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Novel Molybdenum Complexes with the 3,6-Di-*tert*-Butylcatecholate Ligand

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Abstract—A reaction of the cluster $(Et_4N)_2[Mo_3S_7Br_6]$ with potassium 3,6-di-*tert*-butyl-*o*-benzosemiquinolate (KSQ^{36}) afforded the mononuclear complexes $(Et_4N)[Mo(Cat^{36})_3]$ (**I**) and $(Et_4N)[MoO(Cat^{36})_2(HCat^{36})]$ (**II**) with prismatic and octahedral environments of their central metal atoms, respectively. Complex **I** was also obtained by a reaction between $MoCl_5$, sodium 3,6-di-*tert*-butylcatecholate (Na_2Cat^{36}), and Et_4NBr. Complexes **I** and **II** were characterized by X-ray diffraction and EPR spectroscopy. The structures have been deposited with the Cambridge Structural Database (nos. 1009139 (**I**), 1009140 (**I** · 1.5THF), and 1009141 (**II** · Et_2O)).

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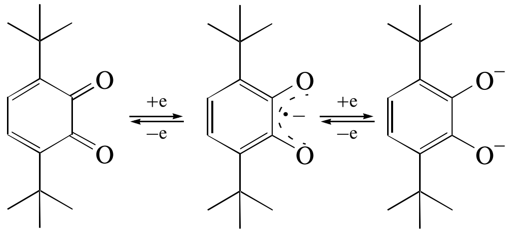
INTRODUCTION

In the last few decades, considerable progress has been made in the chemistry of complexes with so-called redox-active ligands that can exist in several oxidation states when coordinated to a metal atom [1–3]. 1,2-Benzoquinone derivatives are classic examples of such ligands; they tend to undergo stepwise reduction to the *o*-semiquinolate radical anion and the catecholate dianion.



Q^{36} $(SQ^{36})^-$ $(Cat^{36})^{2-}$

Coordination with such a ligand allows redox transformations of metal complexes if several oxidation states are not characteristic of the metal. Examples of reactions that are atypical of Main Group metal complexes include addition of molecular oxygen to antimony complexes [4–6], addition of NO to zinc and lead complexes [7], and addition of stable radicals and alkyl halides to tin [8, 9], gallium [10], and indium compounds [11]. In these reactions, oxidative addition involves the π-system of the ligand without changing the formal oxidation state of the central metal atom. When a metal and a ligand are both redox-active, so-called redox isomerism may result from a thermo- or photoinduced reversible intramolecular electron transfer between the metal and the ligand [12, 13]. Redox isomerism was first discovered just in cobalt [14] and rhodium [15] complexes with *o*-benzoquinone derivatives. Interest in redox isomerism is due to the possibility of using such systems in the design of molecular sensors and nanosized data recording and storage devices.

Polynuclear complexes (especially those of second- and third-row transition metals) with redox-active ligands are less studied. Coordination of 1,2-dithio- and diselenolenes to the cluster framework Mo_3Q_7 (Q = S or Se) has been reported; electroless or electrochemical oxidation of dithiolene complexes gives rise to electron-conducting phases [16–18]. On the other hand, the only documented example of coordination of *ortho*-benzoquinone derivatives with Mo_3Q_7 refers to the complex $(Ph_4P)_2[Mo_3S_7(CatCl_4)_3]$ ($CatCl_4$ is tetrachlorocatecholate) [19]. The goal of this study was to obtain complexes in which 3,6-di-*tert*-butyl-1,2-benzoquinone is coordinated to the cluster framework Mo_3S_7.

EXPERIMENTAL

All syntheses were carried out in an inert atmosphere using standard Schlenk ware. Solvents employed for synthetic purposes were dried and degassed by refluxing and distillation under an inert gas in the presence of appropriate desiccants [20]. The starting compounds $(Et_4N)_2[Mo_3S_7Br_6]$ [21] and 3,6-

di-*tert*-butyl-*o*-benzoquinone [22] were prepared according to known procedures. IR spectra (KBr) were recorded on a SCIMITAR FTS 2000 instrument. EPR spectra (*X* range) were recorded on a Bruker EMX spectrometer at 293 K with diphenylpicrylhydrazyl ($g = 2.0037$) as a standard for determination of the *g* factor. Simulation of EPR spectra was performed with the WinEPR SimFonia program.

