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Synthesis and electrochemistry of dimolybdenum crown ether imido complexes

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Abstract

The complexes [(η-MeC$_5$H$_4$)$_2$Mo$_2$O$_2$(μ-O){μ-N(benzo-15-crown-5)}] (**1**) and [(η-MeC$_5$H$_4$)Mo(O){μ-N(benzo-15-crown-5)}]$_2$ (**2**) have been prepared by reaction of 4′-nitrobenzo-15-crown-5 with [(η-MeC$_5$H$_4$)Mo(CO)$_3$]$_2$. Compounds **1** and **2** react with phenyl isocyanate to yield [(η-MeC$_5$H$_4$)$_2$Mo$_2$(NPh)$_2$(μ-NPh){μ-N(benzo-15-crown-5)}] (**3**) and [(η-MeC$_5$H$_4$)Mo(NPh){μ-N(benzo-15-crown-5)}]$_2$ (**4**) respectively. Acid hydrolysis of **3** yields [(η-MeC$_5$H$_4$)$_2$Mo$_2$O$_2$(μ-NPh){μ-N(benzo-15-crown-5)}] (**5**), whilst hydrolysis of **4** gives **2**. The electrochemistry of compounds **1**, **2** and **3** has been investigated by cyclic voltammetry in acetonitrile. Compound **1** undergoes an irreversible oxidation and an irreversible reduction. Compound **2** undergoes two irreversible oxidations, the first showing reversible behaviour at high scan rates, indicative of an EC mechanism. Compound **3** undergoes a reversible oxidation. Addition of excess sodium cations causes a significant anodic shift in the potential of this oxidation.

Introduction

Recently there has been considerable interest in the coupling of cation binding to shifts of the redox potential of crown ether derivatives of redox-active organometallic compounds. Crown ether derivatives of ferrocene [1–7], di-η-cyclopentadienyl-molybdenum and -tungsten complexes [8], and tris(3,5-dimethylpyrazoyl)hydroborate molybdenum compounds [9] exhibit shifts of redox potential, $E_{1/2}$, upon alkali metal cation binding by the crown ether moiety of up to 320 mV. Intimate contact between the crown ether moiety and the electroactive centre is imperative if large shifts of $E_{1/2}$ upon cation binding are to be observed. In this respect those compounds where the HOMO (in the case of an oxidation) or the LUMO (reduction) are significantly ligand based may show the largest shifts of $E_{1/2}$.

Alper and co-workers have reported the synthesis of trioxoarylimidobis(η-cyclopentadienylmolybdenum) complexes from the reaction between nitro- and nitroso-arenes and [(η-C$_5$H$_5$)Mo(CO)$_2$]$_2$ [10]. More recently, we have reported the synthesis and electrochemistry of the oxo and imido dimolybdenum complexes [(η-MeC$_5$H$_4$)Mo(X)(μ-Y)]$_2$ (X = NPh, O, S; Y = NPh, O) [11,12]. Molecular orbital calculations on the model compound $[(\eta\text{-}C_5H_5)Mo(NH)(\mu\text{-}NH)]_2$ revealed that the HOMO was principally ligand based. It seemed worthwhile to prepare crown ether substituted derivatives of these ligands and study the effect of alkali metal cations upon the redox potentials. Herein we report the synthesis and study of some dimolybdenum imido complexes containing benzo-15-crown-5 groups.

Results and discussion

The reaction between 4′-nitrobenzo-15-crown-5 and $[(\eta\text{-}MeC_5H_4)Mo(CO)_3]_2$ in refluxing toluene gave a mixture of products, from which the monoimido complex $[(\eta\text{-}MeC_5H_4)_2Mo_2O_2(\mu\text{-}O)\{\mu\text{-}N(\text{benzo-15-crown-5})\}]$ (**1**) and the bisimido complex $[(\eta\text{-}MeC_5H_4)MoO\{\mu\text{-}N(\text{benzo-15-crown-5})\}]_2$ (**2**) were separated by chromatography and isolated in 16 and 25% yields, respectively. Their identities were confirmed by elemental analysis, and infrared and ^1H NMR spectroscopy (Table 1). Thus the infrared spectrum of **1** showed strong bands at 1264, 894 and 813 cm^{-1} assignable to the bridging imido, terminal oxo and bridging oxo stretches respectively [11,13–15], while the infrared spectrum of **2** showed strong absorptions at 1263 and 894 cm^{-1}. Compounds **1** and **2** are soluble in common organic solvents and appear stable in air in the solid state but solutions show decomposition within hours.

