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FULL PAPER

Acetonitrile hydration *versus* molybdenum oxidation at the sulfur-rich bimetallic site $\{Mo^{III}{}_2Cp_2(\mu\text{-}SMe)_3\}^+$. Crystal structure of the μ-$\eta^1:\eta^1$-amidato complex $[Mo_2Cp_2(\mu\text{-}MeCONH)(\mu\text{-}SMe)_3]$

Philippe Schollhammer,[a] Marc Le Hénanf,[a] Christine Le Roy-Le Floch,[a] François Y. Pétillon,*[a] Jean Talarmin[a] and Kenneth W. Muir*[b]

[a] *UMR CNRS 6521, Chimie, Electrochimie Moléculaires et Chimie Analytique, Faculté des Sciences, Université de Bretagne Occidentale, BP 809, 29285 Brest-Cédex, France*
[b] *Department of Chemistry, University of Glasgow, Glasgow, UK G12 8QQ*

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The paramagnetic μ-amidato species $[Mo_2Cp_2(\mu\text{-}MeCONH)(\mu\text{-}SMe)_3]BF_4$ **2a**$^+$ (Cp = η-C_5H_5) is formed exclusively when the compound $[Mo_2Cp_2(MeCN)_2(\mu\text{-}SMe)_3]BF_4$ **1a** in powder form is kept in air, whereas the same starting complex in solution gives a mixture of the μ-amidato species and the μ-oxo complex $[Mo_2Cp_2(\mu\text{-}O)(\mu\text{-}SMe)_3]BF_4$ **3**. The oxo complex is formed quantitatively on warming $[Mo_2Cp_2(\mu\text{-}Cl)(\mu\text{-}SMe)_3]BF_4$ in water. The reduction of **2a**$^+$ by $NaBH_4$ in CH_3CN affords the molecular product $[Mo_2Cp_2(\mu\text{-}MeCONH)(\mu\text{-}SMe)_3]$ **2a** which has been characterised by X-ray analysis.

Introduction

Cationic complexes containing weakly coordinated molecules which behave as temporary ligands are useful precursors for molecular activation.[1] This consideration has led us to study the reactivity of electrophilic thiolato-bridged bimetallic sites[2] and, in particular, that of the bis(acetonitrile)dimolybdenum species $[Mo_2Cp_2(MeCN)_2(\mu\text{-}SMe)_3]BF_4$ **1a**. We have recently shown that the nitrile groups in **1a** can be replaced by terminal alkynes and that the dimolybdenum $[Mo_2Cp_2(\mu\text{-}SMe)_3]^+$ site then induces C–C and C–S couplings which give rise to various organosulfur ligands.[3] Furthermore, our results indicate that the electrophilic character of the carbon atoms of the nitrile groups is increased by their coordination to molybdenum atoms: **1a**, for example, readily undergoes nucleophilic attack to form $\{Mo_2(\mu\text{-}N{=}C(CH_3)R)\}$ azavinylidene species, either by condensation of the two acetonitrile groups under basic conditions (R = CH_2CN) or by hydride transfer to one MeCN ligand (R = H).[4,5] Several examples of facile nitrile hydrolyses, which are relevant either to methods for functionalising nitriles or to the understanding of some biotransformations, have been reported at mono- or bi-metallic sites.[6] These reactions also illustrate the enhancement of the electrophilic character of the nitrile upon coordination through its nitrogen atom to some metal frameworks. We now report that the process of hydrolysis of a nitrile coordinated to a metal may be limited by direct attack of the nucleophile on the metal itself. The complex $[Mo_2Cp_2(MeCN)_2(\mu\text{-}SMe)_3]BF_4$, **1a**, undergoes two competitive hydrolysis pathways to give either the novel μ-amidato derivative $[Mo_2Cp_2(\mu\text{-}MeCONH)(\mu\text{-}SMe)_3]BF_4$ **2a**$^+$ or the μ-oxo complex $[Mo_2Cp_2(\mu\text{-}O)(\mu\text{-}SMe)_3]BF_4$ **3**. The nucleophilic addition of hydroxide to **1a** and related complexes has also been investigated.

