Without additional information, however, any further speculation concerning the mechanistic activation process in the present system is unwarranted.

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**Registry No.** $\beta$-*cis*-Co(EDDA)$(OH_2)_2^+$, 26135-75-1.

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# *o*-Quinone Coordination to *cis*-Dioxomolybdenum(VI) Species. Crystal and Molecular Structure of *cis*-Dioxodichloro(9,10-phenanthrenequinone)molybdenum(VI)

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## Introduction

Benzoquinones constitute a pervasive component of the environment. They occur naturally in higher plants, fungi, bacteria, and the animal kingdom.[1] Humic substances are probably the most widely distributed natural products on the earth's surface and contain oxidized and reduced quinone functional groups.[2,3] Polycyclic quinones have been found as atmospheric contaminants over major cities.[4] *o*-Quinones in both their reduced (catecholate) and oxidized forms are ideal chelating agents for transition metals, and this property appears naturally in many diverse functions. Hydroquinone complexes of $Fe^{3+}$ serve as microbial iron transport agents in specific systems.[5] Metal–catecholate complexes appear as intermediates in the function of catechol dioxygenases.[6] Humic acids function as chelating agents for a wide variety of metal ions.[2]

Much recent interest has been focused on the $\pi$-acceptor activity of *o*-quinone ligands coupled with nucleophilic metals.[7] However, the earliest reports of *o*-quinone coordination concern simple adducts of the metal ions $Zn^{2+}$, $Hg^{2+}$, $Fe^{3+}$, and $Mo^{6+}$.[8,9] In these complexes the ligand behaves as a simple oxygen donor. Characterization of quinone ligands in these compounds has been confined to little more than infrared analysis. The *cis*-dioxomolybdenum(VI) species forms complexes with both oxidized and reduced quinone ligands. Semiquinone[10] and catecholate[11,12] complexes of this type have been characterized structurally. We now report the synthesis of the 9,10-phenanthrenequinone adduct of $MoO_2Cl_2$ and its molecular structure.[13] The results of this investigation are compared with the structural features of other quinone, semiquinone, and catecholate complexes of Mo(VI).

## Experimental Section

**Crystal Preparation and Data Collection.** The complex $MoO_2Cl_2(O_2C_{14}H_8)$ was prepared by addition of excess 9,10-phenanthrenequinone to an anhydrous dichloromethane solution of $MoO_2Cl_2$. Slow evaporation of the solvent gave dark green, monoclinic crystals of the complex. A crystal of dimensions 0.26 × 0.22 × 0.11 mm was mounted and aligned on a Syntex P$\bar{1}$ automated diffractometer. Preliminary precession photographs showed monoclinic symmetry with systematic absences ($h0l$, $l \neq 2n$; $0k0$, $k \neq 2n$) consistent with space group $P2_1/c$-$C_{2h}^5$. The settings of 15 reflections with $2\theta$ values greater than 25° were used to give the refined cell constants $a = 6.991$ (1), $b = 10.227$ (1), $c = 21.179$ (3) Å, and $\beta = 107.52$ (2)°. An experimental density of 1.863 (7) g/cm$^3$ agrees with a calculated value of 1.872 g/cm$^3$ for four molecules per unit cell. A complete set of intensity data ($hkl$, $hk\bar{l}$) was collected within the angular range $3° \leq 2\theta \leq 50°$ using Mo K$\alpha$ radiation. The $\theta$–$2\theta$ scan mode was used with a fixed scan rate of 2.0°/min. Scans ranged from 0.6° below the K$\alpha_1$ $2\theta$ setting to 0.7° above K$\alpha_2$. Four check reflections were monitored after every 95 reflections measured. Their variation in intensity over the time required to collect data was less than 2.5%. Values for $F_o^2$ and $\sigma(F_o^2)$ were calculated for the 3083 reflections measured using procedures described previously.[14] Absorption effects were judged to be insignificant with $\mu = 12.4$ cm$^{-1}$.