Heating THF solutions of **1** and **2** with excess of phenyl isocyanate resulted in the substitution of all oxo atoms for phenylimido ligands giving $[(\eta\text{-}MeC_5H_4)_2Mo_2(NPh)_2(\mu\text{-}NPh)\{\mu\text{-}N(\text{benzo-15-crown-5})\}]$ (**3**) and $[(\eta\text{-}MeC_5H_4)Mo(NPh)\{\mu\text{-}N(\text{benzo-15-crown-5})\}]_2$ (**4**), respectively. The compounds **3** and **4** are orange microcrystalline solids and their infrared spectra showed strong bands in the regions 1320–1323 and 1260–1263 cm^{-1} assignable to terminal and bridging imido moieties respectively. The ^1H NMR spectra are entirely consistent with the structures shown in Scheme 1. An analytically pure sample of **4** was not obtained due to the presence of isocyanate oligomers formed during the reaction, which could not be separated.

In a reaction analogous to the hydrolysis of $[(\eta\text{-}MeC_5H_4)Mo(NPh)(\mu\text{-}NPh)]_2$ giving $[(\eta\text{-}MeC_5H_4)MoO(\mu\text{-}NPh)]_2$ [11] compound **3** was hydrolysed cleanly by the addition of aqueous acid to give the yellow, air-stable bisimido complex $[(\eta\text{-}MeC_5H_4)_2Mo_2O_2(\mu\text{-}NPh)\{\mu\text{-}N(\text{benzo-15-crown-5})\}]$ (**5**). Thus the overall transformation of **1** to **5** is that of a selective substitution of a bridging oxo atom. Complex **4** underwent a similar hydrolysis reaction resulting in the regeneration of **2**.

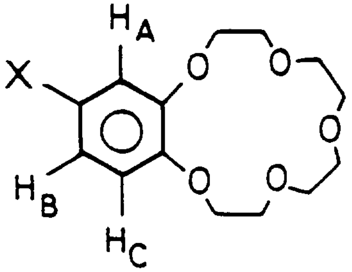
The electrochemistry of compounds **1**, **2** and **3** was investigated by cyclic voltammetry in acetonitrile with 0.1 *M* tetra-n-butylammonium hexafluorophosphate as electrolyte at a scan rate ν of 50 mV s^{-1}. The results are presented in Table 2.

Compound **1** underwent an irreversible reduction, and an irreversible oxidation which gave rise to smaller irreversible waves at −580 and −1050 mV. This electrochemical behaviour is similar to that observed for the phenylimido analogue $[(\eta\text{-}MeC_5H_4)_2Mo_2O_2(\mu\text{-}O)(\mu\text{-}NPh)]$ [16].

Compound **2** underwent two irreversible oxidations, which gave rise to several other irreversible waves. The first oxidation wave at +820 mV showed a small cathodic current at a scan rate of 50 mV s^{-1}, with a value of the peak separation for the wave, $E_p^a - E_p^c$ of 60 mV. At faster scan rates this wave began to show more