Results and discussion

Synthesis and spectroscopic characterisation of $[Mo_2Cp_2(\mu\text{-}MeCONH)(\mu\text{-}SMe)_3]$ 2a and $[Mo_2Cp_2(\mu\text{-}MeCONH)(\mu\text{-}SMe)_3]^+$ 2a$^+$

When **1a**, in the form of a red-brown powder, was kept in contact with air for 48 h, it readily afforded a brown paramagnetic complex **2a**$^+$. This was characterised by its electrochemical behaviour and by its elemental analysis which accords with the formula $C_{15}H_{23}BF_4Mo_2NOS_3$. Cyclic voltammetry in $MeCN\text{–}NBu_4PF_6$ electrolytes shows two reversible, one-electron steps [$E^{1/2}{}_{red} = -0.8$; $E^{1/2}{}_{ox} = -0.04$ V *vs.* ferrocene–ferrocenium] (Fig. 1). Addition of an excess of $NaBH_4$ to an acetonitrile solution of **2a**$^+$ resulted in reduction of the complex to give the molecular product **2a**. Redox relationships between **2a** and **2a**$^+$ were unambiguously established by cyclic voltammetry of the complexes [**2a** : $E^{1/2}{}_{ox1} = -0.8$; $E^{1/2}{}_{ox2} = -0.04$ V]. The reduced form **2a** was identified as a tris(thiolato)-bridged dinuclear species containing a bridging CH_3CONH amidato ligand (Scheme 1). The structure of **2a** was firmly established from its elemental analysis and spectroscopic data and confirmed by a single crystal X-ray diffraction study. ^1H and ^{13}C NMR spectra revealed the presence in variable ratios of two isomers which, we assume, differ only in the orientations of the methyl groups borne by the sulfur atoms. For each isomer the ^1H NMR spectrum displayed two resonances for the

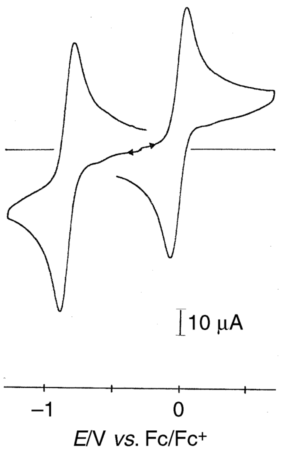

Fig. 1 Cyclic voltammetry of **2a**$^+$ *ca.* 2 mM in $MeCN\text{–}NBu_4PF_6$. The arrows indicate the initial scan directions (vitreous carbon electrode; scan rate 0.2 V s^{-1}).

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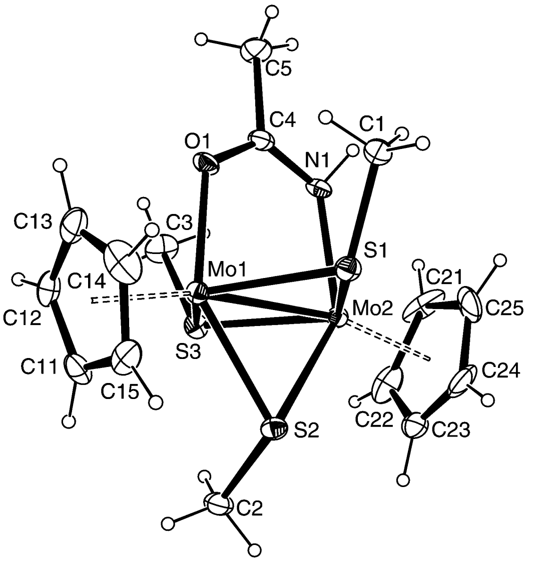
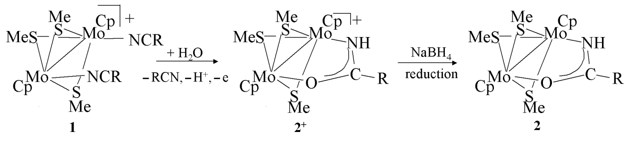


Fig. 2 Molecular structure of **2a** showing the atomic numbering scheme.



