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Synthesis and characterization of d^2-molybdenum imido complexes containing coordinated dithio-ligands. MO rationalization of the preferential isomer formation of $Mo(N-2,4,6-Me_3C_6H_2)(S_2COMe)Cl(PMe_3)_2$

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Abstract

Treatment of the complex $Mo(Nmes)Cl_2(PMe_3)_3$ (**1**) (mes = $2,4,6-Me_3C_6H_2$, 2,4,6-trimethylphenyl) with two equivalents of the potassium salt of methyl xanthate, KS_2COMe, or the potassium 1-pyrrole-carbodithioate, $KS_2C(NC_4H_4)$, gave $Mo(Nmes)(S-S)_2(PMe_3)$ (S-S = S_2COMe **2**; $S_2C(NC_4H_4)$ **3**). When the reaction of **1** with KS_2COMe is carried out in a 1:1 stoichiometry, the complex $Mo(Nmes)(S_2COMe)Cl(PMe_3)_2$ (**4**) is obtained. The chlorine atom in **4** occupies the *trans* position with respect to the organoimido ligand. MO calculations of the EH type rationalize the preferential formation of **4** as the thermodynamically preferred isomer in this metathesis reaction. The reaction of **4** with CO produces $Mo(Nmes)(S_2COMe)Cl(CO)(PMe_3)$ (**5**). Reaction of **1** with carbon disulfide yields the complex $Mo(Nmes)Cl_2(S_2CPMe_3)$ (**6**), containing a trihapto-(*S*,*S*′,*C*) phosphoniumdithiocarboxylate ligand. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Imido; Molybdenum; Dithio-ligand; EHMO calculations

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1. Introduction

Interest in the chemistry of compounds containing organoimido ligands has increased enormously in the last two decades. In particular, the use of this ligand in stabilizing transition metals in high oxidation states [1] and the role they play in the development of well-defined catalysts are well documented [2].

Previous work from our laboratories has resulted in the preparation of a variety of oxo- and imido-derivatives of molybdenum containing dithioacid and related ligands [3,4]. As an extension of this work [5], we now wish to report the synthesis and characterization of some new d^2-imido complexes of molybdenum containing xanthate, dithiocarbamate and phosphoniumdithiocarboxylate ligands. As discussed below, they have been prepared using the complex $Mo(Nmes)Cl_2(PMe_3)_3$ (**1**) (mes = 2,4,6-trimethylphenyl, $Me_3C_6H_2$) as the starting material. A molecular orbital (MO) rationalization of the preferential formation of only one isomer of the complex $Mo(Nmes)(S_2COMe)Cl(PMe_3)_2$ (**4**) is also presented.

2. Experimental

Microanalyses were carried out by the Microanalytical Service of the University of Sevilla. IR spectra were recorded on a Perkin–Elmer model 883 spectrophotometer. ^{1}H-, ^{13}C- and ^{31}P-NMR spectra were run on Bruker AMX-300 and AMX-500 spectrometers. ^{31}P shifts were measured with respect to external 85% H_3PO_4. ^{13}C-NMR spectra were referenced using the ^{13}C resonance of the solvent as an internal standard but are reported with respect to $SiMe_4$. All preparations and other operations were carried out under oxygen-free nitrogen, following conventional Schlenk techniques. Solvents were dried and degassed before use. The light petroleum ether used had b.p. 40–60°C. The compound

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$Mo(Nmes)Cl_2(PMe_3)_3$ was prepared according to the literature procedure [5].

