrum that showed a complex but symmetric pattern, indicating equivalence of the phosphorus donor ligands. The spectrum was characteristic of other  $Rh_2P_4$  A-frame structures<sup>2</sup> and symmetric  $M_2X_4$  systems in general.<sup>3,4</sup> In addition, protonation of **1** led to



**Figure 1.** Perspective view of the inner coordination geometry of  $[Rh_2(CO)_3(dppm)_2]$  (**1**) in which the phenyl rings have been omitted.

formation of  $[Rh_2(\mu-H)(\mu-CO)(CO)_2(dppm)_2]^+A^-$  (eq 1), which was characterized by an X-ray structural study.<sup>1,5</sup>



An interesting feature of the spectroscopic data reported for **1** was the observation of only a single resonance for the methylene protons of the dppm ligand. This observation contrasted with  ${}^1H$  NMR spectra usually seen for A-frame molecules in which two separate resonances for the dppm methylene protons occur<sup>2</sup> and suggested that **1** was fluxional. In the present paper, we report that complex **1** is indeed fluxional and, more importantly, that it possesses a distinctly non-A-frame structure.

The complex  $Rh_2(CO)_3(dppm)_2$  (**1**) was prepared and characterized as previously described.<sup>1,5</sup> NMR data were recorded on a Bruker WH-400 spectrometer at 400 MHz for  ${}^1H$  and at 162 MHz for  ${}^{31}P$  with samples for the  ${}^1H$  and  ${}^{31}P$  NMR experiments in 5- and 10-mm tubes, respectively, flame sealed under vacuum or CO. Dark red-orange crystals suitable for a single-crystal X-ray study were grown from a saturated acetone- $d_6$  solution of the complex.<sup>6</sup> The X-ray intensity data were corrected for Lorentz, polarization, and background effects, after which an empirical absorption correction ( $\mu_{Mo} = 8.75 cm^{-1}$ ) was applied. The structure was solved by conventional heavy-atom techniques. In the final refinements, all non-hydrogen atoms were refined according to an anisotropic thermal model and all hydrogens were placed in fixed positions, assuming  $d(C-H)$  of 0.95 Å and fixed isotropic thermal parameters of 6.0 Å<sup>2</sup>. This refinement of 559 variables and 5363 observations led to convergence with  $R$  and  $R'$  of 0.030 and 0.042, respectively. The final difference Fourier revealed no significant features, with the highest residual peak being 0.65  $e/\text{Å}^3$ . The values of the atomic scattering factors used in the calculations were taken from the usual tabulations, and the effects of anomalous dispersion were included.<sup>7</sup> Tables of final refined positional and anisotropic thermal parameters, observed

(5) Kubiak, C. P.; Woodcock, C.; Eisenberg, R. *Inorg. Chem.* **1982**, *21*, 2119.

(6) Crystallographic data for  $[Rh_2(CO)_3(dppm)_2]$  (**1**): fw 1058.6; monoclinic space group  $P2_1/a$ ;  $a = 19.070$  (5) Å;  $b = 12.145$  (3) Å;  $c = 20.223$  (4) Å;  $\beta$ , 94.88 (2)°;  $V = 4656$  Å<sup>3</sup>;  $Z = 4$ ; calcd density 1.51  $g cm^{-3}$ ; temp 23 °C; cryst dims 0.64 × 0.43 × 0.29 mm. Data for intensity measurements: Enraf-Nonius CAD4 diffractometer; Mo  $K\alpha$  ( $\lambda = 0.71073$  Å); graphite crystal monochromator;  $\omega-2\theta$  scan technique for  $4^\circ \leq 2\theta \leq 46^\circ$ ; octants measured,  $\pm h, +k, +l$ ; final no. of parameters varied, 559; no. of reflections used in least-squares refinement ( $I > 3\sigma(I)$ ), 5363;  $p = 0.05$ ;  $R = 0.0304$ ;  $R_w = 0.0420$ ; goodness of fit, 1.633. All calculations were carried out on a PDP 11/23 computer using the Enraf-Nonius CAD 4-SDP programs and TEXRAY version of Johnson's ORTEP plotting program. In data collection, equivalent reflections other than  $\pm h, k, 0$  were not measured. The  $\pm h, k, 0$  reflections were averaged prior to structural refinements.