**Solution and Refinement of the Structure.** The position of the Mo atom was determined from a Patterson map. Phases derived from the Mo position were used to locate all other nonhydrogen atoms of the structure. Positions of the quinone hydrogen atoms were calculated using the refined positions of the ring carbon atoms and an assumed C–H bond length of 0.98 Å. In final cycles of refinement hydrogens were refined as individual atoms. Full-matrix least-squares refinement of the complete structure with anisotropic thermal parameters for nonhydrogen atoms converged with $R_F = 0.029$ and $R_{wF} = 0.040$. In all calculations only the 2192 values with $F_o^2 \geq 3\sigma(F_o^2)$ were included. The final value for the error in an observation of unit weight is 1.58. Atomic scattering factors for the nonhydrogen atoms were taken from Cromer and Waber,[15] and hydrogen scattering factors were taken from Stewart et al.[16] Corrections for anomalous dispersion were applied for the Mo and Cl atoms with values of $\Delta f'$ and $\Delta f''$ taken from Cromer and Liberman.[17] Final positional and thermal parameters for nonhydrogen atoms are presented in Table I. Tables containing positional and thermal parameters of hydrogen atoms and observed and calculated structure factor amplitudes are available in the supplementary material.

## Description of the Structure

The crystal structure of $MoO_2Cl_2(O_2C_{14}H_8)$ consists of stacks of molecules along the crystallographic $b$ axis. A view of the complex molecule is shown in Figure 1. Bond lengths and angles are presented in Table II. Closest intermolecular contacts along the screw axis occur for Cl(1) with separations of 3.34 and 3.37 Å to carbonyl carbons C(1) and C(2) of the adjacent molecule (Figure 2). Chlorine Cl(2) interacts weakly with ring carbons C(9) and C(14) of the lower molecule in Figure 2 with contacts of 3.60 and 3.45 Å, respectively. The somewhat stronger Cl(1) contact contributes to a longer Mo–Cl(1) length of 2.370 (3) Å compared with the Mo–Cl(2) value of 2.343 (3) Å. Both chloro ligands are bent toward the weakly coordinated quinone by bonding electron-pair repulsions with the strongly bound oxo ligands. The Cl(1)–Mo–Cl(2) angle is 156.3 (1)° with angles between chloro ligands and quinone oxygens of approximately 80°. The cis oxo ligands have characteristically short Mo–O lengths of 1.671 (3) Å with a O(3)–Mo–O(4) angle of 104.8 (2)°. Much the same geometry has been reported for the $MoO_2Cl_2$ portion of $MoO_2Cl_2(DMF)_2$.[18] Phenanthrenequinone oxygens are