Table 1

^{1}H NMR data for compounds **1–5**

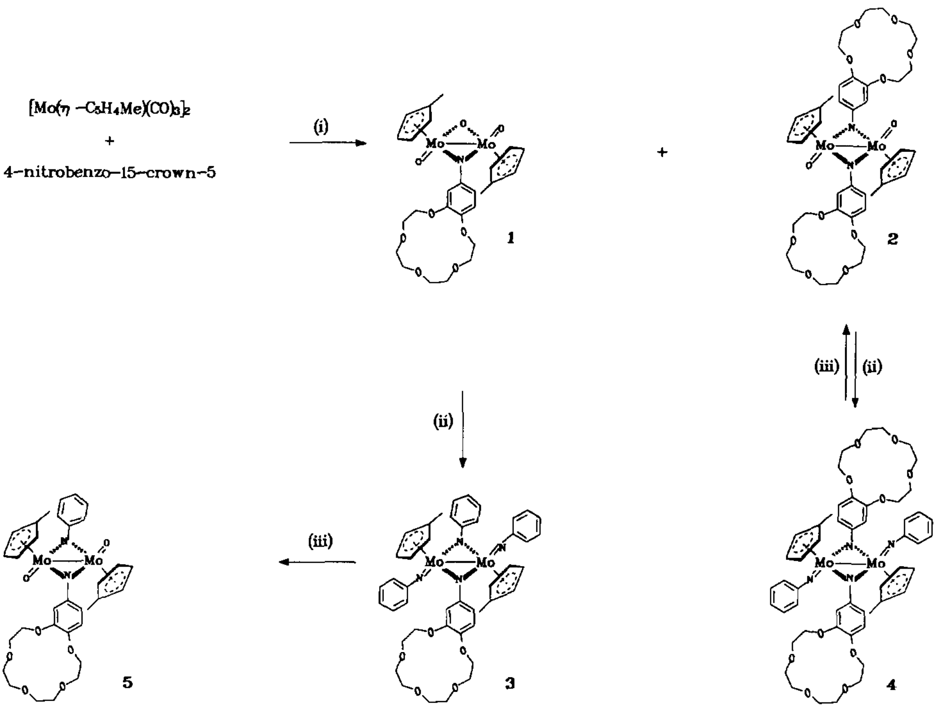


Labelling scheme for benzo-15-crown-5 compounds

Compound	^{1}H NMR data [a]
1	7.35 (m, 2H, H_a and H_b), 7.02 [d, $^{3}J(H_b-H_c)$ 8.7, 1H, H_c], 6.04 (m, 4H, η-C_5H_4), 5.81 (m, 2H, η-C_5H_4), 5.37 (m, 2H, η-C_5H_4), 4.29 (m, 4H, $ArO\mathit{CH}_2$), 4.00 (m, 4H, $ArOCH_2\mathit{CH}_2$), 3.83 (m, 8H, OCH_2), 1.92 (s, 6H, CH_3).
2	7.46 [d, $^{4}J(H_a-H_b)$ 2.4, 2H, H_a], 7.43 [dd, $^{3}J(H_b-H_c)$ 8.3, $^{4}J(H_a-H_b)$ 2.4, 2H, H_b], 7.05 [d, $^{3}J(H_b-H_c)$ 8.3, 2H, H_c], 5.69 [vt, J(H–H) 2.5, 4H, η-C_5H_4], 5.48 [vt, J(H–H) 2.5, 4H, η-C_5H_4], 4.36 (m, 4H, $ArO\mathit{CH}_2$), 4.29 (m, 4H, $ArO\mathit{CH}_2$), 4.02 (m, 8H, $ArOCH_2\mathit{CH}_2$), 4.85 (m, 16H, OCH_2), 1.77 (s, 6H, CH_3).
3	7.72 [dd, $^{3}J(H_{o'}-H_{m'})$ 8.3, $^{4}J(H_{o'}-H_{p'})$ 1.1, 2H, $H_{o'}$], 7.45 [dd, $^{3}J(H_{o'}-H_{m'})$ 8.3, $^{3}J(H_{m'}-H_{p'})$ 6.9, 2H, $H_{m'}$], 7.40 [dd, $^{3}J(H_b-H_c)$ 7.8, $^{4}J(H_a-H_b)$ 1.4, 1H, H_b], 7.24 [d, $^{4}J(H_a-H_b)$ 1.4, 1H, H_a], 7.15 [dd, $^{3}J(H_o-H_m)$ 8.3, $^{3}J(H_m-H_p)$ 7.8, 4H, H_m], 7.06 [tt, $^{3}J(H_{m'}-H_{p'})$ 6.9, $^{4}J(H_{o'}-H_{p'})$ 1.1, 1H, $H_{p'}$], 7.00 [d, $^{3}J(H_b-H_c)$ 7.8, 1H, H_c], 6.89 [tt, $^{3}J(H_m-H_p)$ 7.8, $^{4}J(H_o-H_p)$ 1.1, 2H, H_p], 6.80 [dd, $^{3}J(H_o-H_m)$ 8.3, $^{4}J(H_o-H_p)$ 1.1, 4H, H_o], 6.00 [vt, J(H–H) 2.5, 4H, η-C_5H_4], 5.33 (m, 2H, η-C_5H_4), 5.25 (m, 2H, η-C_5H_4), 4.26 (m, 4H, $ArO\mathit{CH}_2$), 4.00 (m, $ArOCH_2\mathit{CH}_2$), 3.82 (m, 8H, OCH_2), 1.25 (s, 6H, CH_3).
4	6.7–7.5 (m, C_6H_5N and $NC_6H_3O_2$), 5.97 [vt, J(H–H) 2.5, 4H, η-C_5H_4], 5.25 [vt, J(H–H) 2.5, 4H, η-C_5H_4], 4.25 (m, 8H, $ArO\mathit{CH}_2$), 4.00 (m, 8H, $ArOCH_2\mathit{CH}_2$), 3.84 (m, 16H, OCH_2), 1.24 (s, 6H, CH_3).
