However, the metal–metal distance of 2.989 (2)Å is well within bonding range, unlike the tungsten parent but like $(\mu\text{-H})Fe_2(CO)_8^-$. The *ORTEP* plots of Figure 1a,b indicate strict octahedral coordination geometry about tungsten and a distorted trigonal-bipyramidal geometry about iron. The M–M bond and (presumed) bridging hydride serves to complete the coordination spheres of each metal. In contrast to the TBP geometry of $HFe(CO)_4^-$ in which all equatorial C–Fe–C angles are near 120°,[13] the C7–Fe–C8 angle of **1c**$^-$ has opened to 147.6(8)° and is staggered with respect to the same position as is C2 and C3 on the W centers. The position about which this stagger occurs is the most probable location for the H^- ion. Preliminary results indicate that the location of the H^- ion will prove successful; however, what presently appears as a slight disordering of the carbonyls on the W prevents its positive identification at the current level of refinement ($R = 0.059$). Its projected position is just off the Fe–W bond axis. In view of the similarity of the $(OC)_{ax}$–Fe–$(CO)_{eq}$ bond angles in **1c**$^-$ to those of $HFe(CO)_4^-$ (ca. 100° in both) it is tempting to assume that the hydride is more closely associated with the Fe than the W and that $HFe(CO)_4^-$ is serving as a unit ligand to $W(CO)_5{}^0$. Indeed *most* of the chemical reactions presented below are supportive of this notion. However, we have also noted one major difference in the reactivity of $HFe(CO)_4^-$ as derived from **1c**$^-$ vs. that of pure $HFe(CO)_4^-$ (vide infra). Hence further speculation on this structure is declined at this time.[14]

The dimer disrupts in the presence of added ligands, eq 2–4

$$\mathbf{1c}^- \xrightarrow{CO} HFe(CO)_4^- + W(CO)_6 \quad (2)$$

$$\mathbf{1c}^- \xrightarrow{PPh_3} HFe(CO)_4^- + Ph_3PW(CO)_5 \quad (3)$$

$$\mathbf{1c}^- \xrightarrow{HW(CO)_5^-} HFe(CO)_4^- + (\mu\text{-H})W_2(CO)_{10}^- \quad (4)$$

(THF solvent). When the anion is allowed to stand in THF solution at room temperature, eq 5, the same products as in eq

$$\mathbf{1c}^- \xrightarrow[dec]{\Delta} HFe(CO)_4^- + W(CO)_6 \quad (5)$$

2 are obtained, presumably by the standard decomposition pathway as a variety of labile $LW(CO)_5$ species.[15] In all cases of dimer disruption the H^- ligand consistently remained with $HFe(CO)_4^-$. Attempts were made to force the opposite heterolytic cleavage yielding $HW(CO)_5^-$ and $[Fe(CO)_4{}^0]$. For example, it is known that CO_2 inserts into W–H bonds of $HW(CO)_5^-$ (but not easily into $HFe(CO)_4^-$)[16] to yield $(OC)_5WOC(O)H^-$.[17] Also $W(CO)_5$·THF is trapped by $HW(CO)_5^-$ at time of mixing to yield the very stable $(\mu\text{-H})W_2(CO)_{10}^-$. Nevertheless neither CO_2 nor $W(CO)_5$·THF reacted with **1c**$^-$.

The dimer cleavage reactions (3) and (4) proceeded at the same rate with half-lives of ca. 5–6 h, measured at $[\mathbf{1c}^-] = 0.0048$ M, $[PPh_3] = 0.10$ M, and $[HW(CO)_5^-] = 0.0048$ M. The decomposition reaction (5) was slower ($t_{1/2} \sim$ days), because the initial products of cleavage, $HFe(CO)_4^-$ and $W(CO)_5{}^0$, recombine (eq 1). Unexpectedly, in the presence of CO-saturated THF, [CO] estimated at 0.01 to 0.02 M,[18] **1c**$^-$ had a half-life of ca. 10 min (eq 2). Furthermore in the presence of both CO (~0.02 M) and PPh_3 (0.01 M) a reaction equally rapid as (2) occurs; however, the product distribution (85% $W(CO)_5PPh_3$ and 15% $W(CO)_6$) is the same as a similar mixture of CO, PPh_3, and photochemically generated THF·$W(CO)_5$. This suggests that the CO that promotes the dimer cleavage does not necessarily trap the coordinatively unsaturated species produced! A reaction pathway consistent with this result is shown in eq 6.