2.1. Preparation of $Mo(Nmes)(S_2COMe)_2(PMe_3)$ (2)

A mixture of $Mo(Nmes)Cl_2(PMe_3)_3$ (0.07 g, 0.15 mmol) and KS_2COMe (0.04 g, 0.30 mmol) was dissolved in THF (25 ml). The suspension was refluxed overnight, the solvent was removed by pumping under vacuum and the resulting residue extracted with light petroleum. The solution was concentrated and cooled to −20°C. Compound **2** was isolated as an orange–brownish solid in 30% yield. $^{31}P\{^1H\}$-NMR (C_6D_6): δ −4.1 (s). ^1H-NMR (C_6D_6): δ 6.44 (s, 2H, C*H*, Ph), 3.47, 3.43 (s, 3H, OC*H*$_3$), 2.33 (s, 6H, *o*-C*H*$_3$), 1.91 (s, 3H, *p*-C*H*$_3$), 1.17 (d, $J_{HP} = 8.6$ Hz, 9H, PMe$_3$). $^{13}C\{^1H\}$-NMR (125 MHz, C_6D_6): δ 218.4 (s, S$_2$*C*), 172.2 (s, S$_2$*C*), 153.7 (s, *ipso*-C), 135.4, 135.3 (s, *p*-C and *o*-C), 128.7 (s, *m*-C), 57.4, 57.0 (s, O*C*H$_3$), 20.8 (s, *p*-CH$_3$), 19.7 (s, *o*-CH$_3$), 15.1 (d, $J_{CP} = 26.7$ Hz, PMe$_3$). Crystals of complex **2** were contaminated by small amounts of **4**. This makes it difficult to obtain reliable microanalytical data. Anal. Calc. for $C_{16}H_{26}NMoO_2PS_4$: C, 37.0; H, 5.0; N, 2.7. Found: C, 38.5; H, 5.3; N, 2.7%.

2.2. Preparation of $Mo(Nmes)[S_2C(NC_4H_4)]_2(PMe_3)$ (3)

To a solution of $Mo(Nmes)Cl_2(PMe_3)_3$ (0.12 g, 0.23 mmol) in THF (15 ml) was added $KS_2C(NC_4H_4)$ (0.09 g, 0.46 mmol) in THF (15 ml). The mixture was stirred overnight at room temperature (r.t.). The solvent was then removed and the residue extracted with ether/light petroleum. Concentration of the solution and cooling at −20°C gave orange crystals of compound **3** in 50% yield. $^{31}P\{^1H\}$-NMR (C_6D_6): δ −8.33 (s). ^1H-NMR (300 MHz, C_6D_6): δ 7.63, 7.52 (t, $^3J_{HH} = 2.3$ Hz, 2H, NC$_4$*H*$_4$), 6.33 (s, 2H, C*H*, Ph), 6.01, 5.96 (t, $^3J_{HH} = 2.3$ Hz, 2H, NC$_4$*H*$_4$), 1.96 (s, 6H, *o*-C*H*$_3$), 1.84 (s, 3H, *p*-C*H*$_3$), 1.02 (d, $J_{HP} = 9.2$ Hz, 9H, PMe$_3$). $^{13}C\{^1H\}$-NMR (75 MHz, C_6D_6): δ 206.7 (d, $J_{CP} = 4.3$ Hz, eq-S$_2$*C*), 187.3 (s, ax-S$_2$*C*), 153.3 (s, *ipso*-C), 136.4, 135.4 (s, *p*-C and *o*-C), 128.4 (s, *m*-C), 121.2, 118.5, 114.3, 109.2 (s, N*C*$_4$H$_4$), 20.7 (s, *p*-CH$_3$), 19.3 (s, *o*-CH$_3$), 14.7 (d, $J_{CP} = 26.2$ Hz, PMe$_3$). Anal. Calc. for $C_{22}H_{28}N_3MoPS_4$: C, 44.8; H, 4.4; N, 7.1. Found: C, 44.7; H, 4.4; N, 7.0%.