**Table I.** Positional and Thermal Parameters ($\times 10^4$) for the Nonhydrogen Atoms in $MoO_2Cl_2(O_2C_{14}H_8)$

| | $x^a$ | $y$ | $z$ | $\beta_{11}{}^b$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| Mo | 0.04849 (4) | 0.13399 (3) | 0.14578 (1) | 207.4 (9) | 86.0 (4) | 13.6 (1) | −23.3 (4) | 18.1 (2) | −4.3 (1) |
| Cl(1) | 0.20481 (13) | −0.05096 (9) | 0.20741 (5) | 205.2 (20) | 92.5 (10) | 27.1 (3) | 3.7 (11) | 17.0 (6) | −2.8 (4) |
| Cl(2) | −0.13350 (16) | 0.32990 (10) | 0.12627 (5) | 335.1 (29) | 90.4 (10) | 26.3 (3) | 4.4 (13) | 13.2 (7) | 3.2 (4) |
| O(1) | −0.1308 (3) | 0.1077 (2) | 0.2201 (1) | 159 (5) | 95 (3) | 16 (1) | −26 (3) | 9 (1) | −7 (1) |
| O(2) | 0.2048 (3) | 0.2353 (2) | 0.2452 (1) | 185 (5) | 98 (3) | 16 (1) | −36 (3) | 20 (1) | −5 (1) |
| O(3) | −0.1070 (4) | 0.0497 (3) | 0.0837 (1) | 325 (8) | 110 (3) | 20 (1) | −22 (4) | 0 (2) | −12 (1) |
| O(4) | 0.2381 (5) | 0.1780 (3) | 0.1176 (1) | 347 (8) | 162 (4) | 26 (1) | −41 (5) | 58 (2) | 2 (1) |
| C(1) | −0.0568 (5) | 0.1428 (3) | 0.2781 (2) | 145 (7) | 63 (3) | 17 (1) | 2 (4) | 13 (2) | 0 (1) |
| C(2) | 0.1403 (4) | 0.2189 (3) | 0.2929 (1) | 159 (7) | 60 (3) | 15 (1) | 1 (4) | 13 (2) | 0 (1) |
| C(3) | 0.2399 (4) | 0.2602 (3) | 0.3591 (1) | 146 (7) | 61 (3) | 16 (1) | 7 (4) | 8 (2) | −2 (1) |
| C(4) | 0.4208 (5) | 0.3291 (4) | 0.3722 (2) | 186 (8) | 77 (4) | 21 (1) | −6 (4) | 12 (2) | −5 (2) |
| C(5) | 0.5241 (6) | 0.3621 (4) | 0.4363 (2) | 191 (9) | 86 (4) | 27 (1) | −4 (5) | −7 (3) | −12 (2) |
| C(6) | 0.4507 (6) | 0.3269 (4) | 0.4868 (2) | 275 (11) | 91 (4) | 19 (1) | 22 (5) | −13 (3) | −10 (2) |
| C(7) | 0.2702 (6) | 0.2595 (4) | 0.4739 (2) | 297 (10) | 89 (4) | 14 (1) | 26 (5) | 13 (2) | −2 (1) |
| C(8) | 0.1608 (5) | 0.2258 (3) | 0.4104 (2) | 187 (8) | 58 (3) | 16 (1) | 25 (4) | 13 (2) | −1 (1) |
| C(9) | −0.0333 (5) | 0.1549 (3) | 0.3960 (2) | 193 (8) | 61 (3) | 18 (1) | 28 (4) | 24 (2) | 4 (1) |
| C(10) | −0.1174 (6) | 0.1213 (4) | 0.4454 (2) | 285 (11) | 96 (4) | 20 (1) | 50 (5) | 37 (3) | 13 (2) |
| C(11) | −0.2939 (6) | 0.0490 (4) | 0.4311 (2) | 280 (11) | 107 (5) | 32 (1) | 43 (6) | 58 (3) | 23 (2) |
| C(12) | −0.3910 (6) | 0.0100 (4) | 0.3688 (2) | 217 (9) | 101 (4) | 38 (1) | 4 (5) | 49 (3) | 15 (2) |
| C(13) | −0.3168 (5) | 0.0425 (4) | 0.3170 (2) | 183 (8) | 86 (4) | 28 (1) | −6 (4) | 31 (2) | 1 (2) |
| C(14) | −0.1379 (5) | 0.1150 (3) | 0.3312 (2) | 153 (7) | 67 (3) | 19 (1) | 14 (4) | 18 (2) | 2 (1) |

[a] Estimated standard deviations of the least significant figures are given in parentheses. [b] Anisotropic thermal parameters are in the form $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$.

