narrow green band, which was moving down the column at a much slower rate than **3**, $R = OCH_3$, $R' = C_2H_5$, was then eluted with benzene. However, as the chromatography continued, the green band disappeared and only additional **3**, $R = OCH_3$, $R' = C_2H_5$, was isolated. The total yield of the mononuclear complex **3**, $R = OCH_3$, $R' = C_2H_5$, was 1.60 g (92%). Elution with benzene–methylene complex chloride (1:1), or pure methylene chloride, gave an unidentified organic compound (IR: $\nu_{P=O}$ 1166 cm^{-1}).

Reaction of 2, R = OCH_3, with Tri-*n*-butyl Phosphite. A mixture of 2.82 g (4.07 mmol) of **2**, $R = OCH_3$, and tri-*n*-butyl phosphite (4.54 g, 18.0 mmol) in benzene was reacted and worked up as described for triethyl phosphite, except that silica gel was used as the chromatographic absorbent. Pure **3**, $R = OCH_3$, $R' = C_4H_9$, was obtained as a crystalline orange solid, yield 1.23 g (57%).

Reaction of 2, R = OCH_3, with Diethoxyphenylphosphine. Diethoxyphenylphosphine (1.50 g, 7.58 mmol) and **2**, $R = OCH_3$ (1.303 g, 1.88 mmol), in benzene (50 mL) were heated for 15 h at 60 °C. After cooling, filtration, and filtrate concentration, the crude mixture was chromatographed on alumina (Fisher activity grade I, 80–200 mesh) with 2:1 hexane–benzene. Elution with 2:1 hexane–benzene gave 0.54 g of impure **3**, $R = OCH_3$, $R' = C_2H_5$ (contaminated with a small quantity of an organic material). The green-brown ligand substitution product **4**, $R = OCH_3$, $L = PhP(OC_2H_5)_2$, was eluted with benzene. Thin-layer chromatography of the latter indicated that it was impure and rechromatography using 1:1 hexane–benzene resulted in the isolation of **3**, $R = OCH_3$, $R' = C_2H_5$, and 0.57 g (35%) of quite pure **4**, $R = OCH_3$, $L = PhP(OC_2H_5)_2$. However, the latter was gradually converted back to **2**, $R = OCH_3$, on standing. Consequently, attempts to obtain satisfactory analyses for **4**, $R = OCH_3$, $L = PhP(OC_2H_5)_2$, were futile.

Pure **3**, $R = OCH_3$, $R' = C_2H_5$ (0.414 g, 44%), was obtained by rechromatography of the crude complex using 3:1 hexane–*commercial* (not rigorously purified) benzene.

Reaction of 2, R = OCH_3, with Ethoxydiphenylphosphine. Complex **2**, $R = OCH_3$ (0.774 g, 1.12 mmol), and ethoxydiphenylphosphine (1.06 g, 4.60 mmol) were reacted and worked up as described for the reaction of the complex with triethyl phosphite. Elution with 1:1 hexane–benzene gave 0.74 g (72%) of **4**, $R = OCH_3$, $L = Ph_2POC_2H_5$. Slow coversion of the latter to **2**, $R = OCH_3$, occurred over a period of weeks.

Reaction of 2, R = OCH_3, with Triphenyl Phosphite. A benzene (60 mL) solution of **2**, $R = OCH_3$ (3.85 g, 5.56 mmol), and triphenyl phosphite (7.09 g, 22.8 mmol) was refluxed for 3 days. The solution was cooled and filtered, and the concentrated filtrate was chromatographed on alumina (EM reagents 70–230 mesh). Elution with benzene afforded recovered starting material, followed by **4**, $R = OCH_3$, $L = (PhO)_3P$. The latter was further purified by crystallization from hexane; yield 0.81 g (15%).

Reaction of 2, R = H, with Triethyl Phosphite. Complex **2**, R = H (1.00 g, 1.58 mmol), and triethyl phosphite (1.24 g, 7.46 mmol) were reacted and worked up as described for the reaction of **2**, $R = OCH_3$, with $(C_2H_5O)_3P$ (except for the substitution of Baker for EM alumina). Elution with 2:1 hexane–benzene afforded 0.53 g (82%) of orange **3**, R = H, $R' = C_2H_5$. A narrow green-brown band was eluted off the column with 5:1 benzene–hexane but was too unstable to be isolated in analytically pure form.

