

The approximation used in eq 4 requires that the single ion excitations do not cause large lattice distortions. This condition is best satisfied by the highly localized $f-f$ transitions of the lanthanide ions. The approximation is less appropriate for systems such as MnF_2 in which the single ion transitions are more strongly coupled to the lattice. Because of $\text{Mn}^{2+}-\text{Mn}^{2+}$ interactions via the F^- lattice, discrepancies of up to 400 cm^{-1} were observed between the energies of SPE bands and summations of contributing single ion excitations.³⁶

The intensity of the SPE process is related to the square of the transition moment.

$$I_{\text{SPE}} \propto |\bar{\mu}_{\text{SPE}}|^2 = [\text{electronic part}]^2 \langle \chi_{A_s}^0 | \chi_{A_e}^e \rangle^2 \langle \chi_{B_s}^0 | \chi_{B_e}^e \rangle^2 \quad (5)$$

Equation 5 simply represents the SPE electronic intensity as given by Dexter⁴ modified by a product of the Franck-Condon factors of the contributing single ion transitions.

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Radical-Anion Coordination of 9,10-Phenanthrenequinone in $\text{Mo}_2\text{O}_5(\text{PQ})_2$

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Abstract: The photochemical addition of 9,10-phenanthrenequinone (PQ) to $\text{Mo}(\text{CO})_6$ in methylene chloride yields the diamagnetic complex $\text{Mo}_2\text{O}_5(\text{PQ})_2$. Crystals of the complex are triclinic, space group $P\bar{1}$ with $a = 13.024$ (3) Å, $b = 10.134$ (3) Å, $c = 10.389$ (3) Å, $\alpha = 84.95$ (4)°, $\beta = 94.93$ (4)°, and $\gamma = 98.64$ (4)°. Crystals form with one methylene chloride molecule per molecule of complex as solvent of crystallization. The structure was solved by conventional Patterson, Fourier, and least-squares procedures using X-ray data complete to $2\theta = 50^\circ$ (Mo $K\alpha$ radiation). Refinement of the structure converged with final discrepancy indices of $R = 0.045$ and $R_w = 0.054$ for 3319 observed, independent reflections. Bonded to each Mo are two terminal oxo ligands with one oxygen bridging the two metals. Quinone ligands bond terminally to each metal through one oxygen with the second bridging the metals unsymmetrically. The complex molecule possesses approximate C_2 symmetry. Short Mo-O distances (1.68 Å) for the terminal cis oxo ligands are consistent with a complex of Mo(VI). Thus the quinones bond as magnetically coupled radical anions.

Complexes of 1,2-benzoquinones with transition metals have been known for many years but only recently have attempts been made to fully characterize their coordination properties. While 1,4-benzoquinones bond to metals as diene ligands,¹ 1,2-benzoquinones appear to bond exclusively through their oxygens. As such they may chelate to met-

als as oxidized benzoquinones (Ia), fully reduced catecholates (Ic), or as semiquinones (Ib). The 9,10-phenanthrene-

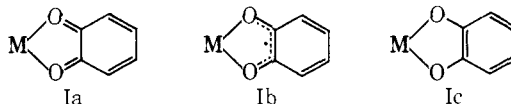


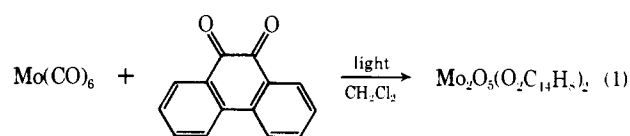
Table I. Final Structural Parameters for $\text{Mo}_2\text{O}_5(\text{O}_2\text{C}_{14}\text{H}_8)_2\cdot\text{CH}_2\text{Cl}_2$