Synthesis of $(Et_4N)[Mo(Cat^{36})_3]$ (I). Method 1. A mixture of $(Et_4N)_2[Mo_3S_7Br_6]$ (120 mg, 0.096 mmol) and 3,6-di-*tert*-butyl-*o*-benzoquinone (66 mg, 0.300 mmol) was placed in a Schlenk vessel, and THF (~25 mL) was added by recondensation under reduced pressure. Then metallic potassium (15 mg, 0.388 mmol) was added. The vessel was cooled, evacuated, and heated at 55°C for 36 h. The reaction mixture was cooled and filtered through a glass filter (G4). The filtrate was concentrated, and organic materials were extracted from a dark residue with diethyl ether (~20 mL). The extract was filtered through a glass filter (G4) into an H-shaped Schlenk vessel. Slow evaporation of the filtrate gave complex **I** as dark blue crystals suitable for X-ray diffraction. The yield of complex **I** was 11 mg (13%).

Method 2. The yellow solution prepared by reduction of 3,6-di-*tert*-butyl-*o*-benzoquinone (488 mg, 2.22 mmol) with excess metallic sodium (~200 mg, 8.70 mmol) was placed in an evacuated Schlenk vessel containing $MoCl_5$ (200 mg, 0.73 mmol). The vessel was heated at 60°C for 20 h. On cooling, Et_4NBr (160 mg, 0.76 mmol) was added, and the reaction mixture was stirred at room temperature for 12 h. The resulting solution was filtered through a glass filter (G4) into an H-shaped Schlenk vessel. Slow evaporation of the filtrate gave complex **I** · 1.5THF as dark violet crystals suitable for X-ray diffraction. The yield was 318 mg (49%).

UV-Vis (hexane; λ_{max}, nm (logε)): 206 (4.85), 268 (4.25), 361 (4.15), 482 (4.31), 610 (4.11), 927 (3.75).

IR (KBr; ν, cm^{-1}): 2954 s, 2906 s, 2866 s, 1582 m, 1535 w, 1484 m, 1463 m, 1391 m, 1359 m, 1340 m, 1284 m, 1200 m, 1170 w, 1124 w, 1028 w, 983 s, 944 m, 925 m, 856 w, 811 m, 711 m, 646 m, 596 s, 506 m, 471 w.

For $C_{56}H_{92}NO_{7.5}Mo$

anal. calcd., %:	C, 67.43;	H, 9.30;	N, 1.41.
Found, %:	C, 66.95;	H, 9.10;	N, 1.60.

The complex $(Et_4N)[MoO(Cat^{36})_2(HCat^{36})]$ (II) was obtained in low yield as dark blue crystals (solvate **II** · Et_2O) under the conditions described for the synthesis of complex **I** (method 1).

IR (KBr; ν, cm^{-1}): 2954 s, 2905 s, 2865 m, 1613 w, 1580 w, 1541 w, 1483 s, 1469 m, 1393 s, 1360 m, 1268 m, 1243 m, 1201 m, 1170 w, 1143 w, 1028 w, 983 s, 975 s, 943 m, 903 s, 807 m, 798 m, 715 s, 655 m, 645 m, 600 s, 579 m, 549 w, 507 w, 476 w, 462 w, 434 w.

(–)-ES-MS (CH_3CN, *m/z*): 775.35 ($[MoO(Cat^{36})_2(HCat^{36})]^-$, 100%).

X-ray diffraction study. Crystallographic and diffraction data for structure determination were collected on a Bruker X8 Apex CCD automated diffractometer (graphite monochromator, $\lambda(MoK_\alpha) = 0.71073$ Å). An absorption correction was applied semiempirically with the SADABS program [23]. The structures were solved by the direct methods and refined anisotropically by the least-squares method (SHELXTL) [24]. The hydrogen atoms were located geometrically and refined under rigid body constraints. Selected crystallographic parameters and the data collection and refinement statistics are given in Table 1. Selected bond lengths and bond angles are listed in Table 2.