5	7.91 [d, $^{3}J(H_o-H_m)$ 7.4, 2H, H_o], 7.57 [dd, $^{3}J(H_o-H_m)$ 7.4, $^{3}J(H_m-H_p)$ 8.2, 2H, H_m], 7.44 [d, $^{4}J(H_a-H_b)$ 2.3, 1H, H_a], 7.40 [dd, $^{3}J(H_b-H_c)$ 8.4, $^{4}J(H_a-H_b)$ 2.3, H_b], 7.14 [t, $^{3}J(H_m-H_p)$ 8.2, 1H, H_p], 7.05 [d, $^{3}J(H_b-H_c)$ 8.4, 1H, H_c], 5.75 [vt, J(H–H) 2.2, 2H, η-C_5H_4], 5.70 [vt, J(H–H) 2.2, 2H, η-C_5H_4], 5.50 (m, 4H, η-C_5H_4), 4.37 (m, 2H, $ArO\mathit{CH}_2$), 4.29 (m, 2H, $ArO\mathit{CH}_2$), 4.03 (m, 4H, $ArOCH_2\mathit{CH}_2$), 3.84 (m, OCH_2), 1.77 (s, 6H, CH_3).

[a] Recorded in chloroform-*d*.

reversible behaviour, with the peak current ratio, i_p^a/i_p^c, approaching unity at 2000 mV s^{-1}, suggesting an EC reaction mechanism. This was confirmed by E_p^a shifting to more anodic potentials and $i_p^a\nu^{-1/2}$ decreasing slightly with increasing ν [17,18]. The second oxidation did not show such behaviour. This result is similar to that of the trisphenylimidooxo complex, $[(\eta\text{-}MeC_5H_4)_2Mo_2(NPh)O(\mu\text{-}NPh)_2]$, which also undergoes an oxidation showing an EC mechanism at high scan rate [16]. Addition



Scheme 1. (i) in toluene at 110 °C for 8 h; (ii) PhNCO in THF at 70 °C for 19 h; (iii) $H^+_{(aq)}$ in acetone.

of an excess of sodium tetrafluoroborate caused the potential of the first oxidation to shift anodically by +150 to +970 mV.

Compound **3** underwent a reversible one-electron oxidation and an irreversible oxidation. Addition of ca. one equivalent of sodium tetrafluoroborate caused an anodic shift of the potential of the reversible wave of 55 mV. Addition of an excess of sodium tetrafluoroborate further shifted the potential by 40 mV.

Unfortunately the presence of impurities in compound **4** precluded an electrochemical study.