Atom	x^a	y	z	β_{11}^b	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo (1)	0.2239 (1)	0.3460 (1)	0.2311 (1)	0.0058 (1)	0.0063 (1)	0.0069 (1)	0.0017 (1)	0.0009 (1)	-0.0019 (1)
Mo (2)	0.3458 (1)	0.1169 (1)	0.3637 (1)	0.0065 (1)	0.0084 (1)	0.0047 (1)	0.0023 (1)	0.0006 (1)	-0.0013 (1)
O (1)	0.2906 (4)	0.2751 (4)	0.3864 (4)	0.0066 (4)	0.0082 (6)	0.0065 (5)	0.0019 (4)	0.0016 (4)	-0.0027 (5)
O (2)	0.1074 (4)	0.3749 (5)	0.2740 (5)	0.0076 (5)	0.0099 (7)	0.0110 (7)	0.0024 (5)	0.0020 (5)	-0.0029 (6)
O (3)	0.2919 (4)	0.4988 (5)	0.2003 (5)	0.0092 (5)	0.0079 (7)	0.0088 (6)	0.0008 (5)	0.0001 (5)	-0.0029 (5)
O (4)	0.4696 (4)	0.1443 (5)	0.4261 (4)	0.0076 (5)	0.0142 (8)	0.0059 (6)	0.0026 (5)	-0.0012 (4)	-0.0029 (5)
O (5)	0.2846 (4)	0.0006 (5)	0.4687 (4)	0.0106 (6)	0.0094 (7)	0.0062 (6)	0.0032 (5)	0.0029 (4)	0.005 (5)
O (6)	0.1911 (3)	0.3137 (4)	0.0396 (4)	0.0049 (4)	0.0065 (6)	0.0071 (5)	0.0014 (4)	0.0002 (4)	-0.0009 (4)
O (7)	0.1800 (3)	0.1238 (4)	0.2226 (4)	0.0053 (4)	0.0067 (6)	0.0062 (5)	0.0018 (4)	0.0020 (3)	-0.0003 (4)
O (8)	0.3711 (3)	0.2379 (4)	0.1758 (4)	0.0048 (4)	0.0068 (6)	0.0055 (5)	0.0008 (4)	0.0000 (3)	-0.0024 (4)
O (9)	0.3526 (3)	-0.0169 (4)	0.2305 (4)	0.0056 (4)	0.0067 (6)	0.0047 (5)	0.0020 (4)	0.0007 (3)	0.0001 (4)
C (1)	0.3628 (5)	0.0253 (6)	0.1085 (6)	0.0039 (6)	0.0060 (10)	0.0047 (12)	0.0021 (9)	0.0001 (6)	-0.0005 (9)
C (2)	0.3727 (5)	0.1657 (6)	0.0777 (6)	0.0035 (8)	0.0080 (12)	0.0047 (10)	0.0015 (7)	0.0003 (8)	-0.0018 (10)
C (3)	0.3953 (5)	0.2221 (6)	-0.0499 (6)	0.0028 (7)	0.0084 (13)	0.0063 (12)	0.0013 (6)	-0.0003 (6)	-0.0020 (10)
C (4)	0.4003 (5)	0.1351 (6)	-0.1467 (6)	0.0032 (8)	0.0091 (12)	0.0052 (10)	0.0011 (7)	0.0002 (8)	-0.0016 (8)
C (5)	0.3766 (5)	-0.0103 (7)	-0.1180 (6)	0.0043 (7)	0.0089 (11)	0.0051 (9)	0.0020 (6)	-0.0005 (8)	-0.0030 (10)
C (6)	0.3624 (5)	-0.0653 (6)	0.0110 (6)	0.0035 (8)	0.0084 (13)	0.0060 (10)	0.0017 (6)	-0.0004 (9)	-0.0028 (10)
C (7)	0.4181 (5)	0.3619 (7)	-0.0765 (6)	0.0041 (8)	0.0079 (13)	0.0078 (12)	0.0017 (9)	-0.0002 (8)	-0.0000 (11)
C (8)	0.4480 (6)	0.4154 (7)	-0.1970 (7)	0.0066 (7)	0.0104 (12)	0.0059 (12)	0.0014 (9)	0.0014 (9)	0.0012 (10)
C (9)	0.4548 (5)	0.3270 (8)	-0.2924 (7)	0.0052 (8)	0.0120 (13)	0.0092 (12)	0.0018 (9)	0.0025 (8)	0.0010 (11)
C (10)	0.4316 (6)	0.1903 (7)	-0.2678 (6)	0.0058 (7)	0.0113 (12)	0.0051 (10)	0.0017 (7)	0.0005 (7)	0.0010 (9)