(7) Cromer, D. T.; Waber, J. T. "International Tables for X-ray Crystallography"; Kynoch Press: Birmingham, England, 1974; Vol. IV, Table 2.2A. Cromer, D. T. *Ibid.* Table 2.3.1.

(1) Kubiak, C. P.; Eisenberg, R. *J. Am. Chem. Soc.* **1980**, *102*, 3637.

(2) Balch, A. In "Homogeneous Catalysis with Metal Phosphine Complexes"; Pignolet, L. H., Ed.; Plenum Press: New York, 1984; Chapter 4.

(3) Cooper, S. J.; Brown, M. P.; Puddephatt, R. *J. Inorg. Chem.* **1981**, *20*, 1374.

(4) Grim, O. S.; Mitchell, J. D. *Inorg. Chem.* **1977**, *16*, 1770.

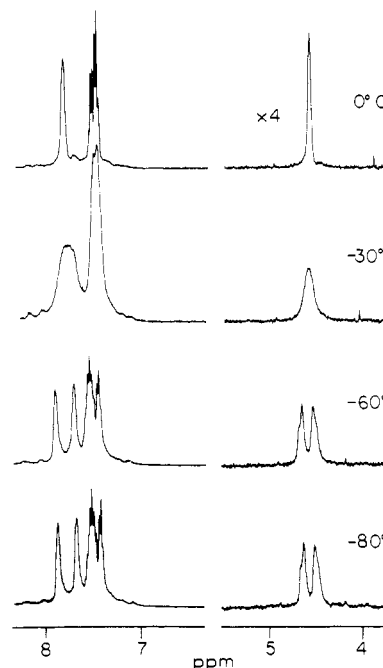
and calculated structure factors, and complete bond distances and angles are available. See the paragraph at the end of the paper regarding supplementary material.

**Solid-State Structure of  $\text{Rh}_2(\text{CO})_3(\text{dppm})_2$  (1).** The crystal structure is composed of discrete molecules of 1. Contrary to expectations, the molecular structure is distinctly non-A-frame. The structure consists of two Rh atoms of different coordination numbers linked by a single Rh–Rh bond of length 2.739 (1) Å and bridging dppm ligands. The coordination geometry about Rh1 is square planar (CN = 4), with Rh2 occupying one of the coordination sites. Also coordinated to Rh1 are a terminal CO trans to Rh2 (C2–Rh1–Rh2 = 177.1 (1)°) and two P donor atoms of the dppm bridges (P1–Rh1–P2 = 166.72 (3)°). The second rhodium atom, Rh2, has CN = 5 and a coordination geometry best described as a distorted trigonal bipyramid. Two P donors and one CO ligand occupy the equatorial positions of the trigonal bipyramid, while the remaining CO ligand and Rh1 are located in axial positions (Rh1–Rh2–C3 = 161.4 (1)°). A perspective view of complex 1 is shown in Figure 1, and selected distances and angles are given below.<sup>8</sup>

In addition to the unexpected unsymmetrical coordination environments of the two rhodium atoms, the structure possesses several unusual features. The first involves the carbonyl ligand C1–O1, which may be described as weakly semibridging. The extent of the semibridging interaction is evidenced by the Rh1...C1 distance of 2.533 (3) Å as compared with the Rh2–C1 distance of 1.857 (3) Å and the Rh2–C1–O1 angle of 167.8 (3)°. These values are comparable to those reported for other complexes containing grossly unsymmetrical carbonyl bridges such as  $\text{Cp}(\text{CO})_3\text{V1–V2Cp}(\text{CO})_2$  and  $(\text{bpy})(\text{CO})_2\text{Fe1–Fe2}(\text{CO})_5$ .<sup>9,10</sup> While a bridging interaction thus appears to exist, the metrical parameters about each Rh and the distances and angles involving C1–O1 suggest that 1 is best viewed as a complex containing a coordinatively unsaturated rhodium atom covalently linked to a saturated rhodium center (formally a 16e–18e complex).

The semibridging nature of the CO ligand in 1 contrasts with that found in other  $\mu$ -CO complexes containing dppm and dpam bridges. For example, the Pt and Pd complexes  $\text{M}_2\text{Cl}_2(\mu\text{-CO})(\text{dpam})_2$  (2) possess symmetrical CO bridges that are unsupported by direct metal–metal bonds and exhibit relatively low  $\nu_{\text{CO}}$  values (respective values for M...M, M–C–M, and  $\nu_{\text{CO}}$  are 3.274 Å, 119°, and 1723  $\text{cm}^{-1}$  for M = Pd<sup>11</sup> and 3.162 Å, 106°, and 1638  $\text{cm}^{-1}$  for M = Pt<sup>12</sup>). These values indicate substantial reduction of bond order in the carbonyl bridges of 2 compared with the semibridging CO of 1.