These studies show that the binding of sodium cations by the crown ether rings of compounds **2** and **3** can effect the electronic nature of the complexes and cause significant anodic shifts of the potentials for oxidation, which may be a conse-

Table 2

Electrochemical data for compounds **1**, **2** and **3** [a]

Compound	Reduction (E_p^c (mV))	Oxidations (E_p^a (mV))	
1	−1965 (irrev)	+915 (irrev)	–
2	–	+820 [b]	+1150 (irrev)
3	–	+390 (rev) [c]	+685 (irrev)
3 + Na^+ (1 equiv)	–	+445 (rev) [d]	+755 (irrev)
3 + Na^+ (excess)	–	+485 (rev) [d]	+755 (irrev)

[a] Recorded in acetonitrile at 50 mV s^{-1}, vs. SCE. [b] EC mechanism indicated by behaviour at high scan rate. [c] $E_{1/2}$, $E_p^a - E_p^c = 85$ mV. [d] $E_{1/2}$, $E_p^a - E_p^c = 105$ mV.

quence of the ligand-based nature of the HOMO. The shift in $E_{1/2}$ of 95 mV observed in compound **3** on addition of sodium cations can be compared with those of 60 mV for [(η-C_5H_5)Fe(η-C_4H_4-benzo-15-crown-5)] [3] and [HB{$N_2C_3(CH_3)_2$}$_3$MoCl$_2$NH(benzo-15-crown-5)] [9], in which the effect of the sodium cation is also transmitted through a benzenoid ring to the electroactive centre.

Experimental

All preparations and reactions were carried out under nitrogen by standard Schlenk-line techniques. All solvents were thoroughly deoxygenated before use by repeated evacuation followed by admission of nitrogen. Solvents were predried over activated molecular sieves and then distilled under nitrogen from over potassium [tetrahydrofuran(THF)], sodium (toluene), sodium–potassium alloy [light petroleum (b.p. 40–60 °C throughout)], phosphorus pentoxide (dichloromethane), calcium hydride followed by phosphorus pentoxide (acetonitrile). Elemental analyses were performed by the Analytical Department of this laboratory. Chromatography was performed on columns of deactivated alumina (6% water) made up in light petroleum.

Infrared spectra were recorded as CsI pellets on a Perkin–Elmer 1510 Infrared Fourier Transform spectrometer. ^1H NMR spectra were determined at 300 MHz using a Bruker AM300 spectrometer. Spectra were referenced internally using the solvent resonances relative to tetramethylsilane ($\delta = 0$ ppm). All chemical shifts are quoted in δ (ppm) and coupling constants in Hertz (Hz). Abbreviations used in multiplicities are: s = singlet, d = doublet, t = triplet, vt = virtual triplet, m = multiplet.

Cyclic voltammetry experiments were performed on an Oxford Instruments potentiostat and recorded on a Rikadenki X–Y chart recorder. A two compartment cell was used, in which the reference electrode was isolated by a medium grade porous frit. A platinum disc (diameter 3 mm) sealed in glass was used as the working electrode. A platinum gauze served as the counter electrode. A platinum "pigtail" electrode was used as a quasi-reference electrode [19]. Ferrocene was added at the end of each experiment as an internal reference; its potential being taken as +355 mV relative to the saturated calomel electrode (SCE) [20]. All potentials are relative to SCE. Experiments were carried out at ambient temperatures (20 ± 5 °C).

Tetra-n-butylammonium hexafluorophosphate (Aldrich) was recrystallized from THF and dried *in vacuo*. Sodium tetrafluoroborate (Aldrich) was dried by heating (> 100 °C) *in vacuo* for several hours. 4′-Nitrobenzo-15-crown-5 [21] and [(η-MeC_5H_4)Mo(CO)$_3$]$_2$ [22] were prepared by the literature methods.