The coordinates of the non-hydrogen atoms have been deposited with the Cambridge Structural Database (nos. 1009139 (**I**), 1009140 (**I** · 1.5THF), and 1009141 (**II** · Et_2O)) and can be retrieved from http://www.ccdc.cam.ac.uk/data_request/cif or can be made available from the authors upon request.

RESULTS AND DISCUSSION

We found that the cluster $(Et_4N)_2[Mo_3^{IV}S_7Br_6]$ does not react with 3,6-di-*tert*-butyl-1,2-benzoquinone in THF. Addition of metallic potassium to the reaction mixture results in the formation of potassium 3,6-di-*tert*-butyl-*o*-semiquinolate (KSQ^{36}). However, the expected coordination of the *o*-semiquinolate radical anion with $(Et_4N)_2[Mo_3S_7Br_6]$ does not occur. Instead, the cluster decomposes to the mononuclear complex $(Et_4N)[Mo^V(Cat^{36})_3]$ (**I**), in which the oxidation state of molybdenum is increased from +4 to +5. 3,6-Di-*tert*-butyl-1,2-benzoquinone and the 3,6-di-*tert*-butyl-1,2-benzosemiquinolate radical anion present in the reaction mixture are possible oxidants. The black residue insoluble in organic solvents seems to be molybdenum sulfide or sulfur complexes. The complex $(Et_4N)[Mo^{VI}O(Cat^{36})_2(HCat^{36})]$ (**II**) was obtained under the same conditions as was complex **I**. This is indicative of the formation of multiple products by decomposition of the cluster. Earlier [25–27], heteroleptic molybdenum oxocatecholates have been detected in a reaction of $Mo(CO)_6$ with an appropriate quinone. The Mo–Mo bond cleavage in the presence of *o*-benzoquinone derivatives has already been observed: $[Mo(OAc)_2]_2$ reacts with tetrachloropyrocatechol to give the anionic complex $[MoO(CatCl_4)_2(Thf)]^-$ [28]. In contrast, a reaction of the cluster complex $(Ph_4P)_2[Mo_3S_7(CatCl_4)_3]$ with iodine results in oxidation and de-coordination of the catecholate ligands, the cluster framework Mo_3S_7 remaining intact [19].

The knowledge of structure **I** allowed us to propose a rational method for the synthesis of this complex by

Table 1. Selected crystallographic parameters and the data collection and refinement statistics for structures **I**, **I** · 1.5THF, and **II** · Et_2O

Parameter	Value		
	I	**I** · $1.5C_4H_8O$	**II** · Et_2O
Empirical formula	$C_{50}H_{80}NO_6Mo$	$C_{112}H_{184}N_2O_{15}Mo_2$	$C_{54}H_{91}NO_8Mo$
M	887.09	1990.49	977.21
Crystal system; space group	Monoclinic; $P2_1/c$	Monoclinic; $C2/c$	Monoclinic; $P2_1/c$
Temperature, K	150	150	150
a, Å	11.3273(4)	84.724(17)	19.2436(6)
b, Å	20.7227(6)	12.547(3)	14.7370(7)
c, Å	20.8290(8)	21.489(4)	19.9320(9)
β, deg	93.711(1)	102.11(3)	98.151(1)
V, Å^3	4879.0(3)	22334(8)	5595.5(4)
Z	4	8	4
F(000)	1908	8592	2112
μ, mm^{-1}	0.315	0.284	0.283
Crystal dimensions, mm	0.20 × 0.18 × 0.13	0.28 × 0.14 × 0.08	0.21 × 0.10 × 0.09
T_{min}, T_{max}	0.940, 0.960	0.925, 0.978	0.943, 0.975
θ Scan range, deg	1.80–26.37	1.47–28.01	1.72–26.37
Ranges of h, k, and l indices	$-14 \le h \le 14, -14 \le k \le 25, -26 \le l \le 25$	$-106 \le h \le 106, -16 \le k \le 16, -26 \le l \le 26$	$-24 \le h \le 14, -18 \le k \le 18, -24 \le l \le 24$
Number of measured, unique, and observed ($I > 2\sigma(I)$) reflections	31289, 9954, 8129	112950, 24701, 17306	36365, 11396, 8310
R_{int}	0.033	0.056	0.041
R_1, wR_2 ($I > 2\sigma(I)$)	0.0375, 0.0894	0.0429, 0.0870	0.0464, 0.1186
R_1, wR_2 (for all reflections)	0.0518, 0.0935	0.0770, 0.0971	0.0741, 0.1269
GOOF	1.032	1.011	1.062
Number of parameters refined	545	1269	601
Number of constraints	0	0	30
$\Delta\rho_{max}$, $\Delta\rho_{min}$, e Å^{-3}	0.83, –0.65	0.55, –0.62	0.83, –0.62