C (11)	0.3428 (5)	-0.2047 (7)	0.0422 (7)	0.0037 (8)	0.0075 (13)	0.0102 (11)	0.0014 (7)	0.0003 (7)	-0.0036 (11)
C (12)	0.3358 (5)	-0.2914 (7)	-0.0533 (6)	0.0055 (7)	0.0084 (12)	0.0061 (11)	0.0018 (9)	0.0001 (8)	-0.0002 (10)
C (13)	0.3489 (6)	-0.2383 (7)	-0.1813 (7)	0.0073 (8)	0.0096 (13)	0.0096 (13)	0.0028 (9)	-0.0006 (8)	-0.0043 (10)
C (14)	0.3695 (6)	-0.1014 (8)	-0.2147 (7)	0.0054 (8)	0.0119 (12)	0.0093 (11)	0.0029 (7)	-0.0006 (7)	-0.0055 (9)
C (15)	0.1676 (5)	0.1929 (6)	0.0024 (6)	0.0031 (6)	0.0054 (10)	0.0075 (9)	0.0012 (6)	-0.0001 (6)	-0.0004 (7)
C (16)	0.1613 (5)	0.0865 (6)	0.1030 (6)	0.0029 (7)	0.0068 (11)	0.0090 (10)	0.0010 (7)	0.0005 (6)	-0.0026 (8)
C (17)	0.1290 (5)	-0.0495 (6)	0.0730 (7)	0.0035 (6)	0.0060 (10)	0.0108 (9)	0.0017 (6)	-0.0001 (6)	-0.0026 (8)
C (18)	0.1097 (5)	-0.0775 (7)	-0.0585 (7)	0.0030 (7)	0.0077 (11)	0.0106 (10)	0.0008 (7)	-0.0004 (7)	-0.0031 (9)
C (19)	0.1205 (5)	0.0321 (7)	-0.1603 (7)	0.0036 (6)	0.0099 (10)	0.0086 (10)	0.0021 (6)	-0.0003 (6)	-0.0033 (8)
C (20)	0.1491 (5)	0.1665 (7)	-0.1299 (6)	0.0036 (5)	0.0082 (8)	0.0077 (8)	0.0013 (5)	-0.0003 (5)	-0.0023 (6)
C (21)	0.1184 (5)	-0.1537 (7)	0.1726 (8)	0.0040 (8)	0.0058 (13)	0.0144 (11)	0.0019 (8)	0.0012 (7)	0.0004 (10)
C (22)	0.0888 (6)	-0.2841 (7)	0.1433 (9)	0.0052 (8)	0.0087 (14)	0.0162 (12)	0.0009 (8)	0.0017 (8)	-0.0020 (10)
C (23)	0.0708 (6)	-0.3096 (8)	0.0107 (9)	0.0048 (8)	0.0094 (13)	0.0171 (12)	-0.0002 (8)	0.0005 (9)	-0.0028 (10)
C (24)	0.0786 (6)	-0.2106 (7)	-0.0871 (8)	0.0049 (8)	0.0075 (14)	0.0143 (13)	0.0001 (8)	0.0006 (8)	-0.0034 (11)
C (25)	0.1573 (5)	0.2736 (8)	-0.2265 (7)	0.0041 (7)	0.0119 (12)	0.0075 (11)	0.0013 (7)	-0.0002 (7)	0.0009 (9)
C (26)	0.1383 (6)	0.2488 (9)	-0.3570 (7)	0.0058 (7)	0.0154 (12)	0.0091 (11)	0.0019 (7)	0.0000 (7)	-0.0024 (9)
C (27)	0.1102 (6)	0.1137 (9)	-0.3884 (8)	0.0056 (9)	0.0191 (14)	0.0091 (13)	0.0027 (9)	0.0003 (8)	-0.0034 (11)
C (28)	0.1015 (6)	0.0084 (9)	-0.2933 (7)	0.0064 (8)	0.0162 (13)	0.0063 (12)	0.0028 (8)	-0.0004 (8)	-0.0043 (10)
Methylene Chloride Solvate									
Cl (1)	-0.3176 (2)	0.4414 (2)	0.4718 (2)	0.0172 (3)	0.0142 (4)	0.0143 (4)	0.0043 (3)	-0.0010 (3)	-0.0040 (3)
Cl (2)	-0.1270 (3)	0.3339 (4)	0.4734 (4)	0.0191 (5)	0.0310 (8)	0.0344 (8)	0.0107 (5)	-0.0038 (5)	-0.0100 (6)
C (29)	-0.2563 (10)	0.3152 (10)	0.5394 (10)	0.0183 (15)	0.0163 (17)	0.0151 (16)	0.0081 (13)	0.0009 (12)	0.0022 (13)