The second unusual feature of the structure involves the cis disposition of the two dppm P donors in the coordination sphere of Rh2 and the consequent twisting of the eight-membered  $\text{M}_2\text{P}_4\text{C}_2$  ring. The cis orientation of the dppm P's at Rh2 makes the structure differ significantly from dppm-bridged A-frame systems in which the P donors at each metal occupy trans positions. While unusual, this arrangement is not unprecedented. For the compounds  $[\text{Rh}_2(\text{CO})\text{Cl}_2(\text{P}(\text{OPh})_2\text{N}(\text{Et})\text{P}(\text{OPh})_2)_2]$  (3)<sup>13</sup> and



**Figure 2.** Variable-temperature  $^1\text{H}$  NMR spectra of  $[\text{Rh}_2(\text{CO})_3(\text{dppm})_2]$  (1) in acetone- $d_6$  under vacuum.

$[\text{Pt}_2\text{Me}_3(\text{dppm})_2]^+$  (4),<sup>14</sup> bridging phosphine donors are in trans positions at one metal center and cis positions at the other. The Rh–Rh bond in 3 is 2.661 (2) Å and corresponds to a covalent bond between formal Rh(0) and Rh(II), whereas in 1 both Rh atoms are formally Rh(0), with a significantly longer single bond of 2.739 (1) Å.

**Variable-Temperature NMR Studies of  $\text{Rh}_2(\text{CO})_3(\text{dppm})_2$  (1) under Vacuum.** At 30 and at 0 °C, the  $^1\text{H}$  NMR spectrum of 1 exhibits single resonances for both the dppm  $-\text{CH}_2-$  protons (4.60 ppm) and the dppm  $\alpha$ -phenyl protons (7.75 ppm). However, below ca.  $-30$  °C each of these resonances splits into two resonances of equal size and shape (4.65 and 4.55 ppm for  $-\text{CH}_2-$  and 7.84 and 7.66 ppm for  $\alpha$ -phenyl) as shown in Figure 2. The exchange rate constant at  $-30$  °C (coalescence temperature) is calculated to be 160  $\text{s}^{-1}$  by using the relationship  $k_{\text{ex}} = (\pi/(2^{1/2})\Delta\nu)$ , where  $\Delta\nu$  is the peak separation at  $-80$  °C. The free energy of coalescence  $\Delta G_c^*$  at  $-30$  °C is thus calculated to be 12 kcal/mol on the basis of the relationship  $\Delta G^* = 2.3 RT_c (10.32 + \log(T_c/k_{\text{ex}}))$ .<sup>15</sup> The  $^1\text{H}$  NMR spectrum at  $-80$  °C is intermediate between the high-temperature spectrum at 0 °C and the anticipated low-temperature limiting spectrum for 1 consistent with the solid-state structure that possesses four different environments for dppm methylene protons. Thus, a lower activation energy process must also be occurring, but it is unobserved down to  $-80$  °C.

At room temperature the  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of 1 is a complex but symmetric pattern centered at 20.4 ppm. A single P environment is indicated by the pattern, which is non-first-order as discussed by Balch, Puddephatt, Mague, and others.<sup>2,3,4,16</sup> The  $^{31}\text{P}$  NMR spectrum of 1, which remains unchanged to 0 °C, broadens below  $-30$  °C. However, the resonance does not split down to  $-80$  °C. It thus appears that the fluxional process seen in the variable-temperature  $^1\text{H}$  NMR spectrum occurs faster than the frequency difference of the nonequivalent phosphorus nuclei of the solid-state structure even at  $-80$  °C.