Reaction of 2, R = CH_3, with Triethyl Phosphite. A mixture of triethyl phosphite (0.70 g, 4.2 mmol) and **2**, $R = CH_3$ (1.01 g, 1.53 mmol), in benzene was reacted and worked up as described for the reaction of **2**, $R = OCH_3$, with tri-*n*-butyl phosphite. Elution with 4:1 hexane–benzene gave 0.394 g (55%) of orange **3**, $R = CH_3$, $R' = C_2H_5$. Elution with benzene afforded **4**, $R = CH_3$, $L = (C_2H_5O)_3P$, which required further purification by recrystallization from 5:1 hexane–methylene chloride; yield 0.37 g (29%).

Reaction of 5 with Triethyl Phosphite. The thiocamphor complex **5** (1.67 g, 2.70 mmol) and triethyl phosphite (1.92 g, 11.6 mmol) were reacted and worked up as described for the reaction of **2**, $R = OCH_3$, with triethyl phosphite to give 0.514 g (46%) of **6** and 0.205 g (10%) of **7**.

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Registry No. **2**, $R = OCH_3$, 72186-35-7; **2**, R = H, 72186-34-6; **2**, $R = CH_3$, 72186-31-3; **3**, $R = OCH_3$, $R' = C_2H_5$, 73985-98-5; **3**, $R = OCH_3$, $R' = C_4H_9$, 73985-99-6; **3**, R = H, $R' = C_2H_5$, 73986-00-2; **3**, $R = CH_3$, $R' = C_2H_5$, 73986-01-3; **4**, $R = OCH_3$, $L = PhP(OC_2H_5)_2$, 73986-02-4; **4**, $R = OCH_3$, $L = Ph_2POC_2H_5$, 73986-03-5; **4**, $R = OCH_3$, $L = (PhO)_3P$, 73986-04-6; **4**, $R = CH_3$, $L = (C_2H_5O)_3P$, 73986-05-7; **5**, 72186-45-9; **6**, 73986-41-1; **7**, 73986-40-0; $P(OC_2H_5)_3$, 122-52-1; $P(OC_4H_9)_3$, 102-85-2; $PhP(OC_2H_5)_2$, 1638-86-4; $Ph_2P(OC_2H_5)$, 719-80-2.

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Syntheses and Characterization of $Mo(ONPh)(S_2CNEt_2)_2$, a Valence Isomer of $MoO(NPh)(S_2CNEt_2)_2$

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Nitrobenzene reacts rapidly with $Mo(CO)_2(S_2CNEt_2)_2$ in CH_2Cl_2 to yield $MoO(ONC_6H_5)(S_2CNEt_2)_2$. Reduction of this complex with triphenylphosphine affords triphenylphosphine oxide and $Mo(ONC_6H_5)(S_2CNEt_2)_2$, a compound which also results from the reaction of nitrosobenzene and $Mo(CO)_2(S_2CNEt_2)_2$. NMR evidence points to η^2 coordination of the nitrosobenzene in this complex. $Mo(ONPh)(S_2CNEt_2)_2$ and its Mo(VI) relative, $MoO(NPh)(S_2CNEt_2)_2$, comprise a unique pair of isomeric compounds displaying identical ligating atoms at the metal center but differing both in formal oxidation state and in the nature of the bonding within the ligands. However, efforts to bring about isomerization of these compounds by either thermal or photolytic reactions have failed.

Introduction

Recently, we reported the synthesis and X-ray structure of $Mo(NC_6H_5)_2(S_2CNEt_2)_2$.[1] The two Mo–N bond lengths and the Mo–N–C bond angles in that complex are different and the NC_6H_5 ligands are mutually cis. In addition, the two nitrogen atoms were found to be separated by 2.78 Å, indicating the absence of bonding between these atoms. We were intrigued by the thought that $Mo(NPh)_2(S_2CNEt_2)_2$ could be regarded as a valence isomer of the hypothetical azobenzene complex $Mo(PhNNPh)(S_2CNEt_2)_2$. Side-on coordination of *trans*-azobenzene has been found in $Cp_2Mo(PhNNPh)$.[2] A related dithiocarbamate complex, $Mo(EtO_2CN$-