Scheme 1

cyclopentadienyl groups and four peaks in the SMe and MeCN regions, indicating that the $\{Mo_2Cp_2(\mu\text{-SMe})_3\}$ core is retained in **2a**, that one MeCN is lost relative to **1a** and that the fourth bridge is unsymmetric. Two broad resonances were also detected at about δ 4 in C_6D_6 and assigned to the NH groups of the amidato ligand. The ^{13}C NMR spectrum of **2a** showed two signals at δ 180.0 and 179.5 which are attributable to the sp^2-hybridised carbon of the amidato group of each isomer.

Molecular structure of $[Mo_2Cp_2(\mu\text{-MeCONH})(\mu\text{-SMe})_3]$ 2a

Most structurally characterised amidato complexes are binuclear and contain a CH_3CONH group in bidentate $\mu\text{-}\eta^1:\eta^1$-coordination mode,[6–10] though monodentate bridging through N is also known,[9] and a single example of a $\mu\text{-}\eta^1:\eta^1\text{-}CH_3C(OH)N$ system has been described.[7a] The structure of **2a,** determined by X-ray analysis of orange crystals obtained from diethyl ether (Table 1 and Fig. 2), consists of a tris-(μ-thiolato)dimolybdenum unit $\{Mo_2Cp_2(\mu\text{-SMe})_3\}$ bridged by a bidentate $\mu\text{-}\eta^1:\eta^1\text{-}CH_3CONH$ group which is σ-bonded to the Mo_2 unit through its oxygen and nitrogen atoms [Mo1–O1 2.157(7), Mo2–N1 2.171(9) Å]. The O1–C4 and N1–C4 bond lengths of 1.269(12) and 1.293(12) Å are close to the average values for such bonds in transition metal amidato complexes [1.277(5) and 1.306(7) Å].[10] They suggest delocalisation of π electrons through the O1–C4–N1 linkage. The O1–C4–N1, O1–C4–C5, N1–C4–C5 angles [respectively 122.6(9), 116.5(9) and 120.9(9)°] are consistent with sp^2 hybridisation at C4. The Mo–Mo distance [2.719(2) Å] indicates a bond of unit order as required by electron counting rules; its length is virtually the same as that of 2.709(1) Å found in the related dimolybdenum(III) complex $[Mo_2Cp_2(\mu\text{-}O_2CCF_3)(\mu\text{-SMe})_3]$ where the trifluoroacetate plays the same $\mu\text{-}\eta^1:\eta^1$ bridging role as the amidate in **2a.**[11]

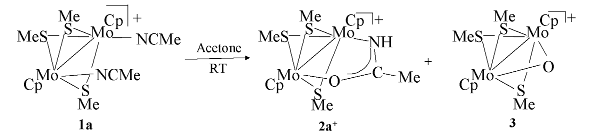
Reaction of 1a in acetone: synthesis of $2a^+$ and $[Mo_2Cp_2(\mu\text{-O})(\mu\text{-SMe})_3]BF_4$ 3

When **1a** was stirred in acetone for 1 h $\mathbf{2a^+}$ (yield: 41%) was again obtained, together with a second product **3** (yield: 55%)