Several specific mechanisms to explain the solution dynamic behavior of 1 can be proposed. These include (a) an intramolecular rearrangement in which an A-frame structure similar to that

(8) Selected bond distances (Å) for  $\text{Rh}_2(\text{CO})_3(\text{dppm})_2$  (1): Rh1–Rh2 = 2.739 (0); Rh1–P1 = 2.296 (1); Rh1–P2 = 2.304 (1); Rh1–C1 = 2.533 (3); Rh1–C2 = 1.842 (3); Rh2–P3 = 2.308 (1); Rh2–P4 = 2.332 (1); Rh2–C1 = 1.857 (3); Rh2–C3 = 1.881 (4). Selected bond angles (deg): Rh2–Rh1–P1 = 88.35 (2); Rh2–Rh1–P2 = 88.56 (2); Rh2–Rh1–C1 = 177.1 (1); P1–Rh1–P2 = 166.72 (3); P1–Rh1–C1 = 96.48 (8); P2–Rh1–C2 = 92.0 (1); P2–Rh1–C1 = 89.63 (8); P2–Rh1–C2 = 91.7 (1); Rh1–Rh2–P3 = 84.73 (2); Rh1–Rh2–P4 = 94.80 (2); Rh1–Rh2–C1 = 63.5 (1); Rh1–Rh2–C3 = 161.4 (1); P3–Rh2–P4 = 104.39 (3); P3–Rh2–C1 = 125.4 (1); P3–Rh2–C3 = 100.5 (1); P4–Rh2–C1 = 120.6 (1); P4–Rh2–C3 = 101.1 (1); C1–Rh2–C3 = 99.6 (2); Rh1–C1–Rh2 = 75.5 (1); Rh1–C1–O1 = 116.8 (2); Rh2–C1–O1 = 167.8 (3).

(9) Cotton, F. A.; Frenz, B. A.; Kruczynski, L. *J. Am. Chem. Soc.* **1973**, *95*, 951.

(10) Cotton, F. A.; Troup, J. M. *J. Am. Chem. Soc.* **1974**, *96*, 1233.

(11) Colton, R.; McCormick, M. J.; Pannan, C. D. *Aust. J. Chem.* **1978**, *31*, 1425.

(12) Brown, M. P.; Keith, A. N.; Manojlovic-Muir, Lj.; Muir, K. W.; Puddephatt, R. J.; Seddon, K. R. *Inorg. Chim. Acta* **1979**, *34*, L2223.

(13) Haines, R. J.; Meintjies, E.; Laing, M. *Inorg. Chim. Acta* **1979**, *36*, L403.

(14) Brown, M. P.; Cooper, S. J.; Frew, A. A.; Manojlovic-Muir, Lj.; Muir, K. W.; Puddephatt, R. J.; Seddon, K. R.; Thomson, M. A. *Inorg. Chem.* **1981**, *20*, 1500.

(15) Binsch, G.; Kessler, H. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 411.

(16) Mague, J. T.; Sanger, A. R. *Inorg. Chem.* **1979**, *18*, 2060.

originally proposed becomes an intermediate, (b) a dissociative mechanism in which CO is lost from 1 to form the intermediate  $\text{Rh}_2(\text{CO})_2(\text{dppm})_2$ , and then recoordinates on the opposite face of this metal-metal-bonded species, (c) an associative process in which a tetracarbonyl species acts as an intermediate, and (d) a process involving dppm dissociation/recombination.

Further studies using  $^{13}\text{C}$  NMR spectroscopy are being undertaken to address this question and to determine conclusively whether all carbonyl ligands are involved in the exchange process. What does seem clear, however, is that the dppm ligands in their bridging mode appear relatively flexible and are not locked into simple trans positions at the metal centers of the binuclear complex.

**Acknowledgment.** We thank the National Science Foundation (Grant CHE83-08064) for partial support of this research and Johnson Matthey and Co. for a generous loan of precious metal salts. We also acknowledge helpful discussions with Professor J. Mague, Professor W. D. Jones, Dr. C. Johnson, and Dr. D. Berry.

Registry No. 1, 74507-92-9.

**Supplementary Material Available:** Listings of refined positional and anisotropic thermal parameters, observed and calculated structure factor amplitudes, and bond distances and angles (65 pages). Ordering information is given on any current masthead page.

