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Isolation and Characterization of Isothiocyanato Complexes of Molybdenum(II) Containing Strong Metal–Metal Bonds

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Although salts of the octaisothiocyanatodirhenate(III) anion, $Re_2(NCS)_8^{2-}$, have been isolated,[1,2] their reactions with tertiary phosphines do not yield metal–metal bonded complexes of the types $Re_2(NCS)_6(PR_3)_2$ and $Re_2(NCS)_4(PR_3)_4$. Instead, the magnetically dilute thiocyanate bridged dimers $(Bu_4N)_2Re_2(NCS)_8(PR_3)_2$ are formed.[2] This behavior is in marked contrast to the ease with which salts of the octahalodirhenate(III) anions, $Re_2X_8^{2-}$ (X = Cl, Br, or I), afford the complexes $Re_2X_6(PR_3)_2$ and/or $Re_2X_4(PR_3)_4$.[3,4] The mild conditions under which the Re–Re quadruple bond of $Re_2(NCS)_8^{2-}$ is cleaved led us to suggest[2] that in this anion the metal–metal bond is weaker than that in the related octahalodirhenate(III) anions, a conclusion which was supported by differences in the electronic absorption spectra of these species.[2]

In order to understand more fully those factors which favor this reaction course, we felt it was important to establish whether those metal–metal bonded dimers which are electronically related to $Re_2(NCS)_8^{2-}$ are inherently unstable or whether this behavior is restricted to the rhenium(III) dimer and is therefore a property of the Re–Re bond. Accordingly, we have investigated the possibility of isolating metal–metal bonded molybdenum(II) dimers containing coordinated thiocyanate ligands.

During the last 10 years or so, many complexes containing the binuclear metal–metal bonded species Mo_2^{4+} have been synthesized.[5-13] There is ample evidence to show that the Mo_2^{4+} unit, which contains a quadruple metal–metal bond,[14] can survive substitution reactions, representative examples being the preparations of $Mo_2Cl_8^{4-}$ from $Mo_2(O_2CCH_3)_4$[8] and $Mo_2Cl_4(PR_3)_4$ from $Mo_2Cl_8^{4-}$.[12] Prior to the present investigation, there have been no reports dealing with thiocyanate complexes of Mo_2^{4+}.

Experimental Section

Starting Material. The preparation of the complex $Mo_2Cl_4(PEt_3)_4$ was achieved using the previously described procedure.[12] All solvents and other starting materials were obtained from commercial sources.

Reaction Procedures. All reactions were carried out in a nitrogen atmosphere, and solvents were deoxygenated by purging with N_2 gas for several hours.

A. Reactions of $Mo_2Cl_4(PEt_3)_4$ with Potassium Thiocyanate. (i) $Mo_2(NCS)_4(PEt_3)_4$ (I). A mixture of $Mo_2Cl_4(PEt_3)_4$ (1.0 g) and KSCN (0.8 g) in 40 mL of methanol was refluxed for at least 15 h. The green reaction mixture was then allowed to cool to room temperature. The resulting dark green crystalline solid was filtered off, washed with water and small portions of methanol and petroleum ether, and then dried in vacuo for several hours. A second crop of I was collected when the volume of the filtrate was reduced to ~10 mL by rotary evaporation; yield 0.60 g; mp 195–200 °C (dec). Anal. Calcd for $C_{28}H_{60}Mo_2N_4P_4S_4$: C, 37.52; H, 6.69; N, 6.25; S, 14.31; mol wt, 896. Found: C, 37.22; H, 6.54; N, 6.41; S, 14.50; mol wt (acetone), 910. Compound I is very soluble in toluene, acetone, acetonitrile, and tetrahydrofuran and slightly soluble in methanol and ether.

(ii) $(Bu_4N)_2Mo_2(NCS)_6(PEt_3)_2$ (II). The green filtrate obtained in (i) was placed in a 50 mL round bottom flask equipped with a magnetic stirrer. The flask was capped with a rubber serum cap and then flushed with nitrogen. A solution of Bu_4NBr (0.8 g) in 10 mL of methanol was added to the contents of the flask by means of a syringe. After stirring this mixture for 4 h at room temperature, the resulting solution was concentrated by rotary evaporation. The royal blue solid II which precipitated was filtered off and washed with water to remove undissolved KSCN, with toluene to remove any contamination from I, and finally with small portions of methanol and petroleum ether before being dried in vacuo; yield 0.23 g; mp 140 °C (dec). This complex can be recrystallized from acetone and ether. Anal. Calcd for $C_{50}H_{102}Mo_2N_8P_2S_6$: C, 47.62; H, 8.09; N, 8.89; S, 15.24. Found: C, 47.58; H, 8.03; N, 8.87; S, 15.22. This salt is very soluble in acetone, acetonitrile, and tetrahydrofuran but is insoluble in toluene.