Table 1 Selected bond lengths (Å) and angles (°) for complex **2a**

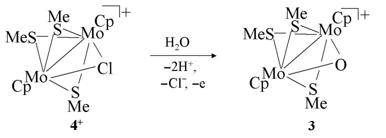
Mo(1)–Mo(2)	2.7186(14)	Mo(2)–N(1)	2.171(9)
Mo(1)–O(1)	2.157(7)	Mo(2)–S(1)	2.452(3)
Mo(1)–S(1)	2.453(3)	Mo(2)–S(2)	2.428(3)
Mo(1)–S(2)	2.430(3)	Mo(2)–S(3)	2.448(3)
Mo(1)–S(3)	2.428(3)	N(1)–C(4)	1.293(12)
O(1)–C(4)	1.269(12)	C(5)–C(4)	1.543(13)
Mo(2)–S(1)–Mo(1)	67.32(7)	N(1)–Mo(2)–Mo(1)	82.6(2)
Mo(2)–S(2)–Mo(1)	68.06(7)	N(1)–Mo(2)–S(1)	84.7(2)
Mo(2)–S(3)–Mo(1)	67.77(7)	N(1)–Mo(2)–S(2)	138.6(2)
O(1)–Mo(1)–Mo(2)	84.86(18)	N(1)–Mo(2)–S(3)	82.3(3)
O(1)–Mo(1)–S(1)	85.24(19)	S(1)–Mo(2)–S(3)	111.91(9)
O(1)–Mo(1)–S(2)	140.77(19)	S(1)–Mo(2)–S(2)	72.22(10)
O(1)–Mo(1)–S(3)	84.3(2)	C(4)–O(1)–Mo(1)	124.3(7)
S(1)–Mo(1)–Mo(2)	56.31(7)	C(4)–N(1)–Mo(2)	125.7(7)
S(2)–Mo(1)–Mo(2)	55.95(7)	O(1)–C(4)–N(1)	122.6(9)
S(3)–Mo(1)–Mo(2)	56.48(7)	O(1)–C(4)–C(5)	116.5(9)
S(1)–Mo(1)–S(2)	72.17(9)	N(1)–C(4)–C(5)	120.9(9)
S(1)–Mo(1)–S(3)	112.56(10)		

which was identified as the μ-oxo species $[Mo_2Cp_2(\mu\text{-O})(\mu\text{-SMe})_3]BF_4$. An unbalanced reaction for the synthesis of $\mathbf{2a^+}$ and **3** is shown in eqn. (1). The yields of $\mathbf{2a^+}$ and **3** did



(1)

not significantly change when water, either in stoichiometric amount or in large excess, was added to the reaction mixture. Complex **3** was also cleanly obtained in very good yields by hydrolysis of $[Mo_2Cp_2(\mu\text{-Cl})(\mu\text{-SMe})_3]BF_4$ $\mathbf{4^+}$ in aqueous solution: a balanced reaction requires the addition of oxide from water and loss of one electron (eqn. 2). Elemental analyses of **3**

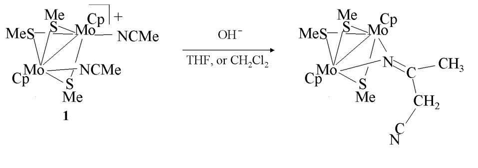


(2)

are consistent with the formula $C_{13}H_{19}BF_4Mo_2OS_3$ and the ^1H NMR spectrum shows one resonance for two equivalent cyclopentadienyl groups at about δ 7 and three signals in the SMe region which accords with a $\{Cp_2Mo_2(\mu\text{-SMe})_3\}$ core.

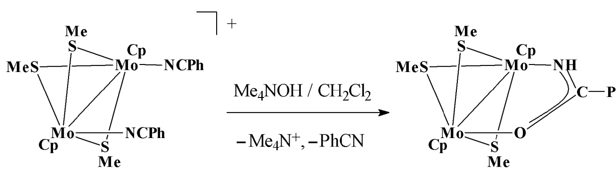
Reaction of $[Mo_2Cp_2(RCN)_2(\mu\text{-SMe})_3]BF_4$ (R = Me 1a or Ph 1b) and $[Mo_2Cp_2(\mu\text{-Cl})(\mu\text{-SMe})_3]$ 4 with hydroxide

Attempts at a more straightforward synthesis of **2a** by addition of OH^- to **1a** were not successful: under base hydrolysis the deprotonation of one acetonitrile in **1a** is followed by its nucleophilic addition to the other MeCN group to give rise to the azavinylidene species $[Mo_2Cp_2(\mu\text{-N=C(CH_3)CH_2CN})]$ (eqn. 3).[4]



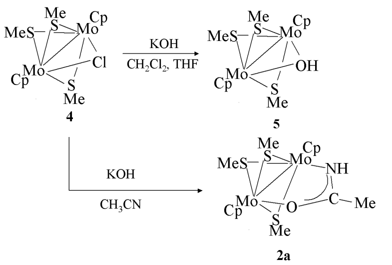
(3)