Department of Chemistry  
University of Rochester  
Rochester, New York 14627

Carrie Woodcock  
Richard Eisenberg\*

Received November 8, 1984

### Synthesis and Characterization of a Tetranuclear Oxomolybdate Containing Coordinatively Bound Diazenido Units. $[\text{Mo}_4\text{O}_8(\text{OCH}_3)_2(\text{NNC}_6\text{H}_5)_4]^{2-}$ : A Versatile Precursor for the Synthesis of Complexes with the $[\text{Mo}(\text{NNC}_6\text{H}_5)_2]^{2+}$ Unit

Sir:

The recent interest in the coordination chemistry of poly-molybdates with species such as  $\text{CH}_3\text{O}^-$ ,<sup>1</sup>  $\text{HCO}^-$ ,<sup>2</sup> and pyridine<sup>3</sup> is a consequence of the importance of these species in developing an understanding of the modes of interaction of small organic molecules with catalytic oxide surfaces.<sup>4,5</sup> In each of the examples cited above, each molybdenum atom is octahedrally coordinated either exclusively to oxygen donors or to five oxygen donors and a single nitrogen donor.

Since these studies establish the ability of polymolybdates to coordinate to nitrogen donors, we sought to extend this chemistry to the organodiazenido group,  $-\text{N}=\text{N}-$ , a ligand known to form robust complexes with molybdenum, indeed often displacing oxo groups to give the *cis*- $[\text{Mo}(\text{N}=\text{N})_2]^{2+}$  structural unit.<sup>6</sup> Furthermore, the organodiazenido group is of some intrinsic interest as a model for understanding the chemistry of metal-bound dinitrogen and  $\text{NO}$ .<sup>7</sup> The parallel between organodiazenido complexes and those of the diazenide ion,  $\text{HN}=\text{N}^-$ , a potential but inaccessible reduction product of dinitrogen, has also been noted.<sup>8</sup>

- (1) McCarron, E. M., III; Harlow, R. C. *J. Am. Chem. Soc.* **1983**, *105*, 6179.
- (2) Adams, R. D.; Klemperer, W. G.; Liu, R.-S. *J. Chem. Soc., Chem. Commun.* **1979**, 256.
- (3) McCarron, E. M., III; Whitney, J. F.; Chase, D. B., *Inorg. Chem.* **1984**, *23*, 3275.
- (4) Day, V. W.; Fredrick, M. F.; Klemperer, W. G.; Liu, R.-S. *J. Am. Chem. Soc.* **1979**, *101*, 491.
- (5) McCarron, E. M., III; Staley, R. H.; Sleight, A. W. *Inorg. Chem.* **1984**, *23*, 1043.
- (6) Dahlstrom, P. L.; Dilworth, J. R.; Shulman, P.; Zubieta, J. *Inorg. Chem.* **1982**, *21*, 933.
- (7) Sutton, D. *Chem. Soc. Rev.* **1975**, *4*, 443.
- (8) Bishop, M. W.; Butler, G.; Chatt, J.; Dilworth, J. R. *J. Chem. Soc., Dalton Trans.* **1979**, 1843.

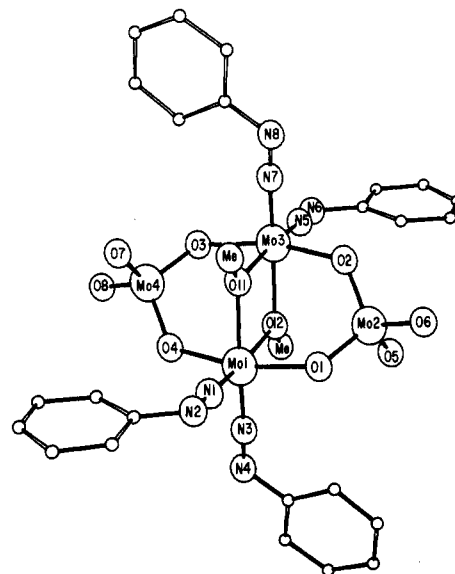


Figure 1. ORTEP view of the tetranuclear anion,  $[\text{Mo}_4\text{O}_8(\text{OCH}_3)_2(\text{NNC}_6\text{H}_5)_4]^{2-}$ , showing the atom-labeling scheme.

We report herein the synthesis and structural characterization of an unusual tetranuclear dianion  $[\text{Mo}_4\text{O}_8(\text{OCH}_3)_2(\text{NNC}_6\text{H}_5)_4]^{2-}$ , a species containing  $[\text{Mo}(\text{NNC}_6\text{H}_5)_2]^{2+}$  units bridged by two methoxy groups and by two  $\text{MoO}_4^{2-}$  units.