2.3. Preparation of $Mo(Nmes)(S_2COMe)Cl(PMe_3)_2$ (4)

A mixture of $Mo(Nmes)Cl_2(PMe_3)_3$ (0.26 g, 0.49 mmol) and KS_2COMe (0.07 g, 0.5 mmol) was dissolved in THF (25 ml). The solution was stirred for 24 h at r.t. The solvent was removed and the dark red solid was recrystallized from ether/light petroleum to give **4** as red crystals in 55% yield. $^{31}P\{^1H\}$-NMR (C_6D_6): δ 0.3 (s). ^1H-NMR (500 MHz, C_6D_6): δ 6.49 (s, 2H, C*H*, Ph), 3.71 (s, 3H, OC*H*$_3$), 2.44 (s, 6H, *o*-C*H*$_3$), 1.94 (s, 3H, *p*-C*H*$_3$), 1.37 (d, $J_{HP} = 7.7$ Hz, 18H, PMe$_3$). $^{13}C\{^1H\}$-NMR (125 MHz, C_6D_6): δ 235.4 (s, S$_2$*C*), 150.8 (s, *ipso*-C), 135.7, 134.7 (s, *p*-C and *o*-C), 128.8 (s, *m*-C), 57.9 (s, O*C*H$_3$), 19.7 (s, *p*-CH$_3$), 19.1 (m, PMe$_3$), 18.3 (s, *o*-CH$_3$). Anal. Calc. for $C_{17}H_{32}NClMoOP_2S_2$: C, 39.0; H, 6.1; N, 2.7. Found: C, 39.0; H, 6.3; N, 2.7%.

2.4. Interaction of 4 with CO: synthesis of $Mo(Nmes)(S_2COMe)Cl(CO)(PMe_3)$ (5)

A solution of $Mo(Nmes)(S_2COMe)Cl(PMe_3)_3$ (0.06 g, 0.11 mmol) in Et_2O (20 ml) was pressurized with CO (2 atm) in a pressure bottle. The mixture was stirred overnight and then the solvent was removed under vacuum. Crystallization of the residue always gave a mixture of the starting material **4** and compound **5**. The separation by fractional crystallization was not possible, but **5** was fully characterized by solution NMR. $^{31}P\{^1H\}$-NMR (C_6D_6): δ −5.65 (s). ^1H-NMR (500 MHz, C_6D_6): δ 6.38 (s, 2H, C*H*, Ph), 3.54 (s, 3H, OC*H*$_3$), 2.31 (s, 6H, *o*-C*H*$_3$), 1.90 (s, 3H, *p*-C*H*$_3$), 1.22 (d, $J_{HP} = 8.9$ Hz, 9H, PMe$_3$). $^{13}C\{^1H\}$-NMR (125 MHz, C_6D_6): δ 238.6 (d, $J_{CP} = 7.6$ Hz, *C*O), 235.8 (s, S$_2$*C*), 150.1 (s, *ipso*-C), 136.9, 136.7 (s, *p*-C and *o*-C), 129.0 (s, *m*-C), 58.2 (s, O*C*H$_3$), 20.8 (s, *p*-CH$_3$), 19.1 (s, *o*-CH$_3$), 16.5 (d, $J_{CP} = 27.5$ Hz, PMe$_3$).

2.5. Preparation of $Mo(Nmes)Cl_2(S_2CPMe_3)$ (6)

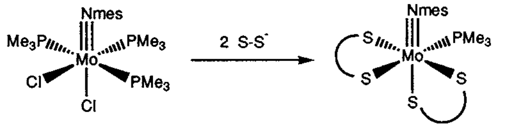
A solution of $Mo(Nmes)Cl_2(PMe_3)_3$ (0.20 g, 0.38 mmol) in THF (20 ml) was treated with one equivalent of CS_2 (0.38 ml of a solution 1 M in THF). The mixture was stirred overnight at r.t. and an orange solid was formed. The resulting suspension was filtered, the solution evaporated to dryness and the residue extracted with THF. Concentration and cooling at −30°C afforded orange crystals of **6**. $^{31}P\{^1H\}$-NMR (acetone-*d*$_6$): δ 42.5 (s). ^1H-NMR (500 MHz, acetone-*d*$_6$): δ 6.80 (s, 2H, C*H*, Ph), 2.36 (s, 6H, *o*-C*H*$_3$), 2.20 (s, 3H, *p*-C*H*$_3$), 1.94 (d, $J_{HP} = 13.8$ Hz, 9H, PMe$_3$). $^{13}C\{^1H\}$-NMR (125 MHz, acetone-*d*$_6$): δ 153.6 (s, *ipso*-C), 139.5, 136.6 (s, *p*-C and *o*-C), 129.7 (s, *m*-C), 97.5 (d, $J_{CP} = 78.2$ Hz, *C*S$_2$), 21.3 (s, *p*-CH$_3$), 18.6 (s, *o*-CH$_3$), 11.2 (d, $J_{CP} = 57.2$ Hz, PMe$_3$). Anal. Calc. for $C_{13}H_{20}NCl_2MoPS_2\cdot\frac{1}{4}THF$: C, 35.7; H, 4.7; N, 3.0. Found: C, 35.3; H, 5.0; N, 2.9%.