Interestingly, the analogous bis-benzonitrile compound $[Mo_2Cp_2(PhCN)_2(\mu\text{-SMe})_3]BF_4$ **1b,** in which the nitrile does not possess acidic protons, readily undergoes addition of hydroxide to a PhCN group to produce the complex $[Mo_2Cp_2(PhCONH)(\mu\text{-SMe})_3]$ **2b** which was identified by its elemental analyses and ^1H NMR spectrum (eqn. 4). It is worth noting that in acetonitrile solution the reaction of **4** with KOH affords **2a** as



(4)

the major product, whereas in other solvents (CH_2Cl_2, THF) **4** readily permits the substitution of chloride by hydroxide to give the unstable species $[Mo_2Cp_2(\mu\text{-}OH)(\mu\text{-}SMe)_3]$ **5** (eqn. 5) whose



(5)

^1H NMR spectrum displays a signal at δ −1.1 which could be assigned to the bridging hydroxide group.[12] Occasionally, in addition to **5** a minor side-product was also observed; it was identified as the tetrakis-thiolato bridged complex $[Mo_2Cp_2(\mu\text{-}SMe)_4]$ by comparison of its NMR spectra with those of an authentic sample.[13] This complex probably arises from reduction and decomposition of **1**.

Mechanistic consideration and conclusion

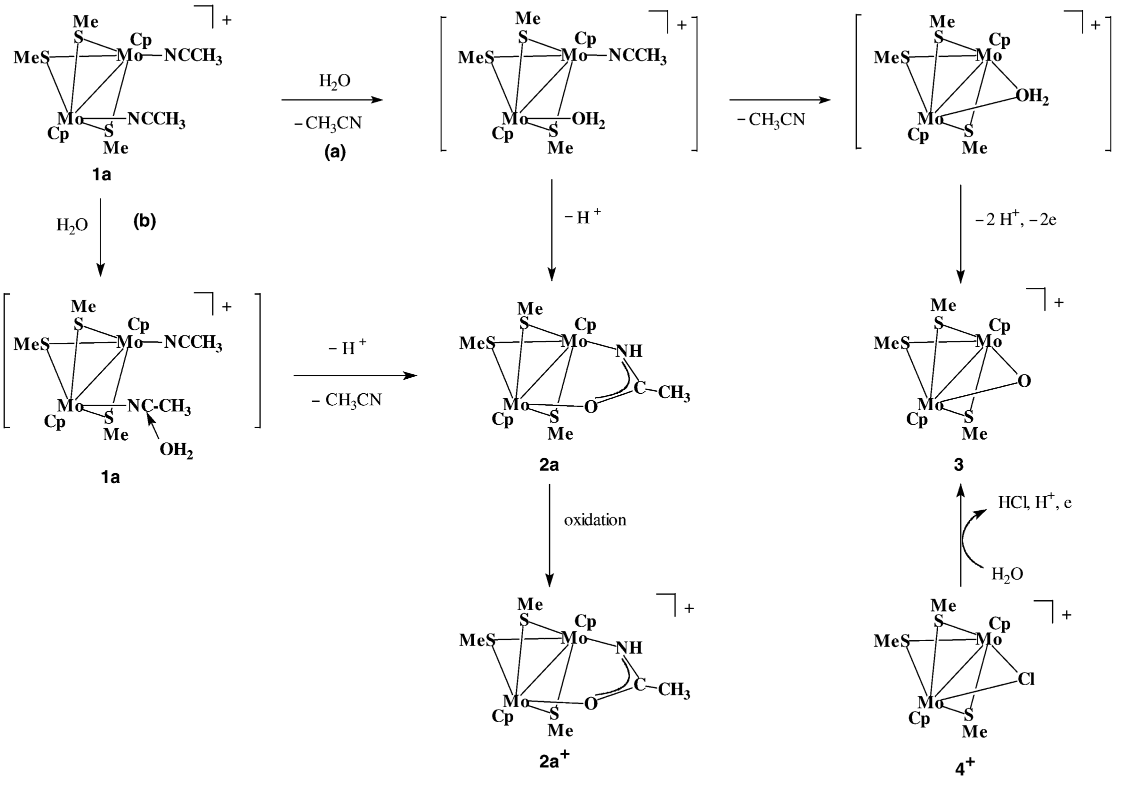
The commonly accepted mechanism of hydration of nitrile at a bimetallic site involves coordination of a water molecule to one metal centre followed by an intramolecular transfer of hydroxide to nitrile (Scheme 2, path a), but direct addition of H_2O to a nitrile has also been proposed (Scheme 2, path b).[6,7,14] In our experiments the final step in the reaction is the oxidation of neutral **2a** into $\mathbf{2a}^+$. An alternative pathway replaces the intramolecular hydroxide transfer by loss of the second acetonitrile, giving finally the oxo derivative **3**. The clean and quantitative transformation of the cationic chloro-bridged species $\mathbf{4}^+$ is noteworthy and may involve oxidation and deprotonation steps,[15] following the initial coordination of water to the dimolybdenum site with concomitant opening of the chloride bridge (Scheme 2, path c).[16]