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$NCO_2Et)(S_2CNEt_2)_2$, has also been reported.[3] Although that complex has not been structurally characterized, most probably the diazene binds through one nitrogen atom and one carbonyl oxygen atom. We tried several synthetic approaches but were unable to isolate $Mo(PhNNPh)(S_2CNEt_2)_2$. However, we have also reported the synthesis and characterization of $MoO(NPh)(S_2CNEt_2)_2$,[4] which should also have a cis configuration of the oxo and NPh ligands, by comparison to the structures of $MoO_2(S_2CNPr_2)_2$[5] and $Mo(NPh)_2(S_2CNEt_2)_2$.[1] Since $Mo(CO)_2(S_2CNEt_2)$ has been shown to add acetylenes to yield complexes of the type $Mo(CO)(alkyne)(S_2CNEt_2)_2$ and $Mo(alkyne)_2(S_2CNEt_2)_2$,[3] it seemed reasonable to examine the reactivity of nitrosobenzene with the dicarbonyl complex. We now report the synthesis and characterization of $Mo(ONPh)(S_2CNEt_2)_2$ which is the desired isomer of $MoO(NPh)(S_2CNEt_2)_2$. Recently, two reports concerning nitrosobenzene complexes of molybdenum have appeared in the literature.[6,7]

Experimental Section

All manipulations were performed either in vacuo or under an argon atmosphere. Solvents were dried and freshly distilled prior to use. The complexes $MoO(S_2CNEt_2)_2$[8] and $Mo(CO)_2(S_2NEt_2)_2$[9] were prepared according to published procedures. $MoO(ONPh)(S_2CNEt_2)_2$ was prepared from nitrosobenzene and $MoO(S_2CNEt_2)_2$.[6] All other materials were reagent grade. Elemental analysis for C, H, and N was performed by using a Hewlett-Packard 185 analyzer.[10] ^1H NMR spectra were recorded at 220 MHz in $CDCl_3$ solutions employing Me_4Si as internal standard with the use of a Varian HR-220 spectrometer. IR spectra were obtained in KBr pellets with a Perkin-Elmer 283 spectrophotometer and calibrated with a polystyrene film. The molecular weight determination employed the Signer method as described by Clark.[11]

Preparation of $Mo(ONPh)(S_2CNEt_2)_2$. Nitrosobenzene (0.217 g; 2.03 mmol) was added to a CH_2Cl_2 solution (25 mL) containing $Mo(CO)_2(S_2CNEt_2)_2$ (0.908 g; 2.03 mmol). Vigorous, immediate gas evolution was observed, and the solution acquired a dark violet color. After the solution was stirred for 10 h at room temperature, the solvent was removed in vacuo, leaving a purple residue. Trituration with pentane (30 mL) and Et_2O (20 mL) yielded the finely divided, glistening maroon product, yield 0.91 g (90%). Anal. Calcd for $Mo(ONC_6H_5)(S_2CNEt_2)_2$: C, 38.47; H, 5.04; N, 8.41. Found: C, 38.12; H, 5.00; N, 8.32. M_r: calcd, 499.59; found, 467 ± 20. In a subsequent experiment, equimolar quantities of $Mo(CO)_2(S_2CNEt_2)_2$ and nitrosobenzene (0.343 mmol) were allowed to react in an analogous manner. At the completion of the reaction, 0.701 mmol of CO (identified by its infrared spectrum) was collected by standard vacuum-line techniques after the gas had been passed through two traps at −196 °C. This experiment proves that the complex is not the imido complex $Mo(NC_6H_5)(S_2CNEt_2)_2$ since the gas would have been composed of an equimolar mixture of CO and CO_2.

The complex, which is soluble in halogenated hydrocarbons and benzene, may be handled in air for brief periods.

Reaction of $Mo(CO)_2(S_2CNEt_2)_2$ and Nitrobenzene. One milliliter of nitrobenzene was added to 35 mL of 1,2-dichloroethane, and the solution was blanketed with argon. $Mo(CO)_2(S_2CNEt_2)_2$ (0.6 g) was added, and the solution was heated to reflux for 30 min. The solvent was then removed in vacuo. The oily residue was triturated with Et_2O (2 × 30 mL) and then dried in vacuo to yield the product, $MoO(ONPh)(S_2CNEt_2)_2$. The infrared and ^1H NMR spectra of this material were identical with those obtained from the material prepared by the previously published method.[6]

Table I. ^1H NMR Data[a]

	δ		
complex	C_6H_5	CH_2	CH_3
$Mo(ONPh)$-$(S_2CNEt_2)_2$	7.50–6.36 (br, m, 5 H)	3.73 (br, m, 8 H)	1.23 (m, 12 H)
$MoO(ONPh)$-$(S_2CNEt_2)_2$	7.55 (d, 2 H), 7.25 (m, 2 H), 6.93 (m, 1 H)	4.04 (m, 8 H)	1.36 (m, 9 H), 1.14 (m, 3 H)
$MoO(NPh)$-$(S_2CNEt_2)_2$	7.38 (d, 2 H), 7.25 (m, 2 H), 7.04 (m, 1 H)	3.74 (q, 8 H)	1.29 (t, 12 H)
ONPh	7.89 (d, 2 H), 7.70–7.52 (m, 3 H)		

[a] Measured in $CDCl_3$ solution; 16 °C; 220 MHz. Chemical shifts are expressed downfield from internal Me_4Si. Abbreviations used: br, broad; m, multiplet; q, quartet; t, triplet; d, doublet; Et, C_2H_5; Ph, C_6H_5.