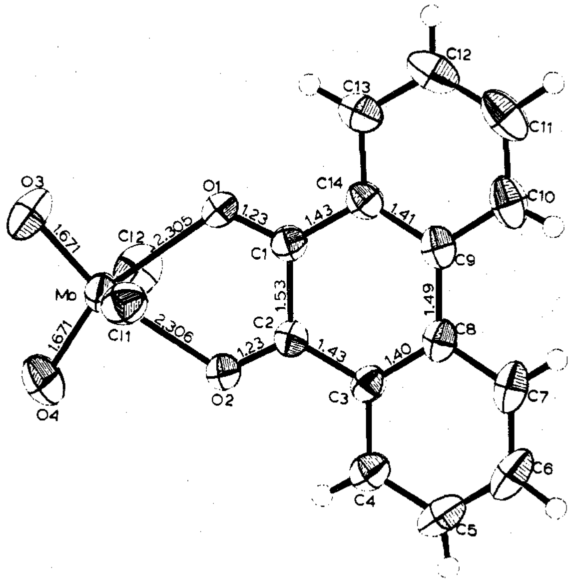


**Figure 1.** Perspective view of the $MoO_2Cl_2(O_2C_{14}H_8)$ molecule.

**Table II.** Inter- and Intramolecular Distances and Angles for $MoO_2Cl_2(O_2C_{14}H_8)$

| | | | |
|---|---|---|---|
| | Distances, A | | |
| Mo–Cl(1) | 2.370 (3) | C(7)–C(8) | 1.377 (5) |
| Mo–Cl(2) | 2.343 (3) | C(8)–C(9) | 1.487 (6) |
| Mo–O(1) | 2.305 (3) | C(9)–C(10) | 1.388 (5) |
| Mo–O(2) | 2.306 (3) | C(9)–C(14) | 1.406 (5) |
| Mo–O(3) | 1.671 (3) | C(10)–C(11) | 1.391 (7) |
| Mo–O(4) | 1.671 (3) | C(11)–C(12) | 1.349 (6) |
| O(1)–C(1) | 1.233 (4) | C(12)–C(13) | 1.389 (5) |
| O(2)–C(2) | 1.235 (4) | C(13)–C(14) | 1.407 (6) |
| C(1)–C(2) | 1.530 (5) | C(14)–C(1) | 1.433 (5) |
| C(2)–C(3) | 1.429 (4) | O(1)---O(2) | 2.596 (7) |
| C(3)–C(4) | 1.400 (6) | Cl(1)---C(1)[a] | 3.341 (7) |
| C(3)–C(8) | 1.404 (5) | Cl(1)---C(2)[a] | 3.369 (7) |
| C(4)–C(5) | 1.374 (5) | Cl(1)---C(3)[a] | 3.570 (8) |
| C(5)–C(6) | 1.367 (7) | Cl(1)---C(14)[a] | 3.511 (7) |
| C(6)–C(7) | 1.392 (7) | Cl(2)---C(14)[b] | 3.447 (6) |
| | | Cl(2)---C(9)[b] | 3.600 (7) |
| | Angles, Deg | | |
| O(1)–Mo–O(2) | 68.5 (1) | O(4)–Mo–Cl(2) | 98.9 (2) |
| O(1)–Mo–O(3) | 96.8 (2) | Cl(1)–Mo–Cl(2) | 156.3 (1) |
| O(1)–Mo–O(4) | 158.2 (1) | Mo–O(1)–C(1) | 119.7 (2) |
| O(1)–Mo–Cl(1) | 77.6 (1) | Mo–O(2)–C(2) | 119.8 (2) |
| O(1)–Mo–Cl(2) | 80.9 (1) | O(1)–C(1)–C(2) | 115.7 (3) |
| O(2)–Mo–O(3) | 165.3 (1) | O(1)–C(1)–C(14) | 125.3 (3) |
| O(2)–Mo–O(4) | 89.8 (2) | O(2)–C(2)–C(1) | 115.4 (3) |
| O(2)–Mo–Cl(1) | 80.9 (1) | O(2)–C(2)–C(3) | 124.9 (3) |
| O(2)–Mo–Cl(2) | 81.9 (1) | C(1)–C(2)–C(3) | 119.6 (3) |
| O(3)–Mo–O(4) | 104.8 (2) | C(2)–C(3)–C(8) | 119.7 (3) |
| O(3)–Mo–Cl(1) | 96.0 (2) | C(2)–C(1)–C(14) | 119.0 (3) |
| O(3)–Mo–Cl(2) | 96.5 (2) | C(3)–C(8)–C(9) | 120.8 (3) |
| O(4)–Mo–Cl(1) | 97.3 (2) | C(1)–C(14)–C(9) | 119.7 (3) |
| | | C(8)–C(9)–C(14) | 121.0 (3) |

