

B021 PREPARATION AND CHARACTERIZATION OF SOME HIGH OXIDATION STATE DINITROGEN COMPLEXES OF TUNGSTEN. M. O'Regan, R. R. Schrock and W. M. Davis, Department of Chemistry, M.I.T., Cambridge, Ma 02139, U.S.A.

$[\text{Cp}^*\text{WMe}_3]_2(\mu\text{-N}_2)$ reacts with $\text{C}_6\text{F}_5\text{COOH}$, $\text{C}_6\text{F}_5\text{OH}$ and $\text{C}_6\text{F}_5\text{SH}$ to give complexes of the form $[\text{Cp}^*\text{WMe}_2(\text{X})]_2(\mu\text{-N}_2)$, $[\text{X}=\text{OC}_6\text{F}_5, \text{etc.}]$.

$[\text{Cp}^*\text{WMe}_3]_2(\mu\text{-N}_2)$ also reacts with tetrabromocatechol to give $[\text{Cp}^*\text{WMe}(\text{O}_2\text{C}_6\text{Br}_4)]_2(\mu\text{-N}_2)$. Another related complex may be prepared by the reaction of $[\text{Cp}^*\text{WMe}_2(\text{OTf})]_2(\mu\text{-N}_2)$ with $\text{NaS-2,4,6-(CH}_3)_3\text{C}_6\text{H}_2$. X-ray structures of $[\text{Cp}^*\text{WMe}_2(\text{OC}_6\text{F}_5)]_2(\mu\text{-N}_2)$ and $[\text{Cp}^*\text{WMe}_2(\text{SMes})]_2(\mu\text{-N}_2)$ show them to have similar structures to that of $[\text{Cp}^*\text{WMe}_3]_2(\mu\text{-N}_2)$ except the N-N bond lengths are shorter: 1.22(1)Å and 1.27(8)Å vs. 1.334(26)Å.

^{15}N NMR spectra are reported for all these complexes.

B022 MoFe COFACTOR OF NITROGENASE: EXTRACTION, PURIFICATION, AND CHARACTERIZATION. A. Hickman, P. McLean¹, T. Salifoglou, D. Wink², D. Wright, D. Coucouvanis³ and W.H. Orme-Johnson. Department of Chemistry, M.I.T., Cambridge, MA 02139, USA; ¹, Biotechnica, Inc.; ², N.I.H.; ³, University of Michigan.

We have recently reported [1] that the MoFe cofactor from the MoFe protein component of nitrogenase can be extracted from protein adsorbed on DEAE cellulose in dimethyl formamide and other non-aqueous solvents, by displacement with organic solvent soluble salts such as $\phi_4\text{AsCl}$. The resultant solutions can be freed of non-cofactor iron and desalted in subsequent chromatographic steps, yielding solutions of highly active cofactor on which meaningful elemental analyses are feasible. We will report the results of such characterization as well as experiments aimed at growing diffraction-grade crystals by anaerobic vapor diffusion methods and spectroscopic studies on cofactor and derivatives.

[1] Wink et al., *Biochemistry* (1989) in press; McLean et al., *ibid* (1989) in press.

B023 PREPARATION AND CHARACTERIZATION OF A MOLYBDOSULFENAMIDE
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A primary molybdosulfenamide containing the $\text{Mo}_2(\mu\text{-SNH}_2)$ unit has been prepared. Treatment of this compound with excess thioacid gives ammonium ion immediately. The amide function bound to a bridge sulfur provides an interesting alternative to a metal-bound amide step during nitrogenase action.