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

quinone (PQ) complex $\text{MoO}_2\text{Cl}_2(\text{PQ})$ has carbonyl bands above 1600 cm^{-1} in the infrared² and is representative of complexes containing unreduced quinone ligands. Much of the chemistry of benzoquinones is centered about their electrophilic activity. In complexes where the metal is sufficiently nucleophilic to reduce the quinone, carbonyl bands drop below 1500 cm^{-1} and structural features more closely related to form Ic are observed. The compound $\text{Pd}(\text{PPh}_3)_2(\text{O}_2\text{C}_6\text{Cl}_4)$ prepared by the oxidative addition of tetrachloro-1,2-benzoquinone to $\text{Pd}(\text{PPh}_3)_3$ is best described as a planar $\text{Pd}(\text{II})$ complex.⁴ Radical-anion semiquinone species are far more unusual. Oxidation of $\text{Pd}(\text{PPh}_3)_2(\text{O}_2\text{C}_6\text{Cl}_4)$ and related compounds leads to a paramagnetic series of cationic complexes with the unpaired electron localized in a quinone orbital.⁵ Radical-anion properties have been proposed for many ligands but these are perhaps the least ambiguous examples of semiquinone coordination.

We have found previously that addition of tetrachloro-1,2-benzoquinone to $\text{Mo}(\text{CO})_6$ yields the dimeric complex $\text{Mo}_2(\text{O}_2\text{C}_6\text{Cl}_4)_5$.⁶ The related reaction between 9,10-phenanthrenequinone (PQ) and $\text{Mo}(\text{CO})_6$ is quite different. A light source is required, and under otherwise similar conditions oxomolybdenum species are obtained.⁷ A reaction carried out in methylene chloride (eq 1) has been found to



yield $\text{Mo}_2\text{O}_5(\text{PQ})_2$ as the product. The complex is diamagnetic and from its formula could contain either $\text{Mo}(\text{V})$ with unreduced benzoquinone ligands (Ia) or semiquinone ligands (Ib) with $\text{Mo}(\text{VI})$. In either case the paramagnetic centers are required to be coupled antiferromagnetically. To help resolve questions concerning the structural and electronic properties of $\text{Mo}_2\text{O}_5(\text{PQ})_2$ and extend our investigation of 1,2-benzoquinone coordination we have determined the molecular structure of the complex crystallographically.

Experimental Section

Crystal Preparation and Data Collection. The synthesis of $\text{Mo}_2\text{O}_5(\text{PQ})_2$ has been described previously.⁷ Crystals of the dark green complex suitable for crystallographic work were grown directly from the reaction mixture. Elemental analyses indicated the presence of one methylene chloride molecule per molecule of complex. Precession and Weissenberg photographs on crystals of $\text{Mo}_2\text{O}_5(\text{PQ})_2\cdot\text{CH}_2\text{Cl}_2$ indicated triclinic symmetry. A crystal of dimensions $0.32 \times 0.34 \times 0.38\text{ mm}$ was aligned on a Picker four-circle diffractometer. Lattice constants determined at ambient room

Table II. Root-Mean-Square Amplitudes of Vibration of Atoms Refined Anisotropically

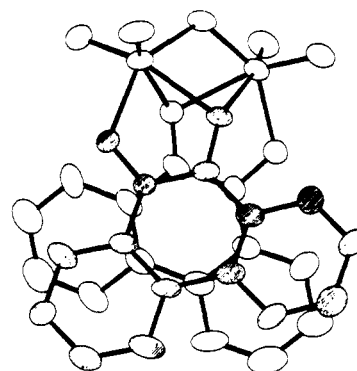
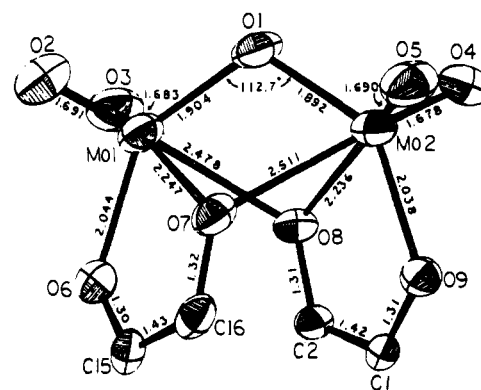
Atom	Min (Å)	Intermed (Å)	Max (Å)
Mo (1)	0.153 (1)	0.205 (1)	0.223 (2)
Mo(2)	0.151 (1)	0.197 (1)	0.240 (1)
O(1)	0.144 (10)	0.222 (11)	0.240 (11)
O(2)	0.184 (12)	0.252 (11)	0.265 (11)
O(3)	0.174 (12)	0.235 (12)	0.280 (12)
O(4)	0.164 (12)	0.244 (11)	0.281 (11)
O(5)	0.169 (11)	0.207 (11)	0.305 (12)
O(6)	0.174 (12)	0.194 (10)	0.207 (11)
O(7)	0.184 (11)	0.252 (11)	0.265 (11)
O(8)	0.144 (11)	0.200 (11)	0.206 (11)
O(9)	0.158 (12)	0.173 (11)	0.223 (11)
C(1)	0.142 (12)	0.159 (12)	0.200 (12)
C(2)	0.144 (12)	0.168 (12)	0.209 (12)
C(3)	0.147 (13)	0.172 (12)	0.217 (13)
C(4)	0.159 (12)	0.162 (13)	0.217 (12)
C(5)	0.139 (12)	0.177 (13)	0.233 (12)
C(6)	0.153 (13)	0.162 (12)	0.227 (13)
C(7)	0.169 (13)	0.207 (12)	0.212 (13)
C(8)	0.167 (12)	0.230 (13)	0.244 (13)
C(9)	0.178 (13)	0.225 (13)	0.264 (13)
C(10)	0.161 (13)	0.218 (12)	0.246 (12)
C(11)	0.154 (12)	0.183 (12)	0.251 (12)
C(12)	0.179 (12)	0.198 (12)	0.222 (13)
C(13)	0.168 (12)	0.227 (12)	0.276 (12)
C(14)	0.164 (12)	0.202 (13)	0.286 (12)
C(15)	0.147 (12)	0.173 (12)	0.204 (12)
C(16)	0.148 (12)	0.172 (12)	0.231 (13)
C(17)	0.141 (13)	0.180 (12)	0.249 (13)
C(18)	0.156 (13)	0.180 (12)	0.251 (13)
C(19)	0.159 (13)	0.186 (12)	0.251 (13)
C(20)	0.169 (12)	0.178 (13)	0.226 (12)
C(21)	0.148 (12)	0.194 (12)	0.282 (13)
C(22)	0.202 (13)	0.208 (13)	0.298 (13)
C(23)	0.189 (12)	0.227 (13)	0.307 (13)
C(24)	0.177 (12)	0.207 (12)	0.284 (12)
C(25)	0.175 (12)	0.204 (12)	0.255 (12)
C(26)	0.213 (13)	0.222 (12)	0.282 (13)
C(27)	0.208 (13)	0.216 (12)	0.315 (13)
C(28)	0.163 (13)	0.222 (12)	0.298 (13)
Cl(1)	0.238 (22)	0.287 (26)	0.386 (24)
Cl(2)	0.319 (36)	0.375 (41)	0.494 (43)
C(29)	0.233 (25)	0.304 (28)	0.404 (26)