a reaction of $MoCl_5$ with sodium 3,6-di-*tert*-butylcatecholate (Na_2Cat^{36}) followed by addition of Et_4NBr.

Structures **I** and **II** were determined from X-ray diffraction data (Figs. 1, 2). The crystals of complex **I** obtained in diethyl ether belong to the space group $P2_1/c$; its crystallization from THF produces a solvate (space group $C2/c$) containing two crystallographically independent complex molecules. In both crystalline phases, the coordination polyhedron of the Mo atom may be approximated by a trigonal prism. Earlier, the prismatic environment of the Mo atom has been found in the complex $Mo(PQ)_3$ (PQ is 9,10-phenanthrenequinone) [29]. However, the Mo atom in that complex deviates considerably from the plane of one ligand. As a result, the dihedral angle between the plane of MoO_2 and the plane of this phenanthrenequinone ligand is 60.3(6)° and the Mo–C bonds are shortened (2.42 Å). In contrast of $Mo(PQ)_3$, the Mo atom in complexes **I** and **I** · 1.5THF is nearly coplanar with the catecholate ligands (its maximum deviation does not exceed 0.25 Å; the angle between the planes of MoO_2 and the benzene ring is 9.4°). It should be noted that the trigonal prism as a coordination polyhedron in metal tris-*o*-quinone complexes is very uncommon. Only three examples of such a coordination fashion, apart from $Mo(PQ)_3$, have been reported by now for cobalt [30] and indium tris-*o*-semiquinolates [31, 32].

Other known examples of Mo(VI) tris-catecholates include the dimeric complexes $[Mo(CatCl_4)_2]_2(\mu\text{-}CatCl_4)_2$ [33] and $[Mo(Cat^{35})_3]_2$ (Cat^{35} is 3,5-di-*tert*-butylcatecholate) [34] with $C.N._{Mo} = 6$ and 7, respectively. Apparently, the dimeric structure is unfavorable for complex **I** because of steric overcrowding of the metal center when moving from $CatCl_4$ and Cat^{35} to Cat^{36}. Tris-*o*-quinone complexes of manganese, cobalt, and nickel are also dimeric for 3,5-di-*tert*-butyl-*o*-benzoquinone derivatives and monomeric for Q^{36} derivatives [12]. Note that Nordlander, Pierpont

Table 2. Selected parameters of the coordination polyhedra and the chelate rings in structures **I** and **II**