2.6. Extended Hückel molecular orbital study

The relative stability of the three isomers (**I–III**) of complex $Mo(Nmes)(S_2COMe)Cl(PMe_3)_2$ (**4**) was studied employing the model compounds $Mo(NH)(S_2COH)Cl(PH_3)_2$ at the extended Hückel level [6] by using a weighted modified Wolfsberg–Helmholz formula [7]. The calculations were performed by means of the CACAO package [8]. The STO parameters used are the standard ones tabulated in the program. The geometry of the model compounds was based on the ideal octahedron and bond distances and angles were adapted from the results on a structural search of the Cambridge Crystallographic Database [9].

3. Results and discussion

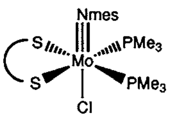
We have recently reported [5] that treatment of compound $Mo(Nmes)Cl_3(dme)$ with two equivalents of PMe_3 and subsequent Na–Hg reduction, in the presence of one additional equivalent of PMe_3, gives $Mo(Nmes)Cl_2(PMe_3)_3$ (**1**). This synthetic methodology provides easy access to the chemistry of the d^2-Mo(Nmes) moiety. We also showed that interaction of **1** with two equivalents of KS_2CO^iPr gave $Mo(Nmes)(S_2CO^iPr)_2(PMe_3)$, in contrast with the behavior previously observed for the oxo-analogue of **1**, which under similar conditions gave $MoO[S_2C(PMe_3)O^iPr](S_2CO^iPr)$ [3b]. With the aim of ascertaining if the attainment of the classical six-coordinate structure is a general observation, we have enlarged the number of d^2-imido derivatives of molybdenum by preparing the complexes $Mo(Nmes)(S_2COMe)_2(PMe_3)$ (**2**) and $Mo(Nmes)[S_2C(NC_4H_4)]_2(PMe_3)$ (**3**). They can be synthesized by reacting **1** with the potassium salts of methyl xanthate and 1-pyrrole-carbodithioate, respectively (Eq. (1)), and are obtained as orange–brownish and orange crystalline solids, respectively.



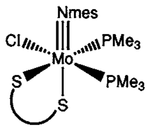
(1)

Both compounds are moderately stable upon exposure to air in the solid state. Their NMR spectra are consistent with the schematic structure shown in Eq. (1). The PMe_3 ligand of compounds **2** and **3** gives a $^{31}P\{^1H\}$-NMR singlet, with a chemical shift characteristic of coordinated PMe_3 ligands. The two dithio-ligands give rise to two different sets of signals. For instance, two resonances are observed for the S_2C carbon atoms of **3** in the $^{13}C\{^1H\}$-NMR spectra (206.7 (d, $J_{CP} = 4.3$ Hz, S_2C eq) and 187.3 (s, S_2C ax) ppm). These resonances are in the range expected for bidentate dithio-ligands.

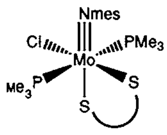
The formation of **2** requires heating for completion. When the reaction is carried out at r.t., the analysis of the reaction mixture by $^{31}P\{^1H\}$-NMR reveals three species: the starting material **1**, complex **2** and a singlet resonance corresponding to a new compound. This complex was identified as the monosubstituted xanthate derivative $Mo(Nmes)(S_2COMe)Cl(PMe_3)_2$, (**4**), which is best prepared by carrying out the reaction of **1** and KS_2COMe in a 1:1 ratio, at r.t. If the substitution of one chloride and one PMe_3 groups by the xanthate, occurs without ligand redistribution, three possible isomers (**I**–**III**) can be envisaged.