temperature from least-squares refinement of the angular settings of 23 strong, independent reflections located using Mo $K\alpha$ radiation are $a = 13.024$ (3) Å, $b = 10.134$ (3) Å, $c = 10.389$ (3) Å, $\alpha = 84.95$ (4)°, $\beta = 94.93$ (4)°, and $\gamma = 98.64$ (4)°. An experimental density of 1.896 (5) g/cm³ agrees with a calculated value of 1.902 g/cm³ for two formula weights of composition Mo₂Cl₂O₉C₂₉H₁₈ per unit cell. Therefore, space group $P\bar{1}$ was chosen for the refinement. The mosaic spread of the crystal determined using the narrow-source open-counter ω -scan technique was acceptable at 0.07°. An independent set of intensity data was collected by the θ - 2θ scan technique using the Zr-filtered Mo $K\alpha$ peak with allowances made for the $K\alpha_1$ - $K\alpha_2$ separation at higher 2θ values. The data set was collected within the angular range $4.5 \leq 2\theta \leq 50^\circ$. Attenuators were inserted automatically if the count rate of the diffracted beam exceeded 9000 counts/sec during the scan. The intensities of five standard reflections in different regions of reciprocal space were monitored during data collection after every 100 reflections measured. None of these standards deviated from its mean value by more than 3% during the time required to collect data. Data were processed in the usual way with values of I and $\sigma(I)$ corrected for Lorentz and polarization effects. No correction for absorption was made since the linear absorption coefficient is small ($\mu = 11.7$ cm⁻¹) and rotation of the crystal at $\chi = 90.00^\circ$ showed no significant variation in intensity of a reflection coincident with the ϕ axis of the instrument. The intensities of 5036 reflections were measured of which 3319 were observed to be greater than 2σ and have been included in the refinement.

Solution and Refinement of the Structure. The position of the Mo atom was determined from a three-dimensional Patterson map. Phases derived from the Mo were used to locate all other

Table III. Refined Positional and Thermal Parameters of Hydrogen Atoms

Atom	x	y	z	$B, \text{Å}^2$
HC(7)	0.409 (6)	0.418 (7)	-0.001 (7)	6. (2)
HC(8)	0.478 (6)	0.531 (7)	-0.224 (7)	7. (2)
HC(9)	0.494 (6)	0.385 (6)	-0.371 (7)	9. (2)
HC(10)	0.442 (6)	0.129 (7)	-0.340 (7)	5. (2)
HC(11)	0.346 (6)	-0.240 (7)	0.151 (7)	8. (2)
HC(12)	0.318 (6)	-0.410 (7)	-0.032 (7)	5. (2)
HC(13)	0.339 (6)	-0.312 (7)	-0.253 (7)	9. (2)
HC(14)	0.369 (6)	-0.070 (7)	-0.324 (7)	9. (3)
HC(21)	0.138 (6)	-0.134 (7)	0.268 (7)	7. (2)
HC(22)	0.081 (6)	-0.357 (7)	0.237 (7)	6. (2)
HC(23)	0.044 (6)	-0.412 (7)	-0.012 (7)	8. (2)
HC(24)	0.070 (6)	-0.235 (7)	-0.190 (7)	7. (2)
HC(25)	0.167 (6)	0.379 (7)	-0.176 (7)	8. (2)
HC(26)	0.137 (6)	0.351 (7)	-0.440 (7)	7. (2)
HC(27)	0.088 (6)	0.090 (7)	-0.509 (7)	8. (2)
HC(28)	0.088 (6)	-0.095 (7)	-0.320 (7)	8. (2)


Figure 1. View from the side of the Mo₂O₅(PQ)₂ molecule.