[(η-MeC_5H_4)$_2$$Mo_2O_2$($\mu$-O){$\mu$-N(benzo-15-crown-5)}] (1) and [(η-MeC_5H_4)MoO{μ-N(benzo-15-crown-5)}]$_2$ (2)

A solution of 4′-nitrobenzo-15-crown-5 (0.86 g, 2.75 mmol) and [(η-MeC_5H_4)Mo(CO)$_3$]$_2$ (1.17 g, 2.3 mmol) in toluene (150 cm^3) was refluxed with a nitrogen purge. A yellow-brown colour developed. When the infrared spectra showed no further change in the intensity of the carbonyl stretching frequencies at 1909 and 1953 cm^{-1} (ca. 8 h), heating was stopped and the solvent was removed under reduced pressure. Chromatography with dichloromethane/methanol (0.5%) as eluant separated unchanged starting material. With dichloromethane/methanol (2%) as eluant two yellow bands were eluted. Removal of the solvent under reduced pressure from the first band and recrystallization from dichloromethane/light petroleum gave **1** as a microcrystalline orange solid. Yield 0.25 g (16%). Analysis. Found: C, 46.5; H, 5.1; N, 2.2. $C_{26}H_{33}NO_8Mo_2$ calc.: C, 46.0; H, 4.9; N, 2.1%. IR (CsI pellet): 1264, ν(Mo–N); 894, ν(Mo=O); 813 cm^{-1}, ν(Mo–O).

Removal of the solvent under reduced pressure from the second band, and recrystallization from dichloromethane/light petroleum gave yellow needles of **2**. Yield 0.25 g (12%). Analysis. Found: C, 50.4; H, 5.5; N, 2.8. $C_{40}H_{52}N_2O_{12}Mo_2$ calc.: C, 50.85; H, 5.55; N, 3.0%. IR (CsI pellet): 1263, ν(Mo–N); 877 cm^{-1}, ν(Mo=O).

$[(\eta\text{-MeC}_5\text{H}_4)_2\text{Mo}_2(\text{NPh})_2(\mu\text{-NPh})\{\mu\text{-N(benzo-15-crown-5)}\}]$ *(3)*

Phenyl isocyanate (ca. 0.7 g, 6 mmol) was added to **1** (0.22 g, 0.3 mmol) in THF (30 cm^3). The orange solution was heated at 70 °C for 19 h after which time the colour had darkened. The solvent was removed under reduced pressure and the product extracted with light petroleum (4 × 50 cm^3). Repeated recrystallizations from light petroleum gave pure **3** as a dark orange solid. Yield ca. 0.10 g (37%). Analysis. Found: C, 58.95; H, 5.3; N, 6.5. $C_{44}H_{48}N_4O_5Mo_2$ calc.: C, 58.4; H, 5.35; N, 6.2%. IR (CsI pellet): 1321, ν(Mo=N); 1263 cm^{-1}, ν(Mo–N).

$[(\eta\text{-MeC}_5\text{H}_4)\text{Mo}(\text{NPh})\{\mu\text{-N(benzo-15-crown-5)}\}]_2$ *(4)*

Compound **4** was prepared similarly to compound **3** using **2** (0.07 g, 0.08 mmol) and phenyl isocyanate (ca. 0.60 g, 5 mmol). Repeated recrystallizations from light petroleum failed to give satisfactory analyses. Characterization was based on spectroscopic data (Table 1) and by comparison with analogous compounds [11] and its hydrolysis to give **2**. IR (CsI pellet): 1323, ν(Mo=N); 1260 cm^{-1}, ν(Mo–N).

$[(\eta\text{-MeC}_5\text{H}_4)_2\text{Mo}_2\text{O}_2(\mu\text{-NPh})\{\mu\text{-N(benzo-15-crown-5)}\}]$ *(5)*

Concentrated hydrochloric acid (ca. 10 cm^3) was added to compound **3** (ca. 0.02 g, 0.02 mmol) in acetone (30 cm^3) to give a dark red solution. Water (30 cm^3) was added, giving a yellow solution, and the product extracted with dichloromethane (3 × 50 cm^3). The extracts were dried over anhydrous magnesium sulphate, filtered, and the solvent removed from the filtrate under reduced pressure affording a small amount of **5** as a yellow solid. Characterization was based on spectroscopic data (Table 1) and by comparison with compound **2** and other analogous compounds [11]. IR (CsI pellet): 1262, ν(Mo–N); 880 cm^{-1}, ν(Mo=O).

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