Reaction of  $[\text{MoO}_2(\text{butane-2,3-diolate})_2] \cdot 2\text{-butane-2,3-diol}$ <sup>9</sup> with excess phenylhydrazine and  $\text{N}(\text{C}_2\text{H}_5)_3$  in acidified methanol yields lustrous black crystals, analyzing as  $(\text{HNEt}_3)[\text{Mo}_2\text{O}_4(\text{OMe})(\text{NNC}_6\text{H}_5)_2]$ .<sup>10</sup> Conductivity in acetonitrile revealed that the complex behaved as a 2:1 electrolyte. The infrared spectrum showed bands at 1631, 1616, 1574, and 1522  $\text{cm}^{-1}$  that are characteristic of  $\nu(\text{N}-\text{N})$  for *cis* coordination of the diazenido groups in  $[\text{Mo}(\text{N}=\text{N})_2]^{2+}$  units.

An X-ray diffraction study<sup>11</sup> of this complex reveals the tetranuclear structure of the anion (Figure 1). The location of a center of symmetry on the midpoint of the  $\text{Mo1}-\text{Mo3}$  vector imposes planarity on the  $\text{Mo1}-\text{Mo3}-\text{O11}-\text{O12}$  rhombus; in fact, the entire  $[\text{Mo}_2(\text{OCH}_3)_2(\text{NN})_4]$  unit is nearly planar, with maximum deviations of ca. 0.05 Å from the least-squares plane through all atoms. The overall geometry may be viewed as two  $[\text{Mo}(\text{N}=\text{N})_2]^{2+}$  units bridged by two methoxy groups and two  $\text{MoO}_4^{2-}$  units, functioning as bidentate bridging ligands. The  $\text{Mo}_2\text{O}_4$  heterocycle is slightly puckered, with Mo2 below the  $\text{Mo1}-\text{Mo3}-\text{O1}-\text{O2}-\text{O3}-\text{O4}$  plane and with Mo4 somewhat above this plane. The bridging  $\text{MoO}_4^{2-}$  units display tetrahedral geometry about the molybdenum atoms with valence angles in the range 106.3 (3)–113.0 (3)° and average Mo-terminal oxo distances and Mo-bridging oxo distances of 1.716 (9) and 1.798 (9) Å, respectively. The diazenido-coordinated molybdenum atoms

- (9) Butcher, R. J.; Penfold, B. R. *J. Cryst. Mol. Struct.* **1976**, *6*, 1.
- (10) Synthesis of  $(\text{NHEt}_3)_2[\text{Mo}_4\text{O}_8(\text{OMe})_2(\text{NNC}_6\text{H}_5)_4]$ : A mixture of phenylhydrazine (4.32 g) and triethylamine (8.08 g) in 50 mL of methanol was added to a refluxing solution of  $[\text{MoO}_2(\text{C}_4\text{H}_9\text{O}_2)_2] \cdot 2\text{C}_2\text{H}_5\text{O}_2$  (2.43 g) in methanol/trimethylsilyl chloride (200 mL/1 mL). A deep purple color developed immediately. After refluxing for 0.5 h, the solution was allowed to cool to room temperature whereupon ethyl ether was added until a perceptible turbidity formed. After the mixture was allowed to stand for 1 week, lustrous black crystals were observed. No special precautions were required as the reaction appears to be neither air nor moisture sensitive. Anal. Calcd for  $\text{C}_{38}\text{H}_{58}\text{Mo}_4\text{N}_{10}\text{O}_{10}$ : C, 38.1; H, 4.84; N, 11.7. Found: C, 37.7; H, 4.79; N, 11.8. Yield: 20–30%.
- (11) Crystal data for  $\text{C}_{38}\text{H}_{58}\text{Mo}_4\text{N}_{10}\text{O}_{10}$ : space group triclinic  $P\bar{1}$ ,  $a = 12.326$  (3) Å,  $b = 12.799$  (3) Å,  $c = 16.190$  (3) Å,  $\alpha = 86.56$  (1)°,  $\beta = 83.94$  (1)°,  $\gamma = 83.67$  (1)°,  $V = 2521$  (1) Å<sup>3</sup>,  $Z = 2$ . The X-ray data were collected in a Nicolet R3m diffractometer, Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å. The structure was solved by using Patterson and difference Fourier methods using local versions of the SHELXTL program package. Final anisotropic refinement for all non-carbon and non-hydrogen atoms, based on 4243 independent reflections with  $I_o \geq 3\sigma(I_o)$ , converged at residual of 0.050, with a goodness of fit of 1.48.