$$(\mu\text{-H})FeW(CO)_9^- + L \rightleftharpoons [(\mu\text{-H})FeW(CO)_9^-\cdot L] \longrightarrow LW(CO)_5 + HFe(CO)_4^- \quad (6)$$
$$\downarrow +L' \qquad\qquad +$$
$$L'W(CO)_5 + HFe(CO)_4^-$$

In contrast to both parent dimers, $(\mu\text{-H})W_2(CO)_{10}^-$ and $(\mu\text{-H})Fe_2(CO)_8^-$, **1c**$^-$ is highly CO labile. When ^{13}CO is used in reaction 2 *both* products are found to be highly enriched. Extensive ligand exchange has thus occurred on the intact dimer prior to disruption. The reaction described by eq 7 was run in an

$$HFe(CO)_4^- + W(^{13}CO)_5\cdot THF \xrightarrow{\text{room temperature, THF}} (\mu\text{-H})[Fe(^*CO)_4][W(^*CO)_5]^- \quad (7)$$

* = mixture of ^{13}CO and ^{12}CO

attempt to generate $(\mu\text{-H})[Fe(^{12}CO)_4][W^{13}CO)_5]^-$. However, both the ν(CO) IR spectrum, taken within 5 min of mixing, and the 23 °C ^{13}C NMR spectrum, recorded within a few hours of synthesis, indicated that the label was equally distributed over the Fe and W centers. Hence it is impossible to tell whether the CO exchange is specific for one metal center.

In conclusion, the structure of a newly synthesized heterobimetallic, $(\mu\text{-H})FeW(CO)_9^-$, has been determined and found to be intermediate between that of its homobimetallic parents. The dimer heterolytically disrupts with the hydride ligand remaining on the iron center. The new anion shows reactivity with CO remarkably different from both parent molecules. The reason for this reactivity difference is currently under investigation.

Acknowledgment. We gratefully acknowledge the financial support of the National Science Foundation (CHE 83-04162) and the donors of the Petroleum Research Fund, administered by the American Chemical Society. The assistance of Dr. S. C. Kao is greatly appreciated.

Registry No. $PPN^+\mathbf{1a}^-$, 88326-11-8; $PPN^+\mathbf{1b}^-$, 88326-13-0; $PPN^+\mathbf{1c}^-$, 88326-15-2; $HFe(CO)_4^-$, 18716-80-8; THF·$Cr(CO)_5$, 15038-41-2; THF·$Mo(CO)_5$, 53248-43-4; THF·$W(CO)_5$, 36477-75-5; $HW(CO)_5^-$, 77227-36-2; CO, 630-08-0; PPh_3, 603-35-0; ^{13}CO, 1641-69-6.

(13) Smith, M. B.; Bau, R. *J. Am. Chem. Soc.* **1973**, *95*, 2388.
(14) The X-ray crystal structures of $PPh_4^+(\mu\text{-H})FeW(CO)_9^-$ and of $PPh_4^+(\mu\text{-H})FeCr(CO)_9^-$ are under investigation. Large crystals of the latter, hopefully suitable for neutron diffraction analysis, have been obtained.
(15) Hyde, C. L.; Darensbourg, D. J. *Inorg. Chem.* **1973**, *12*, 1286.
(16) Evans, G. O.; Newell, C. J. *Inorg. Chim. Acta* **1978**, *31*, L387.
(17) Darenbourg, D. J.; Rokicki, A.; Darensbourg, M. Y. *J. Am. Chem. Soc.* **1981**, *103*, 3223.
(18) We have estimated the concentration of CO in a CO-saturated THF solution at 22 °C to be similar to that of acetone (8.8×10^{-3} M at 20 °C) or diethyl ether (1.6×10^{-2} M at 0 °C) whose values were taken from: "International Critical Tables"; 1933: Vol. III, p 265.