Parameter	**I**	**I** · $1.5C_4H_8O$		**II** · Et_2O
	Bond, Å			
Mo–O_{cat}	Mo(1)–O(11) 1.9720(15)	Mo(1)–O(11) 1.9728(16)	Mo(2)–O(41) 1.9703(16)	Mo(1)–O(11) 1.9578(19)
	Mo(1)–O(12) 1.9679(15)	Mo(1)–O(12) 1.9718(16)	Mo(2)–O(42) 1.9698(16)	Mo(1)–O(12) 1.9603(19)
	Mo(1)–O(21) 1.9585(15)	Mo(1)–O(21) 1.9730(15)	Mo(2)–O(51) 1.9732(16)	Mo(1)–O(21) 1.9534(19)
	Mo(1)–O(22) 1.9871(15)	Mo(1)–O(22) 1.9674(16)	Mo(2)–O(52) 1.9695(16)	Mo(1)–O(22) 2.1189(18)
	Mo(1)–O(31) 1.9794(15)	Mo(1)–O(31) 1.9759(16)	Mo(2)–O(61) 1.9693(16)	Mo(1)–O(31) 1.9273(18)
	Mo(1)–O(32) 1.9687(15)	Mo(1)–O(32) 1.9710(16)	Mo(2)–O(62) 1.9753(16)	Mo(1)–O(12) 1.9603(19)
Mo=O				Mo(1)–O(1) 1.6989(19)
	Bite angle, deg			
O_{cat}–Mo–O_{cat}	O(11)Mo(1)O(12) 77.26(6)	O(11)Mo(1)O(12) 77.16(6)	O(41)Mo(2)O(42) 77.08(7)	O(11)Mo(1)O(12) 77.85(8)
	O(21)Mo(1)O(22) 76.87(6)	O(21)Mo(1)O(22) 77.13(6)	O(51)Mo(2)O(52) 76.93(6)	O(21)Mo(1)O(22) 74.69(8)
	O(31)Mo(1)O(32) 76.84(6)	O(31)Mo(1)O(32) 77.14(6)	O(61)Mo(2)O(62) 77.08(6)	

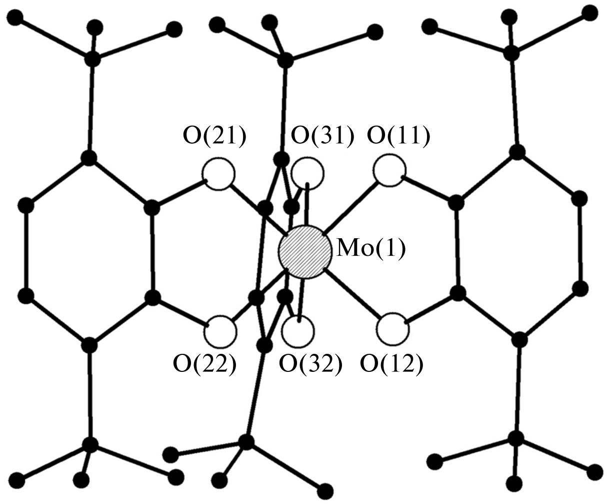


Fig. 1. Structure of the anion in complex **I**. The H atoms are omitted.

et al. [26, 27] failed to obtain the homoleptic tris-catecholate complex Mo(Cat^{36})$_3$: a reaction of Mo(CO)$_6$ with 3,6-di-*tert*-butyl-*o*-benzoquinone produces the tetranuclear oxo complex [Mo(Cat^{36})$_2$O]$_4$. The average Mo–O$_{cat}$ bond lengths in structures **I** and **I** · 1.5THF (1.972(10) and 1.971(2) Å, respectively) are shorter than those in the dimer [Mo(Cat^{35})$_3$]$_2$ (2.02(5) Å).

Crystallization of complex **II** yields a solvate (space group $P2_1/c$) with a diethyl ether molecule. The coordination polyhedron MoO$_6$ is a distorted octahedron. The Mo–O$_{cat}$ bond that is *trans* to the Mo=O bond is substantially longer (2.1189(18) Å) than the other bonds (Table 2). One catecholate ligand is monoprotonated and coordinated to molybdenum only through one O atom. The H atom is hydrogen-bonded to an adjacent catecholate (O(32)···O(22) 2.638(3), H(32)···O(22) 1.87 Å; angle O(32)–H(32)···O(22) 151°). The Mo atom is considerably (by 0.92 Å) off the plane of the benzene ring of the protonated catecholate; the dihedral angle between the plane Mo(1)O(31)O(32) and the plane of the benzene ring is 31°. The presence of one monoprotonated and two deprotonated catecholates in the complex anion is confirmed by the mass spectrum (ESI) of complex m/z 775.35.