An alternative procedure which may be used for the isolation of II is as follows. The reaction mixture from (ii) was evaporated to dryness and the resulting solid dissolved in a small volume of acetone. It was then filtered to remove any insoluble KSCN, and petroleum ether was added slowly to the clear filtrate until it became cloudy. Crystals of II appeared upon keeping this mixture under an atmosphere of nitrogen at 0 °C overnight.

(iii) The Interconversion of I and II. A mixture of I (0.08 g) and KSCN (0.05 g) in 8 mL of methanol was refluxed for 2 h followed by stirring the resulting solution with Bu_4NBr (0.15 g) for an additional hour. The royal blue complex II separated upon concentrating the reaction solution. It was filtered off and washed with toluene and petroleum ether. Similarly, the dark green complex I was isolated upon refluxing II (0.05 g) with PEt_3 (0.2 mL) in 7 mL of methanol for 12 h.

B. Reactions of $Mo_2(NCS)_4(PEt_3)_4$ with 1,2-Bis(diphenylphosphino)ethane, Bis(diphenylphosphino)methane, 2,2′-Bipyridyl, and 1,10-Phenanthroline. The compounds $Mo_2(NCS)_4(L–L)_2$ (where L–L is a bidentate ligand) were generally prepared by refluxing $Mo_2(NCS)_4(PEt_3)_4$ with an excess of ligand in methanol or absolute ethanol for at least 20 h. The resulting precipitates were collected and washed with toluene to remove any unreacted molybdenum starting material, then with methanol or ethanol, and finally with diethyl ether. The products were allowed to dry in vacuo for 5 h prior to analysis.

(i) $Mo_2(NCS)_4(dppe)_2$ (III). A mixture of 0.1 g of I and 0.13 g of 1,2-bis(diphenylphosphino)ethane (dppe) in 13 mL of methanol or ethanol was refluxed for 24 h. The yellow crystalline solid was filtered off and treated as above; yield 0.09 g. Recrystallization can be accomplished by dissolution in a small volume of dichloromethane followed by the addition of ethanol. Anal. Calcd for $C_{56}H_{48}Mo_2N_4P_4S_4$: C, 55.09; H, 3.96; N, 4.59. Found: C, 54.86; H, 4.01; N, 4.42. This complex is soluble in dichloromethane, slightly soluble in acetone, but insoluble in most other organic solvents.

Compound III can also be obtained from the reaction of II with dppe. However, attempts to synthesize it by reacting KSCN with α-$Mo_2Cl_4(dppe)_2$[13] were unsuccessful. The reaction of III with excess PEt_3 in refluxing methanol failed to give I.

(ii) $Mo_2(NCS)_4(dppm)_2$ (IV). This complex was obtained as a crop of dark green crystals, greenish yellow when powdered, when I (0.09 g) and bis(diphenylphosphino)methane (dppm) (0.13 g) were reacted in 15 mL of ethanol under reflux conditions for 30 h; yield 0.06 g. Anal. Calcd for $C_{54}H_{44}Mo_2N_4P_4S_4$: C, 54.37; H, 3.72; N, 4.70; mol wt, 1193. Found: C, 53.25; H, 4.00; N, 4.78; mol wt, 1150 (acetone). The solubility of IV is noticeably different from that of III. Thus, in addition to being soluble in dichloromethane, it also dissolves readily in acetone and acetonitrile, and it is slightly soluble in methanol.

(iii) $Mo_2(NCS)_4(bpy)_2$ (V). Refluxing I (0.11 g) with 2,2′-bipyridyl (bpy) (0.12 g) in 15 mL of methanol afforded the insoluble aqua green complex V; yield 0.07 g. Anal. Calcd for $C_{24}H_{16}Mo_2N_8S_4$: C, 39.14; H, 2.17; N, 15.21. Found: C, 38.88; H, 2.34; N, 14.91. This complex is insoluble in most organic solvents.

(iv) $Mo_2(NCS)_4(phen)_2$ (VI). The reaction of I (0.11 g) with 1,10-phenanthroline (phen) (0.12 g) in 15 mL of ethanol yielded the dark green complex; yield 0.10 g. Anal. Calcd for $C_{28}H_{16}Mo_2N_8S_4$: C, 42.86; H, 2.06; N, 14.28. Found: C, 43.28; H, 1.88; N, 13.71. This complex exhibits the same lack of solubility as V.