Reaction of $MoO(ONPh)(S_2CNEt_2)_2$ and Ph_3P. A CH_2Cl_2 solution containing $MoO(ONPh)(S_2CNEt_2)_2$ (0.185 g; 0.36 mmol) and Ph_3P (0.094 g; 0.36 mmol) was stirred at room temperature for 4 days. The solution was evaporated in vacuo to yield a dark purple, glassy solid. The infrared spectrum of this material showed the presence of both Ph_3PO and $Mo(ONPh)(S_2CNEt_2)_2$. In addition, the strong band observed at 935 cm^{-1} in the spectrum of $MoO(ONPh)(S_2CNEt_2)_2$ due to $\nu(MoO)$[6] diminished in intensity.

Results and Discussion

The quantitative displacement of CO from $Mo(CO)_2(S_2CNEt_2)_2$ by nitrosobenzene, as shown in eq 1, occurs readily

$$Mo(CO)_2(S_2CNEt_2)_2 + C_6H_5NO \rightarrow Mo(ONC_6H_5)(S_2CNEt_2)_2 + 2CO \tag{1}$$

in CH_2Cl_2 at room temperature. A related reaction, shown in eq 2, was recently described by Sharpless, Ibers, et al.[6]

$$MoO(S_2CNEt_2)_2 + C_6H_5NO \rightarrow MoO(ONC_6H_5)(S_2CNEt_2)_2 \tag{2}$$

These workers also showed that the oxo/nitrosobenzene complex could be obtained from $MoO_2(S_2CNEt_2)_2$ and phenylhydroxylamine. We have found that $MoO(ONPh)(S_2CNEt_2)_2$ can be prepared a third way, as shown in eq 3. This reaction

$$Mo(CO)_2(S_2CNEt_2)_2 + C_6H_5NO_2 \rightarrow MoO(ONC_6H_5)(S_2CNEt_2)_2 + 2CO \tag{3}$$

also occurs rapidly and yields a product whose infrared and ^1H NMR spectra are identical with those of the product as prepared via eq 2. Reduction of $MoO(ONPh)(S_2CNEt_2)_2$ with a stoichiometric amount of triphenylphosphine affords $Mo(ONPh)(S_2CNEt_2)_2$ and triphenylphosphine oxide as shown in eq 4.

$$MoO(ONPh)(S_2CNEt_2)_2 + Ph_3P \rightarrow Mo(ONPh)(S_2CNEt_2)_2 + Ph_3PO \tag{4}$$

In a study of the reactions of uranocenes with nitro compounds, it has been shown that complete deoxygenation of nitrobenzene occurs, forming UO_2 and azobenzene in high yield.[12] Nitrosobenzene is believed to be an intermediate in this reaction since it also is rapidly deoxygenated by uranocenes to give azobenzene in quantitative yield. Since the formation of stable uranium–oxygen bonds is probably a major driving force in the above reactions, it is perhaps surprising that the reactions in eqs 2 and 3 do not proceed in an analogous manner

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to yield $MoO_2(S_2CNEt_2)_2$ and azobenzene in view of the well-known avidity of molybdenum for the formation of strong multiple bonds with oxygen.

Table I presents ^1H NMR data for $Mo(ONPh)(S_2CNEt_2)_2$ and related compounds. The most noticeable aspect of the data for $Mo(ONPh)(S_2CNEt_2)_2$ is the fact that the resonances due to the phenyl-ring protons are shifted upfield when compared to those of either $MoO(ONPh)(S_2CNEt_2)_2$ or nitrosobenzene itself. A similar shift of these resonances has been observed by Otsuka et al. in the spectra of a number of η^2-nitrosobenzene complexes of the nickel triad metals.[13] This upfield shift is indicative of considerable transfer of electron density from nitrosobenzene to molybdenum and supports our contention that in $Mo(ONPh)(S_2CNEt_2)_2$ the nitrosobenzene is η^2 coordinated. Certainly other coordination modes are possible. In $Pd(ONPh)Cl_2$, the nitrosobenzene is coordinated solely through the nitrogen atom,[14] while in uranyl complexes of *p*-nitroso-*N*,*N*-dimethylaniline, coordination is believed to occur through the very basic nitroso oxygen atom.[15] The nitroso group may also act as a bridging ligand.[16,17] However, in the present case, η^2 coordination would be expected since that geometry is found in several related nitrosobenzene complexes of both tungsten and molybdenum, including $MoO(ONPh)(S_2CNEt_2)_2$.[6,7] Side-on coordination would also be expected to contribute to the stability of $Mo(ONPh)(S_2CNEt_2)_2$ both by furnishing more electron density to the metal and by reducing coordinative unsaturation as compared with η^1 coordination.