It is obvious that the transformation of nitrile into an amidato ligand at the bimetallic site $\{Mo_2Cp_2(\mu\text{-}SMe)_3{}^+\}$ in the presence of hydroxide is limited by the possibility of deprotonation if the nitrile substrate has suitable hydrogen atoms available. It is possible that dissolving **4** in a suitable nitrile and adding hydroxide may also afford a direct pathway to bridging amidates. An attempt at catalytic transformation of acetonitrile into acetamide at the Mo_2 site was not successful because the reaction stopped at the amidato stage. Nevertheless, attempts to liberate the amidato ligand as an amide molecule were made: **1a** was readily and completely regenerated by addition of protons (HBF_4–H_2O) to acetonitrile solutions of **2a** and ^1H NMR and IR monitoring of the reaction mixture was consistent with the presence of acetamide in solution.

The reaction which gives rise to $\mathbf{2a}^+$ is a novel example of the activation by the $\{Mo_2Cp_2(\mu\text{-}SMe)_3\}^+$ core of an unsaturated substrate. Reactions of **1** with amines and alcohols may extend the scope of these transformations of nitrile at the tris-thiolato-bridged dimolybdenum(III) site.

Experimental

General experimental techniques were those we described recently.[16] Infrared spectra were obtained with a Perkin-Elmer 1430, and NMR spectra on a AC300, spectrophotometer. Chemical shifts are given on the δ scale relative to tetramethylsilane for ^1H and ^{13}C. The preparation and the purification of the supporting electrolyte NBu_4PF_6 and the electrochemical equipment were as described previously.[17] All the potentials (text, Fig. 1) are quoted against the ferrocene–ferrocenium couple; ferrocene was added as an internal standard at the end of the experiments. Chemical analyses were performed by the



Scheme 2 Possible pathway to the formation of 2^+ and **3**.

"Centre de Microanalyses du CNRS", Vernaison. Literature methods were used for the preparation of $[\mathrm{Mo_2Cp_2(\mu\text{-}Cl)(\mu\text{-}SMe)_3]BF_4}$ and $[\mathrm{Mo_2Cp_2(MeCN)_2(\mu\text{-}SMe)_3]BF_4}$.[18]

Preparations

$[\mathrm{Mo_2Cp_2(\mu\text{-}MeCONH)(\mu\text{-}SMe)_3}]$ 2a and $[\mathrm{Mo_2Cp_2(\mu\text{-}MeCONH)(\mu\text{-}SMe)_3}]^+$ 2a$^+$. On standing for 48 h in contact with air a red-brown powder of **1a** (0.2 g, 0.32 mmol) afforded a red-brown compound **2a$^+$** (0.19 g, 97%) which was used without further purification. Calc. for $\mathrm{C_{15}H_{23}BF_4Mo_2NOS_3}$: C, 29.6; H, 3.8. Found: C, 29.7; H, 3.8%.