Figure 2. View of the inner-coordination geometry of the Mo₂O₅(PQ)₂ molecule.

nonhydrogen atoms. Isotropic refinement of all atoms gave discrepancy indices

$$R = \sum ||F_d| - |F_o|| / \sum |F_o|$$

and

$$R_w = (\sum_w (|F_d| - |F_o|)^2 / \sum_w F_o^2)^{1/2}$$

of 0.121 and 0.128, respectively. Further refinement with anisotropic thermal parameters for all nonhydrogen atoms gave discrepancy indices of $R = 0.050$ and $R_w = 0.061$. The positions of all hydrogen atoms of the Mo₂O₅(PQ)₂ molecule were determined from a Fourier map and included in the refinement giving final discrepancy indices of $R = 0.045$ and $R_w = 0.054$. Methylene chloride hydrogens could not be located with certainty. During all cycles of refinement the function minimized was $\sum_w (|F_d| - |F_o|)^2$ and the weights, w , were taken as $4F_o^2 / \sigma^2(F_o^2)$. Standard deviations $\sigma(F^2)$ were estimated by procedures described previously.⁹ In all calculations the atomic scattering factors for the nonhydrogen atoms were those of Cromer and Waber¹⁰ with hydrogen scatter-

Table IV. Principle Intramolecular Bonding Parameters for $\text{Mo}_2\text{O}_5(\text{O}_2\text{C}_14\text{H}_9)_2 \cdot \text{CH}_2\text{Cl}_2$

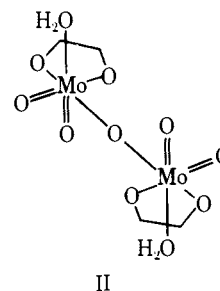
Mo_2O_5 Fragment			
Atoms	Distances (Å)	Atoms	Distances (Å)
Mo(1)–O(1)	1.904 (5)	Mo(1)–O(6)	2.044 (4)
Mo(2)–O(1)	1.892 (4)	Mo(1)–O(7)	2.247 (5)
Mo(1)–O(2)	1.691 (5)	Mo(1)–O(8)	2.478 (4)
Mo(1)–O(3)	1.683 (5)	Mo(2)–O(9)	2.038 (4)
Mo(2)–O(4)	1.678 (5)	Mo(2)–O(8)	2.236 (4)
Mo(2)–O(5)	1.690 (5)	Mo(2)–O(7)	2.511 (5)
Atoms	Angles (deg)	Atom	Angles (deg)
O(1)–Mo(1)–O(2)	104.8 (2)	O(1)–Mo(2)–O(4)	104.9 (2)
O(1)–Mo(1)–O(3)	104.9 (2)	O(1)–Mo(2)–O(5)	104.2 (2)
O(1)–Mo(1)–O(7)	76.7 (2)	O(1)–Mo(2)–O(8)	77.1 (2)
O(1)–Mo(1)–O(8)	71.0 (2)	O(1)–Mo(2)–O(7)	70.4 (2)
O(2)–Mo(1)–O(3)	105.1 (2)	O(4)–Mo(2)–O(5)	105.0 (2)
O(2)–Mo(1)–O(6)	101.7 (2)	O(4)–Mo(2)–O(9)	102.4 (2)
O(2)–Mo(1)–O(7)	96.0 (2)	O(4)–Mo(2)–O(8)	96.6 (2)
O(3)–Mo(1)–O(6)	93.8 (2)	O(5)–Mo(2)–O(9)	93.6 (2)
O(3)–Mo(1)–O(8)	90.9 (2)	O(5)–Mo(2)–O(7)	91.0 (2)
O(6)–Mo(1)–O(7)	73.8 (2)	O(8)–Mo(2)–O(9)	73.9 (2)
O(6)–Mo(1)–O(8)	76.0 (2)	O(7)–Mo(2)–O(9)	76.1 (2)
O(7)–Mo(1)–O(8)	68.0 (1)	O(7)–Mo(2)–O(8)	67.5 (1)
O(1)–Mo(1)–O(6)	142.0 (2)	O(1)–Mo(2)–O(9)	142.1 (2)
O(2)–Mo(1)–O(8)	164.0 (2)	O(4)–Mo(2)–O(7)	164.0 (2)
O(3)–Mo(1)–O(7)	157.3 (2)	O(5)–Mo(2)–O(8)	157.1 (2)
		Mo(1)–O(1)–Mo(2)	112.7 (2)
		Mo(1)–O(7)–Mo(2)	83.0 (1)
		Mo(1)–O(8)–Mo(2)	84.0 (1)
Phenanthrenequinone I			
Atoms	Distances (Å)	Atoms	Distances (Å)
O(8)–C(2)	1.310 (7)	C(7)–C(8)	1.384 (9)
O(9)–C(1)	1.314 (7)	C(8)–C(9)	1.411 (10)
C(1)–C(2)	1.420 (8)	C(9)–C(10)	1.378 (10)
C(2)–C(3)	1.434 (8)	C(4)–C(10)	1.401 (9)
C(3)–C(4)	1.407 (8)	C(6)–C(11)	1.411 (9)
C(4)–C(5)	1.469 (9)	C(11)–C(12)	1.371 (9)
C(5)–C(6)	1.421 (9)	C(12)–C(13)	1.406 (9)
C(6)–C(1)	1.427 (8)	C(13)–C(14)	1.392 (10)
C(3)–C(7)	1.411 (9)	C(5)–C(14)	1.413 (9)
Atoms	Angles (deg)	Atoms	Angles (deg)
Mo(2)–O(8)–C(2)	113.2 (4)	O(9)–C(1)–C(6)	121.8 (6)
Mo(1)–O(8)–C(2)	127.0 (4)	O(8)–C(2)–C(1)	115.4 (5)
Mo(2)–O(9)–C(1)	119.9 (4)	O(8)–C(2)–C(3)	123.4 (6)
O(9)–C(1)–C(2)	116.8 (5)		
Phenanthrenequinone II			
Atoms	Distances (Å)	Atoms	Distances (Å)
O(6)–C(15)	1.301 (7)	C(21)–C(22)	1.374 (9)
O(7)–C(16)	1.324 (7)	C(22)–C(23)	1.419 (11)
C(15)–C(16)	1.432 (9)	C(23)–C(24)	1.363 (11)
C(16)–C(17)	1.433 (8)	C(18)–C(24)	1.399 (9)
C(17)–C(18)	1.415 (9)	C(20)–C(25)	1.410 (9)
C(18)–C(19)	1.465 (9)	C(25)–C(26)	1.397 (10)
C(19)–C(20)	1.414 (9)	C(26)–C(27)	1.422 (11)
C(20)–C(15)	1.419 (9)	C(27)–C(28)	1.388 (11)
C(17)–C(21)	1.411 (9)	C(19)–C(28)	1.418 (9)
Atoms	Angles (deg)	Atoms	Angles (deg)
Mo(1)–O(7)–C(16)	113.2 (4)	O(6)–C(16)–C(20)	122.4 (6)
Mo(2)–O(7)–C(16)	126.4 (4)	O(7)–C(16)–C(15)	115.7 (5)
Mo(1)–O(6)–C(15)	120.9 (4)	O(7)–C(16)–C(17)	123.4 (6)
O(6)–C(15)–C(16)	116.3 (6)		