Heterocycles in Synthesis: Chiral Amino Acids/Dipeptides via a Novel Photooxidative Cleavage of Trisubstituted Imidazoles

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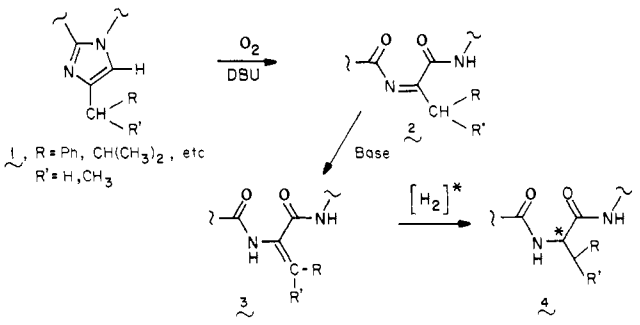
Although heteroaromatic nuclei have demonstrated potential to serve as latent functional group equivalents,[1] use of the imidazole ring in this capacity, to our knowledge, is without precedent.[2] We envisioned a general, asymmetric synthesis of N-protected amino

(1) Some recent examples: (a) Furans:Van Royen, L. A.; Mijngheer, R.; De Clerq, P. J. *Tetrahedron Lett.* **1983**, *24*, 3145. (b) Isoxazoles: L'abbe, G.; Godts, F.; Toppet, S. *Ibid.* **1983**, *24*, 3149. (c) Oxazoles: Wasserman, H. H.; Lu, T.-J. *Ibid.* **1982**, *23*, 3831 and references therein. Jacobi, P. A.; Walker, D. G. *J. Am. Chem. Soc.* **1981**, *103*, 4611. Levin, J. I.; Weinreb, S. M. *Ibid.* **1983**, *105*, 1397. (d) Thiazoles: Biskupiak, J. E.; Ireland, C. M. *J. Org. Chem.* **1983**, *48*, 2302. (e) Groth, U., Schollkopf, U. *Synthesis* **1983**, 37.
(2) Grimmett, M. R. *Adv. Heterocycl. Chem.* **1980**, *27*, 241. Grimmett, M. R. *Ibid.* **1970**, *12*, 103.

0002-7863/84/1506-0457$01.50/0

Scheme I

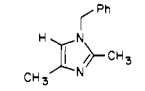
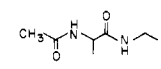
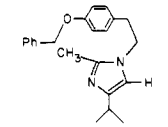
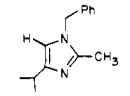
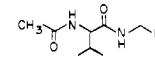
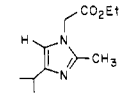
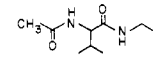
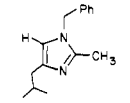
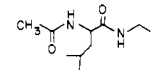
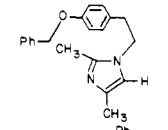
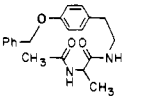
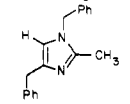
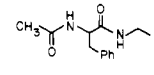


acids/dipeptides via appropriately substituted imidazoles, with potential applications to natural products chemistry, e.g., the cyclopeptide alkaloids.[3,4] 1,4-Dicarbonyl insertion through the agency of singlet molecular oxygen followed by imine to enamine isomerization[5] and olefin hydrogenation would lead to the desired array, as illustrated in Scheme I.

As the sequence proceeds through a dehydro amino acid, chirality might be induced under the influence of a rhodium salt/chiral phosphine complex as hydrogenation catalyst.[6] Herein we describe this novel photooxidative conversion of 1,2,4-trisubstituted imidazoles to both diamides and dipeptides. Furthermore, it is demonstrated that chiral products can indeed be realized in excellent overall chemical and optical yields.