As in complex **I**, the C–C (1.34–1.37 Å) and C–O bond lengths (1.39–1.42 Å) in the chelate rings MoOCCO of structure **II** correspond to the expected values for coordinated catecholates [35], thus confirming the redox state of the ligand.

An EPR study provides evidence for the electron density distribution in structure **I**. This complex is paramagnetic in the crystalline state as well as in solution. The isotropic EPR spectrum of complex **I** in THF (Fig. 3) consists of a singlet with satellite bands due to splitting on the magnetic isotopes of molybdenum: ^{95}Mo (15.92%, $I = 5/2$, $\mu_N = 0.9133$) and ^{97}Mo (9.55%, $I = 5/2$, $\mu_N = 0.9335$) [36]. The EPR parameters ($a_i(^{95,97}\text{Mo}) = 50.45$ G, $g_i = 1.954$) agree with the d^1-configuration of the central Mo(V) atom [36] surrounded by three diamagnetic 3,6-di-*tert*-butylcatecholate dianions. According to EPR data, complex **II** is diamagnetic.

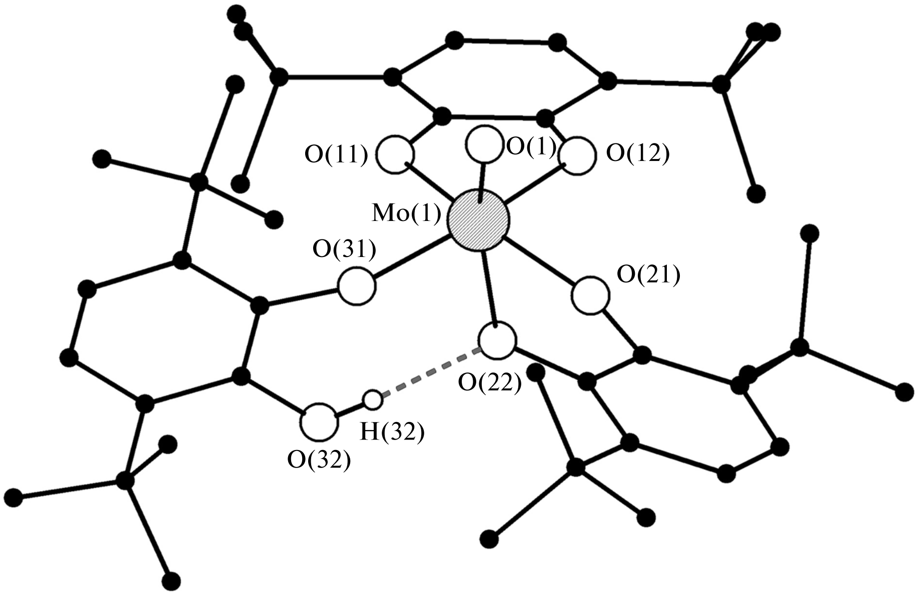


Fig. 2. Structure of the anion in complex **II**. The H atoms of the benzene rings and those of the *tert*-butyl groups are omitted.

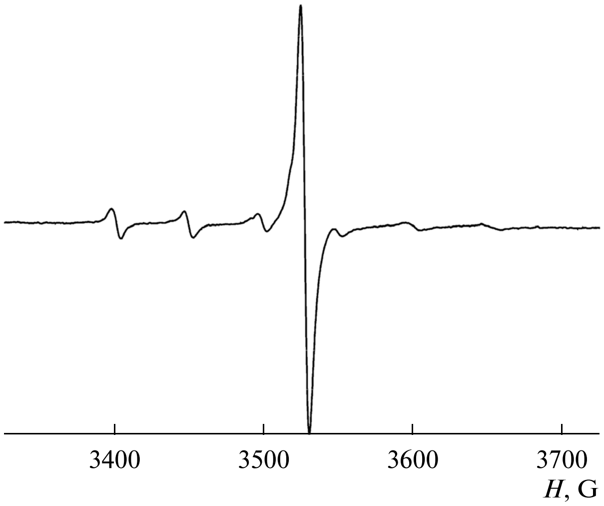


Fig. 3. Isotropic EPR spectrum of complex **I** (THF, 293 K).

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