I



II

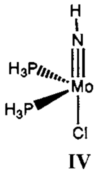


III

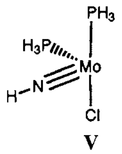
The imido ligand exerts a strong *trans* influence [1b,10]. By assuming that the *trans* effect follows the same trend of the *trans* influence, one would expect that the metathesis occurs with the initial substitution of the chloride ligand that occupies the *trans* position with respect to the imido group. This substitution will produce the isomers **II** or **III**. However, the isolated complex **4** is characterized by a singlet in the $^{31}P\{^1H\}$-NMR spectrum and by the presence of a doublet for the PMe_3 ligands in the ^1H-NMR spectrum, with a coupling constant J_{HP} of 7.7 Hz. Consequently, the spectroscopic data of **4** are in agreement with structure **I**, in which the two PMe_3 ligands are equivalent and mutually *cis*. We have no evidence for the formation of complexes with either structure **II** or **III**.

In order to rationalize the preferential formation of only isomer **I** in this reaction, we have performed an EHMO study with the model compound $Mo(NH)(S_2COH)Cl(PH_3)_2$ in the three geometries, **I**–**III**. The main electronic features are not affected by the substitution of the terminal 2,4,6-$Me_3C_6H_2$ group by a single H atom. The absolute energy minimum is calculated to correspond to the experimentally observed isomer **I**, which is ca. 0.2 eV more stable than **II** and **III**.

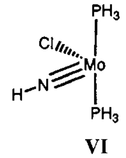
In view of the limitations of the EHMO method for the prediction of the correct energies, we decided to analyze the fragment molecular orbital (FMO) interactions of the three model compounds. In all cases, the common dithio-ligand, S_2COH, was considered to interact with a metallic fragment of the type d^2-ML_4 and the geometry defined in **IV**–**VI**.



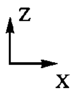
IV



V



VI



Despite the fact that the presence of the triple bonded imido functionality makes these d^2-ML_4 fragments unusual, their FMOs can be inferred from the classical C_{2v}-ML_4 fragment [11]. Fig. 1 illustrates the metal FMOs of **IV**. For comparison, the energies of the analogous FMOs of **V** and **VI** have been also included.

They consist of a high-lying σ-hybrid, **3a**′, a pair of hybridized d_{xy} and d_{yz} orbitals (**2a**″ and **1a**″) and the **2a**′ FMO (d_{xz} orbital). The FMOs **1a**″, **2a**′ and **2a**″ show the antibonding combination with the π-contribution of the imido and the chloride ligands. The HOMO **1a**′ of **IV** is essentially the $d_{x^2-y^2}$ orbital. The presence of the formally π-donor chloride ligand in the *cis* position with respect to the imido functionality produces a destabilization (four electron repulsion) of the HOMO in the fragments **V** and **VI**. Accordingly, the minimum energy corresponds to the metal fragment **IV**, which is found to be ca. 0.6 eV more stable than the other two structures.

The FMOs of some dithio-ligands have been outlined previously [12], and include two filled in-plane combinations of sulfur lone pairs (in phase, σ_{ip}, and out-of-phase, σ_{op}) and the π-S_2C system. The interaction diagrams for the three isomers (not represented) reveal that the π combinations of the dithio-ligand basically do not participate in the bonding scheme and only the two filled σ-FMOs (σ_{op} and σ_{ip}) contribute to the construction of the MOs.

As stated above, the energy minimum corresponds to isomer **I**. Moreover, the best overlap populations between the metallic fragments and the S_2COH ligand occurs also for this isomer. In particular, the FMO **1a**″ shows an appreciable overlap population with the σ_{op} contribution. This interaction has no match for the isomers **II** or **III**. Finally, the decrease in the energy difference between **I** and the other isomers (from 0.6 eV in the metal fragments to 0.2 eV) has its origin in a four electron repulsion between the **1a**′ fragment (HOMO for **IV**) and the σ_{ip} FMO.

For a d^2 metal configuration, the presence of the formally π-donor chloride ligand in the *trans* position with respect to the imido ligand is electronically more favorable than the structure containing the chloride ligand occupying a *cis*-coordination site. In conclusion, our calculations indicate that isomer **I** is the most stable configuration for the model compound $Mo(NH)(S_2COH)Cl(PH_3)_2$ and this fact is in agreement with the preferential formation of this isomer, observed experimentally.