ing factors taken from the report of Stewart et al.¹¹ The effects of anomalous dispersion were included in the calculated structure factors with values of $\Delta f'$ and $\Delta f''$ for the Mo and Cl atoms taken from the report of Cromer and Liberman.¹² At the completion of the refinement the standard deviation of an observation of unit weight was 1.64. The final positional and thermal parameters of all nonhydrogen atoms are presented in Table I. Root-mean-square vibrational amplitudes of atoms refined anisotropically appear in Table II. Positional and thermal parameters of refined hydrogen atoms appear in Table III. A table of the final F_o and F_c values is available.¹³

Discussion

Structural features of the $\text{Mo}_2\text{O}_5(\text{PQ})_2$ molecule include

an oxomolybdenum geometry uniquely characteristic of Mo(VI) with a highly unusual coordination configuration for the 9,10-phenanthrenequinone ligands. A view of the $\text{Mo}_2\text{O}_5(\text{PQ})_2$ molecule is presented in Figure 1. The Mo_2O_9 inner-coordination geometry shown in Figure 2 resembles a confacial bioctahedron, two distorted MoO_6 octahedra sharing a common face. Bonded strongly to each metal are cis oxo ligands with short Mo–O distances (1.678 (5)–1.691 (5) Å) and large O–Mo–O angles of 104.8 (2) and 105.1 (2)°. A complete set of interatomic distances and bond angles is given in Table IV. Cis to the terminal oxo ligands is an additional oxygen bridging the metals with a Mo(1)–O(1)–Mo(2) angle of 112.7 (2)° and Mo–O distances of 1.892 (4) and 1.904 (5) Å. The Mo(1)–Mo(2) separation of 3.160 (2) Å is nonbonding and considerably longer than metal–metal bonded values found in Mo(V) complexes which are normally between 2.5 and 2.6 Å.¹⁴ The 9,10-phenanthrenequinone ligands each bond terminally through one oxygen with the second bridging the metals unsymmetrically. Terminal quinone oxygens are trans to the bridging oxo ligand and have shorter Mo–O distances (2.044 (4), 2.038 (4) Å) than the bridging quinone oxygens. These are trans to terminal oxo ligands, have longer Mo–O distances to the metal associated with their respective terminal quinone oxygen (2.2364 (4), 2.247 (5) Å), and bridge weakly to the adjacent metal with relatively long Mo–O distances of 2.478 (4) and 2.511 (5) Å. The entire $\text{Mo}_2\text{O}_5(\text{PQ})_2$ molecule has approximate C_2 symmetry. Features of the coordinated oxo ligands in $\text{Mo}_2\text{O}_5(\text{PQ})_2$ are similar to various other oxomolybdenum(VI) systems.¹⁴ The cis dioxo group is well known for monomeric Mo(VI) complexes but oxo bridged $\text{Mo}_2\text{O}_2^{2+}$ fragments are more unusual. The polymeric anion $[\text{Mo}_3(\text{C}_2\text{O}_4)]_n^{2-}$ contains metal centers linked unsymmetrically by bent –Mo–O–Mo–O– bridges.¹⁵ A cyclopentadienyl complex, $\text{Mo}_2\text{O}_5(\text{C}_5\text{H}_5)_2$, is also reported to contain a single oxo bridge.¹⁶ Perhaps the most closely related system examined structurally is the centrosymmetric $[\text{Mo}_2\text{O}_5(\text{C}_2\text{O}_4)_2(\text{H}_2\text{O})_2]^{2-}$ anion (II).¹⁷