Previous singlet oxygenations on somewhat related substrates, predominantly by Wasserman and Wolff,[7] when carried out under protic conditions afforded products resulting from solvent participation. Nonetheless, their contributions of fundamental importance established that imidazoles are prone toward behaving as azabutadienes in a Diels–Alder-like cycloaddition scheme. Our procedure, which appears to be quite general, calls for exposure of an imidazole **1** as educt to photochemically generated singlet oxygen[8] in dry THF containing ca. 1–2 equiv of DBU[9] with hematoporphyrin as sensitizer. Following consumption of starting material, rapid filtration through silica gel removes both the base and sensitizer affording the imine diamide **2** in >94% yield. Isomerization with KO-*t*-Bu in THF/*t*-BuOH at room temperature leads to the corresponding enamines **3** (77–85%). Alternatively, the initial photoadduct could be used without purification in the next step to give the enamine diamides directly in 75–83% overall yield. Our results are summarized in Table I. Interestingly, only the 4-methylimidazole cases (i.e., latent alanine equivalents, entries 1 and 6) provided the isomerized material under the conditions of photooxidation. Hydrogenation of the so-formed dehydro amino acids could be effected using traditional catlaysts (Pd/C, PtO_2) under pressure (8–22 psi) essentially quantitatively. More significantly, however, optically active derivatives **4** were readily obtained, the extent of induction varying as a function of the chiral phosphine employed.[10] Hence, while

Table I. Photooxidation/Isomerization/Reduction of 1,2,4-Trisubstituted Imidazoles in THF with DBU

entry	imidazole 1	conditions ($^1O_2$)	reduction (%)[a] 4	% ee[b]
1		0°, 1.25 h	 (100)[c] (95)[d] (100)[e]	– 59 92
2		23°, 2.4 h	—[f]	–
3		0°, 2.15 h	 (97)[c]	–
4		23°, 2.5 h	 (97)[g]	–
5		0°, 2.0 h	 (100)[d] (98)[e]	43 95
6		23°, 3.25 h	 (97)[e]	88
7		23°, 3.1 h	 (98)[e]	96

[a] Isolated yields of purified products. All new compounds gave satisfactory IR, NMR, MS, and exact mass or combustion analysis data. [b] Determined by ^1H NMR at 300 MHz using $Eu(hfc)_3$ as shift reagent. [c] Using H_2, Pd/C. [d] Using H_2, $(Rh(NBD)Cl)_2$, (*S,S*)-DIOP. [e] Using H_2, $(Rh(NBD)Cl)_2$-(*R,R*)-DIPAMP. [f] Dehydrovaline portion could not be selectively reduced in the presence of the benzyl ether. [g] Using H_2, PtO_2.

the catalyst prepared from $(Rh(NBD)Cl)_2$-(+)-(*S,S*)-DIOP (MeOH, room temperature) led to only 43–59% ee (entries 1 and 5),[11] substitution of (*R,R*)-DIPAMP[12] as the bidentate ligand gave excellent results (88–96% ee).[13]

In cases where the substituent at the 4-position cannot isomerize, as in the example below, hydride reduction is a particularly facile process. Opportunities exist, therefore, for potential chiral induction using either a chiral auxiliary on nitrogen or a chiral source of hydride.[14]

In summary, amino acid diamides, including those of alanine, valine, leucine, and phenylalanine, have been secured by means

(3) (a) Lipshutz, B. H.; Hungate, R. W.; McCarthy, K. E. *J. Am. Chem. Soc.*, in press. (b) Lipshutz, B. H.; McCarthy, K. E.; Hungate, R. W. *Tetrahedron Lett.* in press. (c) Lipshutz, B. H.; Morey, M. C. *J. Org. Chem.* **1983**, *48*, 3745.

(4) For general reviews on the phenylcyclopeptines, see: (a) Bycroft, B. W.; Wels, C. M. *Amino-Acids, Pept., Proteins* **1976**, *8*, 310. (b) Tschesche, R.; Kauszman, E. U. *Alkaloids (N.Y.)* **1955**, *15*, 165. (c) Pais, M., Jarreau, F.-X. *Chem. Biochem. Amino Acids, Pept., Proteins* **1971**, *1*, 127. (d) Warnhoff, E. M. *Alkaloids (London)* **1971**, *1*, 444.