We believe therefore that $Mo(ONPh)(S_2CNEt_2)_2$ is a monomeric, six-coordinate complex. We have previously reported the synthesis and characterization of *cis*-$MoO(NPh)(S_2CNEt_2)_2$.[4] Assignment of the cis stereochemistry was based on the fact that the cis disposition would allow maximum multiple bonding between molybdenum and the oxo and NPh ligands. In addition, both $MoO_2(S_2CNPr_2)_2$[5] and $Mo(NPh)_2(S_2CNEt_2)_2$[1] exhibit cis stereochemistries. Thus, although it was not confirmed by X-ray structural analysis, we believe that $Mo(ONPh)(S_2CNEt_2)_2$ and $MoO(NPh)(S_2CNEt_2)_2$ constitute a unique pair of compounds displaying identical ligating atoms of similar stereochemical disposition around the metal center yet differing both in formal oxidation state and in the nature of the bonding within the ligands. Spectroscopically, the two complexes are quite distinct, as illustrated by the ^1H NMR data in Table I. In addition, $MoO(NPh)(S_2CNEt_2)_2$ displays a strong MoO vibration at 872 cm^{-1} in its infrared spectrum, which has been verified by labeling experiments,[18] while the infrared spectrum of $Mo(ONPh)(S_2CNEt_2)_2$ is devoid of any similarly assignable bands.

In a formal sense, $MoO(NPh)(S_2CNEt_2)_2$ and $Mo(ONPh)(S_2CNEt_2)_2$ are related by a four-electron intramolecular electron-transfer process. In a practical sense, however, the compounds show no tendency for isomerization. In previous work,[4] we found no tendency for $MoO(NPh)(S_2CNEt_2)_2$ to isomerize to the nitrosobenzene complex. We would expect, however, that the Mo(VI) complex would be thermodynamically more stable than the nitrosobenzene complex due to the presence of the multiply bound oxo ligand. However, refluxing a 1,2-dichloroethane solution of $Mo(ONPh)(S_2CNEt_2)_2$ for 12 h did not produce any $MoO(NPh)(S_2CNEt_2)_2$. Similarly, photolysis of a CH_2Cl_2 solution of the nitrosobenzene complex (using a tungsten filament lamp) for 10 h failed to produce the oxo imido complex. However, if vigorous drying procedures are not employed, CH_2Cl_2–ether solutions of $Mo(ONPh)(S_2CNEt_2)_2$ will deposit yellow acicular crystals of $Mo_2O_4(NPh)(S_2CNEt_2)_2$. We have previously obtained this material from solutions of $MoO(NPh)(S_2CNEt_2)_2$.[4] The infrared spectrum of $Mo_2O_4(NPh)(S_2CNEt_2)_2$ shows two very strong bands at 946 and 910 cm^{-1}, which suggests that each molybdenum atom in the dimer is bound to two cis terminal oxo ligands. This arrangement of the oxo ligands has been observed previously in the structures of the $[Mo_2O_5(C_6H_4O_2)_2]^{2-}$[19] and $[Mo_2O_5(C_2O_4)_2(H_2O)_2]^{2-}$[20] anions. While we are unsure about the mechanism of formation of $Mo_2O_4(NPh)(S_2CNEt_2)_2$ from $Mo(ONPh)(S_2CNEt_2)_2$, scission of the nitroso nitrogen–oxygen bond has occurred, albeit in an unexpected manner.

Registry No. $Mo(ONPh)(S_2CNEt_2)_2$, 74096-96-1; $MoO(ONPh)(S_2CNEt_2)_2$, 68976-20-5; $Mo(CO)_2(S_2CNEt_2)_2$, 18947-43-8; $C_6H_5NO_2$, 98-95-3; $MoO(NPh)(S_2CNEt_2)_2$, 70749-56-3; ONPh, 586-96-9.

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