2a$^+$ (0.19 g, 0.31 mmol) was dissolved in acetonitrile (10 mL) and treated at room temperature with an excess of $\mathrm{NaBH_4}$ (40 mg, 1.03 mmol). After stirring (5 min) the solvent was removed under vacuum and the residue extracted with diethyl ether affording **2a** as an orange powder (0.13 g, 80%). Calc. for $\mathrm{C_{15}H_{23}Mo_2NOS_3}$: C, 34.5; H, 4.4; N, 2.7. Found: C, 35.5; H, 4.6; N, 2.6%. Data for **2a**: IR (KBr pellets, $\mathrm{cm^{-1}}$) ν(NH) 3340 (w); ν(C=O) 1560(s); ^{1}H NMR ($\mathrm{C_6D_6}$) δ 5.22 and 4.83, 5.20 and 4.82 (4s, 4 × 5H, $\mathrm{C_5}H_5$), 4.00 and 3.89 (2s, broad, $\mathrm{Mo_2(\mu\text{-}CH_3CON}H$)), 1.77, 1.69, 1.55_9, 1.55_6, 1.54, 1.52, 1.46, 1.40 (8s, 8 × 3H, $\mathrm{Mo_2(\mu\text{-}C}H_3$CONH and $\mathrm{SC}H_3$); ^{13}C-{^{1}H} NMR ($\mathrm{C_6D_6}$) δ 180.0 and 179.5 ($\mathrm{Mo_2(\mu\text{-}CH_3}C$ONH), 93.5 and 90.9, 93.1 and 90.5 ($C_5\mathrm{H_5}$), 25.0, 23.7, 15.5, 15.2,14.9,13.9, 13.1, 12.5 ($\mathrm{Mo_2(\mu\text{-}}C\mathrm{H_3}$CONH and S$C\mathrm{H_3}$).

$[\mathrm{Mo_2Cp_2(PhCN)_2(\mu\text{-}SMe)_3]BF_4}$ 1b and $[\mathrm{Mo_2Cp_2(PhCONH)(\mu\text{-}SMe)_3}]$ 2b. The bis-benzonitrile complex **1b** was prepared in a way similar to that for **1a**[16] and characterised by elemental analyses and ^{1}H NMR spectroscopy. Calc. for $\mathrm{C_{27}H_{29}BF_4Mo_2N_2S_3}$: C, 42.9; H, 3.8; N, 3.7. Found: C, 42.9; H, 3.8; N, 3.8%. ^{1}H NMR ($\mathrm{CD_3COCD_3}$): δ 7.35 and 7.21 (m, 10H, $\mathrm{C_6}H_5$CN), 5.53 (s, 10H, $\mathrm{C_5}H_5$), 2.28 (s, 3H, $\mathrm{C}H_3$), 2.25 (s, 3H, $\mathrm{C}H_3$), 1.47 (s, 3H, $\mathrm{C}H_3$).

Complex **2b** was prepared by adding an equimolar amount of $\mathrm{Me_4NOH}$ (in MeOH solution) to a dichloromethane solution (15 ml) of **1b** (0.2 g, 0.26 mmol). The orange colour of the solution disappeared within 10 min. The solvent was then removed under vacuum. The residue was extracted twice with pentane (10 mL), affording **2b** as an orange powder (0.1 g, 68%) by slow evaporation of the solvent. Calc. for $\mathrm{C_{20}H_{25}Mo_2NOS_3}$: C, 41.2; H, 4.3; N, 2.4. Found: C, 40.4; H, 4.3; N, 2.5%. ^{1}H NMR (Toluene-$\mathrm{d_8}$): δ 7.1 and 7.0 (m, $\mathrm{Mo_2(\mu\text{-}}Ph$CONH)), 5.49 (s, $\mathrm{Mo_2(\mu\text{-}PhCON}H$)), 5.34 and 4.92 (s, 2 × 5H, $\mathrm{C_5}H_5$), 1.62 (s, 3H, $\mathrm{C}H_3$), 1.59 (s, 3H, $\mathrm{C}H_3$), 1.51 (s, 3H, $\mathrm{C}H_3$). Data for **3**: ^{1}H NMR ($\mathrm{CD_3COCD_3}$) δ 7.01 (s, 10H, $\mathrm{C_5}H_5$), 2.36 (s, 3H, $\mathrm{SC}H_3$), 1.85 (s, 3H, $\mathrm{SC}H_3$), 1.81 (s, 3H, $\mathrm{SC}H_3$).