(5) Poisel, H.; Schmidt, U. *Chem. Ber.* **1975**, *108*, 2547. Poisel, H. *Ibid.* **1977**, *110*, 948.

(6) For a review, see: Halpern, J. *Science (Washington, D.C.)* **1982**, *217*, 401.

(7) Wasserman, H. H.; Wolff, M. S. In "Singlet Oxygen"; Wasserman, H. H., Murray, R. W., Eds.; Academic Press: New York, 1979; Chapter 9, p 481. Wasserman, H. H.; Ives, J. L. *Tetrahedron* **1981**, *37*, 1825.

(8) Oxygen was bubbled through the reaction solution onto which was directed a 250-W floodlamp.

(9) Without DBU present, the reaction takes a considerably different course, see ref 3c.

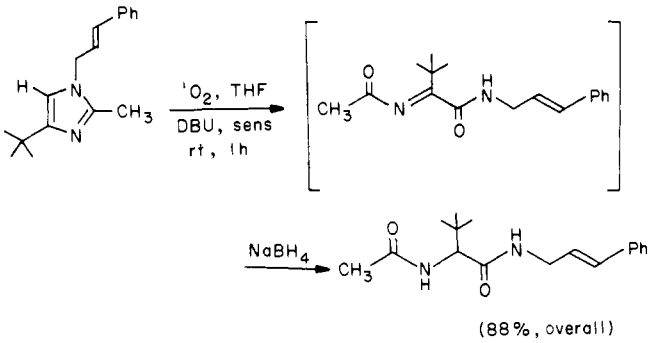
(10) For leading references, see: (a) Brown, J. M.; Parker, D. *Organometallics* **1982**, *1*, 950. (b) Brown, J. M.; Parker, D. *J. Org. Chem.* **1982**, *47*, 2722. (c) Brown, J. M.; Chaloner, P. A. *J. Chem. Soc., Perkin Trans. 2* **1982**, 711. (d) Achiwa, K.; Chaloner, P. A.; Parker, D. *J. Organomet. Chem.* **1981**, *218*, 249. (e) Brown, J. A.; Chaloner, P. A.; Kent, A. G.; Murrer, B. A.; Nicholson, P. N.; Parker, D.; Sidebottom, P. J. *Ibid.* **1981**, *216*, 263. (f) Ojima, I.; Yoda, N. *Tetrahedron Lett.* **1982**, *23*, 3913. (g) Meyer, D.; Poulin, J.-C.; Kagan, H. B.; Levine-Pinto, H.; Morgat, *J.-L.*; Fromageot, P. *J. Org. Chem.* **1980**, *45*, 4680. (h) Scott, J. W.; Keith, D. D.; Nix, G.; Parrish, D. R.; Remington, S.; Roth, G. P.; Townsend, J. M.; Valentine, D.% Yang, R. *Ibid.* **1981**, *46*, 5086. (i) MacNeil, P. A.; Roberts, N. K.; Bosnich, B. *J. Am. Chem. Soc.* **1981**, *103*, 2273.

(11) (*S,S*)-DIOP is well-known to give products of the *S* configuration at the newly generated chiral center.[6,10h]

(12) We are indebted to Dr. W. S. Knowles (Monsanto) for supplying the (*R,R*)-DIPAMP used in this study.

(13) (*R,R*)-DIPAMP has been shown to afford products containing a new chiral center of the *S* configuration.[6,10h] This was confirmed by spectral comparison with an authentic sample of the phenylalanine bis-amide (*S* configuration; Table I, entry 7).

(14) For example, see: Nishizawa, M.; Noyori, R. *Tetrahedron Lett.* **1980**, *21*, 2821 and references therein.



of a new and efficient photooxidative transformation of imidazoles. Curiously, excess DBU is required, in spite of the well-known ability of amines to efficiently quench singlet oxygen.[15] Both diamides and dipeptides (cf. entry 4) may be unmasked using this three-step protocol. Chiral products with high ee's result from hydrogenations using an optically active Rh(I)/DIPAMP combination as catalyst. Application of this technology[16] to the generation of α-alkylated amino acids,[17] as well as the phencyclopeptines,[4] will be reported in due course.