C. Stability of the Molybdenum(II) Thiocyanate Complexes. The molybdenum thiocyanate complexes reported herein appear to be

Table I. Spectral Properties of Isothiocyanato Complexes of Molybdenum(II)

Complex	Medium[a]	Electronic absorption maxima (2000–400 nm)	Infrared spectra ν(C–N), cm^{-1}	Mo $3d_{3/2,5/2}$ binding energies, eV[b,c]
$Mo_2(NCS)_4(PEt_3)_2$ (I)	A	683 s, 500 m, 395 s	2048 s, br, 1990 sh	232.2, 229.2 (1.7)
	B	690 s, 500 m, 400 s		
$(Bu_4N)_2Mo_2(NCS)_6(PEt_3)_2$ (II)	A	682 s, 520 vw, sh, 460 w, 415 sh	2080 s, br	~232.0, 228.6 (1.4)
	B	685 s, 515 vw, sh, 460 w, 410 sh		
$Mo_2(NCS)_4(dppe)_2$ (III)	A	830 s, 625 vw, sh?, 518 sh, 445 s	2028 s	232.2, 229.1 (1.3)
$Mo_2(NCS)_4(dppm)_2$ (IV)	A	750 s, 710 sh, 530 m–w, 435 s	2040 s, 2030 sh, 2020 sh	232.3, 229.2 (1.6)
$Mo_2(NCS)_4(bpy)_2$ (V)	A	655 s, 565 sh?, 425 sh	2090 s, 2060 s	231.7, 228.6 (1.3)
$Mo_2(NCS)_4(phen)_2$ (VI)	A	660 s, 570 sh?, 425 sh	2065 s, 2040 s	231.7, 228.7 (1.6)

[a] A = diffuse reflectance; B = Nujol mull. [b] Full-width half-maximum values for the $3d_{5/2}$ component are given in parentheses; binding energies are referenced to the carbon 1s binding energy of 285.0 eV for the phosphine or amine ligands. [c] N 1s, S 2p, and P 2p binding energies were also recorded and were found to correspond very closely with the related data for mixed isothiocyanato–tertiary phosphine and amine complexes of rhenium(III) (see ref 2).

moderately air and temperature sensitive (especially compounds I, II, and IV). This is evident by their discoloration if they are kept in air at room temperature for about 2 days. The infrared spectra of the decomposed compounds show an intense band at 960 cm^{-1}, characteristic of ν(Mo=O), which is not present in the freshly prepared products. To inhibit their decomposition, these complexes should either be kept under vacuum or under a nitrogen atmosphere at temperatures below 10 °C.

Physical Measurements. These were recorded using the instrumentation previously described.[2]

Results and Discussion

Although our attempts to prepare salts of the $Mo_2(NCS)_8^{4-}$ anion by the reactions of $K_4Mo_2Cl_8$ and $Mo_2(O_2CCH_3)_4$ with KSCN and NH_4SCN were unsuccessful, nonetheless, we have been able to isolate derivatives of this anion. Thus, the reaction of the triethylphosphine complex $Mo_2Cl_4(PEt_3)_4$[11,12] with KSCN in refluxing methanol affords $Mo_2(NCS)_4(PEt_3)_4$ (compound I) together with a solution containing the $Mo_2(NCS)_6(PEt_3)_2^{2-}$ anion. The latter species may be isolated as its *n*-butylammonium salt (compound II). The close structural relationship between I and II is shown by their interconvertibility (eq 1).

$$Mo_2(NCS)_4(PEt_3)_4 \underset{PEt_3}{\overset{SCN^-/Bu_4N^+}{\rightleftarrows}} (Bu_4N)_2Mo_2(NCS)_6(PEt_3)_2 \quad (1)$$

The dimeric nature of I was confirmed by a molecular weight determination in acetone. Conductivity data for acetonitrile solutions of these two complexes support their formulation:[15] I, $\Lambda_m = 6.5\ \Omega^{-1}$ cm^2 for $c_m = 4.91 \times 10^{-4}$ M; II, $\Lambda_m = 262\ \Omega^{-1}$ cm^2 for $c_m = 2.86 \times 10^{-4}$ M. Magnetic susceptibility measurements on II show that it possesses only weak paramagnetism ($\chi_g = -0.14 \times 10^{-6}$ cgsu at 300 K).