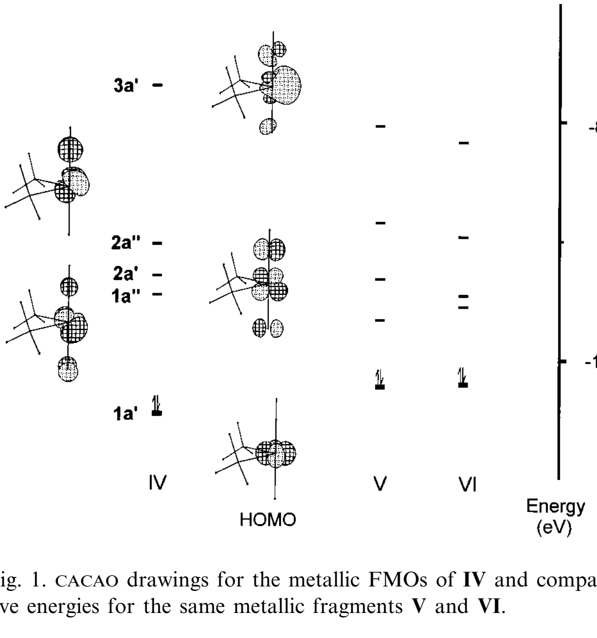
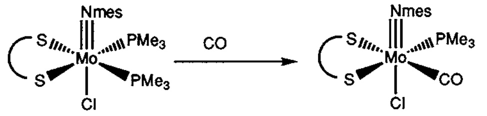


Fig. 1. CACAO drawings for the metallic FMOs of **IV** and comparative energies for the same metallic fragments **V** and **VI**.

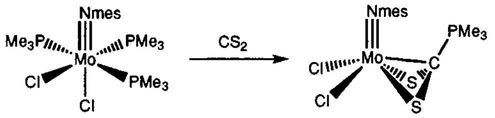
Interaction of solutions of **4** with CO takes place with substitution of one PMe_3 ligand and formation of complex $Mo(Nmes)(S_2COMe)Cl(CO)(PMe_3)$ (**5**) (Eq. (2)). The presence of the CO group was spectroscopically confirmed by IR and was further indicated by the appearance of a doublet at 238.6 ppm in the $^{13}C\{^1H\}$-NMR spectrum ($^2J_{CP} = 7.6$ Hz).



(2)

Other NMR data are collected in Section 2 and need no further comment. As outlined before, the MO analysis of **4** shows a HOMO composed essentially of the filled $d_{x^2-y^2}$ metal orbital. Accordingly, the substitution of one PMe_3 ligand by CO stabilizes the HOMO by the π-acceptor capability of the latter ligand [1b,5,13].

Addition of carbon disulfide to transition metal–phosphine derivatives is a common entry to the chemistry of S_2CPR_3 complexes [14]. The easy dissociation of PMe_3 from complex **1**, makes it a useful candidate for the investigation of this reaction. Treatment of solutions of **1** with one equivalent of carbon disulfide affords compound $Mo(Nmes)Cl_2(S_2CPMe_3)$ (**6**), as an orange microcrystalline solid (Eq. (3)). The S_2CPMe_3 ligand gives characteristic ^1H (doublet at 1.94 ppm, $J_{HP} = 13.8$ Hz) and $^{31}P\{^1H\}$ (singlet at 42.5 ppm) resonances, attributable to a phosphonium group.



(3)

In addition, a ^{13}C doublet at 97.5 ppm ($J_{CP} = 78.2$ Hz) can be assigned to the central carbon atom of the S_2CPMe_3 ligand. The chemical shift of this signal is indicative of a trihapto-(*S*,*S*′,*C*) coordination [14,15]. The structure displayed in Eq. (3) for complex **6** was proposed on the basis of previous MO studies [16] carried out on related d^2-systems that contain a multiple-bonded ligand and the trihapto-(*S*,*S*′,*C*) S_2CPR_3 group.

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