[a] Adjacent molecule ($\bar{x}, -{}^1/_2 + y, {}^1/_2 - z$). [b] Adjacent molecule ($\bar{x}, {}^1/_2 + y, {}^1/_2 - z$).

weakly bound to the metal with Mo–O lengths of 2.305 (3) and 2.306 (3) Å consistent with the strong trans influence of oxo ligands and the weak donor activity of the ketonic quinone oxygens. Molybdenum–oxygen lengths for the DMF ligands in $MoO_2Cl_2(DMF)_2$ are 0.1 Å shorter, with values of 2.20 (1) Å. Lability of the quinone ligand in $MoO_2Cl_2(O_2C_{14}H_8)$ has inhibited study of the complex in solution.

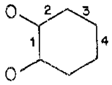
**Quinone Coordination in *cis*-Dioxomolybdenum(VI) Complexes.** Structural reports have appeared for *cis*-dioxomolybdenum(VI) complexes with catecholate ($MoO_2(O_2C_6H_4)_2{}^{2-}$, $Mo_2O_5(O_2C_6H_4)_2{}^{2-}$)[11,12] and semiquinone ($Mo_2O_5(O_2C_{14}H_8)_2$)[10] ligands (Table III). Formal ligand charge is reflected in carbon–oxygen bond lengths: 1.41 and 1.36 Å for catecholates, 1.31 Å for semiquinone ligands, and 1.234 (4) Å for the unreduced quinone in $MoO_2Cl_2(O_2C_{14}H_8)$. Carbon–carbon lengths at ring positions which correspond to bonds C(1)–C(2) and C(8)–C(9) in the present structure are also sensitive to ligand charge. For catecholate ligands these values are consistent with the aromatic character of the ring while in the present structure they have localized single bond values of 1.530 (5) and 1.487 (6) Å. Corresponding bonds in $Mo_2O_5(O_2C_{14}H_8)_2$ have intermediate values of 1.420 (8) and 1.432 (9) Å between carbonyl carbons and values of 1.465 (9) and 1.469 (9) Å for the bonds opposite quinone C–C bonds. Ring bonds C(1)–C(14) and C(2)–C(3) of the phenanthrenequinone ligand in $MoO_2Cl_2(O_2C_{14}H_8)$, which might be expected to have nearly single bond lengths, have values of 1.433 (5) and 1.429 (4) Å. However, partial delocalization over upper and lower regions of the quinone ring appears as a general feature of *o*-quinones.[19]

Oxygen donor strength at positions trans to oxo ligands is also dependent upon ligand charge. The binuclear complexes

**Table III.** Structural Parameters for *cis*-Dioxomolybdenum(VI) Complexes with *o*-Quinone Ligands

| | $MoO_2Cl_2(O_2C_{14}H_8)$ | $Mo_2O_5(O_2C_{14}H_8)_2$[10] | $Mo_2O_5(O_2C_6H_4)_2^{2-}$[11] | $MoO_2(O_2C_6H_4)_2^{2-}$[12] |
|---|---|---|---|---|
| Mo–$O_Q$, Å | 2.305 (3) | 2.041 (5),[a] 2.242 (5),[b] 2.495 (5)[b] | 1.98,[a] 2.16,[b] 2.37[b] | 2.05 (2),[c] 2.15 (2)[d] |
| Mo–$O_{(oxo)}$, Å | 1.671 (3) | 1.686 (5) | 1.70 | 1.77 (2) |
| C–O, Å | 1.234 (4) | 1.31 (1) | 1.36 | 1.39 (3) |
| $(C–C)_1$, Å[e] | 1.530 (5) | 1.43 (1) | 1.39 | 1.41 (3) |
| $(C–C)_2$, Å | 1.431 (5) | 1.43 (1) | 1.42 | 1.36 (3) |
| $(C–C)_3$, Å | 1.405 (5) | 1.41 (1) | 1.40 | 1.42 (3) |
| $(C–C)_4$, Å | 1.487 (6) | 1.47 (1) | 1.41 | 1.36 (3) |
| $O_Q$–Mo–$O_Q$, deg | 68.5 (1) | 73.8 (2) | *f* | 75 (1) |
| $O_{(oxo)}$–Mo–$O_{(oxo)}$, deg | 104.8 (2) | 105.0 (2) | *f* | 101 (1) |