$[\mathrm{Mo_2Cp_2(\mu\text{-}O)(\mu\text{-}SMe)_3]BF_4}$ 3. Complex **3** was prepared by stirring an aqueous solution of **4$^+$**[16] (0.2 g, 0.34 mmol) at 40 °C for 10 min. Two extractions with dichloromethane (2 × 10 mL) were then performed, affording **3** as a greenish powder (0.16 g, 83%) by evaporation of the solvent. Calc. for $\mathrm{C_{13}H_{19}BF_4Mo_2OS_3}$: C, 27.6; H, 3.4. Found: C, 27.3; H, 3.6%.

Alternatively, 0.1 g (0.16 mmol) of **1a** was stirred at room temperature for 1 h in acetone–water (10 : 4, 14 mL). The solvent was then removed under vacuum and the residue solubilized in THF–$\mathrm{Et_2O}$ (3 : 1): **3** precipitated as a greenish powder (0.05 g, 55%) from the solution. By evaporating the filtrate to dryness, **2a$^+$** (0.04 g, 41%) was isolated.

$[\mathrm{Mo_2Cp_2(\mu\text{-}OH)(\mu\text{-}SMe)_3}]$ 5. A THF–water (5 : 1) solution (10 mL) of **4** (0.15 g, 0.3 mmol) and an excess of KOH (6 equivalents) was stirred at room temperature for 4 h. Complex **5** was then extracted twice with ether (2 × 10 mL) from the mother solution. The solvent was removed to afford **5** as a brown powder in moderate yield (0.09 g, 62%). Small amounts of the complex $[\mathrm{Mo_2Cp_2(\mu\text{-}SMe)_4}]$ (up to 10%) were occasionally detected by ^{1}H NMR in samples of **5**. The two products were unseparable. A similar procedure but in MeCN solution afforded **2a**. Data for **5**: ^{1}H NMR ($\mathrm{CDCl_3}$) δ 5.45 (s, broad, 10H, $\mathrm{C_5}H_5$), 1.72 (s, 3H, $\mathrm{SC}H_3$), 1.30 (s, 3H, $\mathrm{SC}H_3$), 1.28 (s, 3H, $\mathrm{SC}H_3$), −1.10 (s, broad, 1H, OH).

Table 2 Crystallographic data for $[\mathrm{Mo_2Cp_2(\mu\text{-}MeCONH)(\mu\text{-}SMe)_3}]$ **2a**

Empirical formula	$\mathrm{C_{15}H_{23}Mo_2NOS_3}$
Formula weight	521.40
Crystal system	Orthorhombic
Space group	*Pbca*
a/Å	15.790(6)
b/Å	13.841(5)
c/Å	16.717(12)
V/Å^3	3653(3)
Z	8
μ/mm^{-1}	1.716
Reflections collected	5371
Independent reflections	3767 ($R_{\mathrm{int}} = 0.13$)
$R1$, $wR2$ [$I > 2\sigma(I)$]	0.062, 0.134 [1793 data]
[all data]	0.191, 0.158

Crystal structure determination of complex 2a

Crystal data for complex **2a** are summarised in Table 2. The crystals were grown in diethyl ether. Measurements were made at 20 °C with Mo-Kα radiation, $\lambda = 0.71073$ Å, using a Nonius CAD4 diffractometer. The structure was refined on F^2 using anisotropic U_{ij} values for all non-hydrogen atoms[19] The crystals available were of poor quality and the specimen size was less than optimum. The data set contains 52% unobserved reflections. In consequence R_{int} and the R factors on all the data are high. Despite this the H atom attached to N1 was successfully refined. The assignment of chemical types to O1 and N1 was made on the basis of the plausibility of the thermal ellipsoids.

CCDC reference number 161768.

See http://www.rsc.org/suppdata/dt/b1/b100986i/ for crystallographic data in CIF or other electronic format.

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