Acknowledgment. Financial support provided by the National Institutes of Health (GM 28128) is gratefully acknowledged. We thank Dr. H. Webb for obtaining the mass spectral data and Dr. W. S. Knowles (Monsanto) for providing a generous supply of (*R,R*)-DIPAMP.

Registry No. 2,4-Dimethyl-1-(phenylmethyl)-1*H*-imidazole, 52726-31-5; 2-methyl-4-(1-methylethyl)-1-[2-[4-(phenylmethyl)phenyl]-ethyl]-1*H*-imidazole, 88336-71-4; 2-methyl-4-(1-methylethyl)-1-(phenylmethyl)-1*H*-imidazole, 54416-18-1; ethyl 2-methyl-4-(1-methylethyl)-1*H*-imidazole-1-acetic acid, 88336-72-5; 2-methyl-4-(2-methylpropyl)-1-(phenylmethyl)-1*H*-imidazole, 88336-73-6; 2,4-dimethyl-1-[2-[4-(phenylmethoxy)phenyl]ethyl]-1*H*-imidazole, 86921-46-2; 2-methyl-1,4-bis(phenylmethyl)-1*H*-imidazole, 88336-74-7; 2-(acetylamino)-*N*-(phenylmethyl)propanamide, 86921-48-4; 2-(acetylamino)-3-methyl-*N*-(phenylmethyl)butanamide, 88336-75-8; 2-(acetylamino)-3-methyl-*N*-[(ethoxycarbonyl)methyl]butanamide, 88336-76-9; 2-(acetylamino)-4-methyl-*N*-(phenylmethyl)pentanamide, 88336-77-0; 2-(acetylamino-*N*-[2-[4-(phenylmethoxy)phenyl]ethyl]propanamide, 88336-78-1; 2-(acetylamino)-*N*-(phenylmethyl)benzenepropanamide, 67509-08-4.

Supplementary Material Available: IR, NMR, mass spectral and high-resolution MS analytical and physical constants for selected intermediates **2**, **3**, and **4** (3 pages). Ordering information is given on any current masthead page.

(15) Foote, C. S. In "Singlet Oxygen"; Wasserman, H. H., Murray, R. W., Eds.; Academic Press: New York, 1979; Chapter 5. The appropriate control reactions (i.e., i, no light; ii, no sensitizer) clearly established that singlet oxygen is the reactive reagent.

(16) A typical procedure for the photooxidation/isomerization/hydrogenation sequence is illustrated using 1-benzyl-2-methyl-4-isobutylimidazole (Table I, entry 5). A solution of the imidazole (217 mg, 0.95 mmol) in 10 mL of dry THF containing 121 μL (1.44 mmol) of DBU and ca. 5 mg of hematoporphyrin is fitted with a gas dispersion tube through which dry oxygen is continuously passed.[18] The solution is cooled with a stream of tap water (20–25 °C) and irradiated externally with a 275-W sunlamp for 2.25 h. Following consumption of starting material (as monitored by TLC) the mixture was concentrated in vacuo, diluted with 5 mL of dry THF, and placed under Ar. To the crude imine (**2**, R = $CH(CH_3)_2$; R′ = H) dissolved in 5 mL of THF was added a solution of KH (108 mg, 35% in oil, 1.0 mmol) and 136 mg (2.0 mmol) of *tert*-butyl alcohol in 5 mL of dry THF. The mixture was maintained at room temperature until complete by TLC. Extractive workup (EtOAc) followed by drying ($MgSO_4$) and chromatography on SiO_2 (1:1 EtOAc/Et_2O) gave 205 mg (83%) of the *N*-acetyldehydroleucine *N*-benzylamide as a white solid, mp 124–126 °C. A flask charged with 13.7 mg (0.053 mmol) of the dehydroamino acid diamide and 3 mL of degassed MeOH is placed under 1 atm of H_2. A solution of ca. 1 mg of Rh(NBD)Cl dimer and ca. 0.5 mg of (*R,R*)-DIPAMP in 2 mL of degassed MeOH is prehydrogenated (1 atm) for 10 min. The resulting solution is then syringed into the dehydrodiamide in MeOH and monitored by TLC over time. Upon completion, the mixture was concentrated in vacuo, diluted with 3 mL of dry THF, and filtered through a short plug of SiO_2 affording 13.6 mg (98%) of the desired material. The enantiomeric excess of 95% in the above mixture was determined via Eu(hfc)$_3$-perturbed[19] 300-MHz NMR, by measuring the relative peak heights of the acetyl methyl and/or the benzyl singlets, both of which were easily resolved and viewed using a stacked plot program.