[a] Terminal quinone oxygen. [b] Bridging quinone oxygens. [c] Position trans to catecholate oxygen. [d] Position trans to an oxo ligand. [e] Ring carbon–carbon bonds are numbered sequentially:



[f] Bond angles not reported.

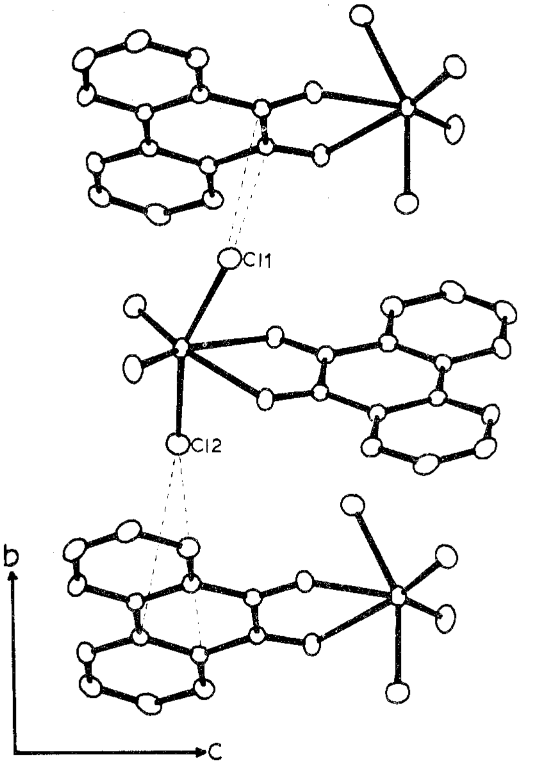
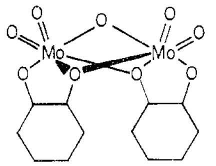


**Figure 2.** View of the shortest intermolecular contacts between molecules stacked along the $2_1$ screw axis.

$Mo_2O_5(O_2C_6H_4)_2^{2-}$ and $Mo_2O_5(O_2C_{14}H_8)_2$ differing by two electrons have related structures (I) with semiquinone ligands



I

paired in the diamagnetic $Mo_2O_5(O_2C_{14}H_8)_2$ molecule. Molybdenum–oxygen lengths of 2.15 (2) Å for $MoO_2(O_2C_6H_4)_2^{2-}$ and 2.16 Å for the shorter of the two bridging lengths in $Mo_2O_5(O_2C_6H_4)_2^{2-}$ reflect stronger donor activity for the catecholates relative to the corresponding Mo–O length of 2.24 Å for $Mo_2O_5(O_2C_{14}H_8)_2$ and the long values of 2.305 (3) Å in the present structure. These structural features are in accord with the anticipated trend that, in the absence of π-acceptor bonding, oxygen donor activity decreases as ketonic character of the C–O bond increases.

**Registry No.** $MoO_2Cl_2(O_2C_{14}H_8)$, 63915-44-6.

**Supplementary Material Available:** Tables containing positional and thermal parameters for hydrogen atoms of the structure and a listing of structure factor amplitudes (16 pages). Ordering information is given on any current masthead page.

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## Chemistry of Polylithiated Species. Reactions of Polylithiated Silanes and Germanes

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The interaction of a number of halogenated alkanes with a gaseous stream of lithium atoms has been shown to result in the formation of several poly- and perlithioalkanes including $CLi_4$, $C_2Li_6$, and $C_3Li_8$.[12] Under similar conditions, the re-