The complex $Mo_2(NCS)_4(PEt_3)_4$ may itself be used as a starting material for the synthesis of complexes of the type $Mo_2(NCS)_4(L\text{–}L)_2$, where L–L represents a bidentate ligand. Displacement of the PEt_3 ligands by 1,2-bis(diphenylphosphino)ethane (dppe), bis(diphenylphosphino)methane (dppm), 2,2′-bipyridyl (bpy), and 1,10-phenanthroline (phen) occurs readily in refluxing methanol or ethanol. The dppm complex is the most soluble one of the four, and a molecular weight measurement in acetone confirmed its dimeric formulation.

The spectral properties of the new complexes (I–VI) are summarized in Table I. In the absence of x-ray crystallographic data, infrared spectroscopy can often be used to infer the mode of bonding of the ambidentate thiocyanate ligand.[16,17] The ν(C–N) modes of complexes I–VI (Table I) occur in a frequency range (2090–1990 cm^{-1}) which is usually considered to be characteristic of N-bonded thiocyanate.[16,17] A comparison of the infrared spectrum of $Mo_2(NCS)_4(PEt_3)_4$ with that of the related chloro complex $Mo_2Cl_4(PEt_3)_4$ shows that the ν(C–S) and δ(N–C–S) modes occur at 858 (s) and 485 (m) cm^{-1}, respectively. Bands at 842 (m) and 485 (m) cm^{-1} in the related infrared spectrum of II are probably due to these same vibrations. These observations are once again in agreement with Mo–NCS rather than Mo–SCN.[16,17] Unfortunately, the unambiguous assignment of the ν(C–S) and δ(N–C–S) for the other complexes listed in Table I is complicated by the occurrence of L–L modes in the appropriate spectral region. However, a band which is present at 490–480 cm^{-1} in the spectra of all these thiocyanate complexes is most likely assigned to δ(N–C–S). Thus, the available infrared spectroscopic evidence supports these complexes as being isothiocyanato derivatives.

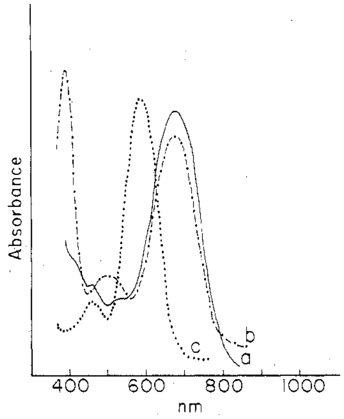

Figure 1. The diffuse reflectance spectra (1200–400 nm) of (a) $(Bu_4N)_2Mo_2(NCS)_6(PEt_3)_2$, (b) $Mo_2(NCS)_4(PEt_3)_4$, and (c) $Mo_2Cl_4(PEt_3)_4$.

The x-ray photoelectron spectra (XPS) of compounds I–VI show that they possess Mo $3d_{5/2}$ energies between 229.2 and 228.6 eV (see Table I), a range which is characteristic of molybdenum(II) dimers of the type $Mo_2X_4L_4$.[18] The N 1s binding energy spectra of II, V, and VI each exhibit two peaks: one at ~398 eV due to NCS and one at either 402.3 eV (II) or ~400.0 eV (V and VI) due to $[Bu_4N]^+$, bpy, or phen. The intensity ratios of these two peaks are either 3:1 (II) or 1:1 (V and VI), in accord with the known stoichiometric ratios of different types of nitrogen atoms in these complexes.

The electronic absorption spectra of I and II are very similar (Table I and Figure 1), implying a close structural relationship. The spectra of all the complexes listed in Table I are, like the chloride and bromide derivatives of the types $Mo_2X_4L_4$ and $Mo_2X_4(L\text{–}L)_2$,[10,12,13] dominated by an intense absorption band in the visible region anywhere between 530 and 840 nm. The wavelength of this band is very sensitive to the nature of the ligands.