(17) For a recent approach to these materials, see: Seebach, D., Boes, M., Naef, R., Schweizer, W. B. *J. Am. Chem. Soc.* **1983**, *105*, 5390.

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Induced Internal Redox Processes in Molybdenum–Sulfur Chemistry: Conversion of MoS_4^{2-} to $Mo_2S_8^{2-}$ by Organic Disulfides

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In molybdenum–sulfur chemistry redox usually involves either changes in the metal oxidation state[1,2] or in a few cases changes in the oxidation state of the sulfur ligands.[3,4] However, in some complex reactions[5-7] both Mo- and S-based redox occur. In this paper, we report and rationalize a simple albeit counterintuitive reaction in which both molybdenum and sulfur change their oxidation states.

The tetrathiomolybdate ion, MoS_4^{2-}, has molybdenum in its maximal hexavalent oxidation state. We find that this ion can nevertheless be readily *oxidized* by organic disulfides to give the dinuclear complex, $Mo_2S_8^{2-}$, containing *reduced* pentavalent molybdenum. This stoichiometrically simple reaction involves induced internal electron transfer[8] from sulfide to molybdenum, and its documentation and implications are the subjects of this communication.

The stoichiometry of reaction I has been established to be

$$2MoS_4^{2-} + RSSR \rightarrow Mo_2S_8^{2-} + 2RS^- \quad (I)$$

Reaction of 2 equiv of $(NH_4)_2MoS_4$ with 1 equiv of $C_6H_5SSC_6H_5$ in DMF at 90 °C for ~1 h results in the formation of $Mo_2S_8^{2-}$. The anion has been isolated in >80% yield as the $P(C_6H_5)_4^+$ salt by precipitation with diethyl ether/isopropyl alcohol. The organic product was detected and quantitated as RSH by reversed-phase HPLC.[9] The conversion of MoS_4^{2-} to $Mo_2S_8^{2-}$ can also be effected at 90 °C by $C_6H_5SeSeC_6H_5$ or at room temperature with p-$O_2NC_6H_4SSC_6H_4$-p-NO_2. Salts of $Mo_2S_8^{2-}$ display analytical[10] and spectral[11] data consistent with their formulation. The product is identical with one formed in good yield by the reaction of $Mo_2(S_2)_6^{2-}$ with 8 equiv of thiolate.[4]

Single crystals suitable for X-ray diffraction were obtained by vapor diffusion of diethyl ether into an acetonitrile solution of

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(9) Reverse-phase HPLC, Altex Ultrasphere ODS column, 4.6 mm × 15 cm, 1:1 MeOH/CH_3CN (1% acetic acid), 1 mL min^{-1}, UV–vis detection (250 nm).
(10) For the $[N(C_2H_5)_4]_2Mo_2S_8$ complex analysis for $C_{16}H_{40}N_2Mo_2S_8$ gives, calcd, C, 27.1, H, 5.69, N, 3.95, found, C, 27.1, H, 5.46, N, 3.74.
(11) Infrared spectra in KBr disk of $[N(C_2H_5)_4]_2Mo_2S_8$ shows bands at 520, 535, 460, and 350 cm^{-1} assignable to molybdenum–sulfur vibrational modes. The electronic spectrum in CH_3CN displays bands at 573 (ϵ2590), 467 (2130), and 295 nm.

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