It has previously been shown[1,2] that the lowest energy absorption band in the spectrum of the $Re_2(NCS)_8^{2-}$ anion (assigned to the $\delta \rightarrow \delta^*$ transition of a species containing the $\sigma^2\pi^4\delta^2$ ground-state configuration)[14] occurs at a much longer wavelength than does the analogous transition in the spectra of $Re_2X_8^{2-}$, where X = Cl or Br.[19-21] A similar trend is observed between the spectra of I, IV, V, and VI (Table I and Figure 1) and their chloride analogues.[10,12,13] In these instances, the lowest energy transition in the spectra of the isothiocyanato complexes exhibits a red shift of 100–135 nm relative to $Mo_2Cl_4(PEt_3)_4$ and $Mo_2Cl_4(L{-}L)_2$, where L–L = dppm, bpy, and phen. The 830-nm absorption of complex III (Table I) is at a lower energy than the corresponding band in the spectra of either α-$Mo_2Cl_4(dppe)_2$ or β-$Mo_2Cl_4(dppe)_2$ (at 675 and 780 nm, respectively).[13] These two molybdenum complexes possess different structures:[13] the α isomer contains chelating dppe molecules, a cis disposition of Mo–Cl bonds, and an eclipsed ligand configuration, while β-$Mo_2Cl_4(dppe)_2$ is believed to have a staggered noncentrosymmetric structure wherein the dppe ligands bridge the molybdenum atoms within the dimer. At the present time we have no definitive evidence as to which (if either) structure is appropriate for $Mo_2(NCS)_4(dppe)_2$. However, in view of the significant shift observed between the lowest energy electronic absorption band of the other isothiocyanato complexes listed in Table I and their chloride analogues (i.e., >100 nm), III may structurally resemble α-$Mo_2Cl_4(dppe)_2$ since here the red shift is 155 nm.

In summary, it is clear that, in contrast to the failure of $Re_2(NCS)_8^{2-}$ to yield derivatives of the type $Re_2(NCS)_6L_2$ via ligand substitution reactions, little difficulty was experienced in isolating the molybdenum complexes $Mo_2(NCS)_4L_4$ and $Mo_2(NCS)_4(L{-}L)_2$. Accordingly, substitution of thiocyanate for halide in species derived from dimers of the type $M_2X_8^{n-}$ does not necessarily lead to cleavage of the metal–metal bond, the actual reaction course being dependent upon the nature of M. In view of our successful isolation of the salt $(Bu_4N)_2Mo_2(NCS)_6(PEt_3)_2$ and the existence of $(Bu_4N)_2Re_2(NCS)_8$,[1,2] it is possible that salts of the octaisothiocyanatodimolybdate(II) anion can be isolated upon choice of a suitable cation.

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Registry No. I, 64784-48-1; II, 64784-50-5; III, 64784-51-6; IV, 64784-41-4; V, 64784-42-5; VI, 64784-43-6; $Mo_2Cl_4(PEt)_4$, 59780-36-8.

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Polarized Absorption Spectroscopy of Anisotropic Single Crystals

Sir:

Polarized absorption spectra of single crystals have been used extensively in recent years in the study of the electronic structure of transition metal complexes. Both linearly polarized and circularly polarized spectra of anisotropic crystals are frequently of interest. A large number of linearly polarized spectra[1] and a significant number of circularly polarized spectra[2] have appeared in the literature.

It is well known that both of these types of spectra are subject to the effects of birefringence and that the circularly polarized spectra are affected by the linear dichroism of the crystal.[3,4] On the other hand, it does not seem to be generally recognized that linearly polarized spectra can be affected by circular dichroism in the crystal.

Unfortunately, there seems to be no readily available general analysis of these possible interferences,[5] and thus the information in such spectra and their limitations have probably not been as clear as they should be. Such a general analysis is fairly simple, when the problem is formulated in terms of the Mueller matrix calculus,[6] and is presented here.

The usual experimental procedure is to place a reasonably thin slice of crystalline material with parallel faces in the appropriate instrument with the crystal faces perpendicular to the propagation vector, k_i, of the incident light beam. Under these conditions, the effect of an anisotropic crystal upon the light beam may be analyzed in a straightforward way in terms of the Stokes vector of the light beam and the Mueller matrix of the crystal.[7]

The Stokes vector is the four-dimensional vector

$$\mathbf{S} = \begin{bmatrix} s_0 \\ s_1 \\ s_2 \\ s_3 \end{bmatrix} \qquad (1)$$

where s_0 is the intensity and the remaining s_i describe the degree and type of polarization. If we consider a "nearly" monochromatic beam in a right-handed coordinate system with the propagation vector, k, in the positive z direction, the Stokes vector becomes[8]

$$\mathbf{S} = \begin{bmatrix} s_0 \\ s_1 \\ s_2 \\ s_3 \end{bmatrix} = \begin{bmatrix} \langle a_x^2 + a_y^2 \rangle \\ \langle 2a_x a_y \cos\delta \rangle \\ \langle 2a_x a_y \sin\delta \rangle \\ \langle a_x^2 - a_y^2 \rangle \end{bmatrix} \qquad (2)$$

where δ is the phase angle between the instantaneous amplitudes of the electric field in the x and y directions, a_x and a_y. δ is defined so that when $\pi > \delta > 0$, circular polarization of the beam is right handed.

The effect of any optical device can be expressed as a 4 × 4 matrix, the Mueller matrix, $\mathbf{F}$, which transforms the Stokes

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