This complex contains strongly bound terminal oxo ligands with Mo–O distances of 1.68 (2) and 1.70 (2) Å, a linear oxo bridge (Mo–O = 1.876 (2) Å), chelating oxalate ligands, and water molecules occupying the sixth coordination positions about each metal. One might speculate that upon reduction to a dianionic complex in the presence of additional ligands, $\text{Mo}_2\text{O}_5(\text{PQ})_2$ might adopt a similar geometry.

Coordination of the Semiquinone Ligands. As a complex of Mo(VI) the quinone ligands in $\text{Mo}_2\text{O}_5(\text{PQ})_2$ coordinate formally as radical-anions. This formulation is consistent with the C–O bond lengths which average to 1.313 (8) Å, intermediate between values of 1.22 Å normally found for benzoquinones and 1.38 Å found in hydroquinones.¹⁸ Carbonyl bond lengths of 1.313 (8) Å were also found for the bent phenanthrenequinone ligand in trigonal prismatic $\text{Mo}(\text{PQ})_3$ while quinone ligands in this structure which chelate normally to the metal have somewhat longer C–O lengths of 1.345 (8) Å.⁷ Chelated tetrachloro-1,2-benzoqui-

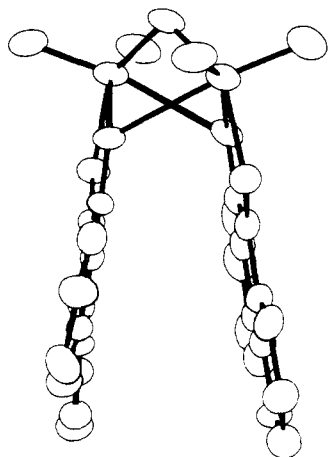


Figure 3. View showing the paired 9,10-phenanthrenequinone ligands. Ligand planes form a dihedral angle of 19.3 (3)°.

none ligands in $\text{Mo}_2(\text{O}_2\text{C}_6\text{Cl}_4)_6$ ¹⁹ and $\text{Pd}(\text{PPh}_3)_2(\text{O}_2\text{C}_6\text{Cl}_4)_4$ have C–O lengths of 1.33 (1) and 1.34 (1) Å, respectively. The reduced bridging quinone ligands in $\text{Mo}_2(\text{O}_2\text{C}_6\text{Cl}_4)_6$ have longer carbonyl distances of 1.37 (1) Å. This range of carbonyl distances reflects metal–ligand charge distribution and relative contributions of bonding structures Ia to Ic.

The observed diamagnetism of the complex requires that the semiquinone ligands be magnetically coupled. Such coupling might be facilitated by the Mo_2O_2 ring formed by the bridging ligand oxygens. However, the parallel orientation of the quinone ligands in the $\text{Mo}_2\text{O}_5(\text{PQ})_2$ molecule (Figure 3) suggests that direct interaction between radical-anions is likely. A dihedral angle of 19.3 (3)° exists between ligand planes with extremely short interatomic and interplanar separations between ligand carbonyl groups (Table V). Oxygens O(7) and O(8) are only 2.25 Å from the plane of the opposite ligand while shortest interatomic separations range from 2.65 to 2.83 Å between oxygens and carbonyl carbons of adjacent ligands. Diamagnetic coupling between quinone and quinoid radical anions is highly unusual. Coupling between hydroquinone radicals has been reported to occur in an acetonitrile clathrate matrix but contributes to a triplet ESR signal.²⁰ Strong interactions between partially reduced tetracyanoquinodimethane (TCNQ) molecules result in short interplanar separations (3.11–3.17 Å) in columnar, conductive TTF–TCNQ crystals.²¹ However, these systems are remotely related to the $\text{Mo}_2\text{O}_5(\text{PQ})_2$ molecule where the Mo_2O_5 fragment behaves as a template at which pairing of the semiquinone ligands occurs.

Molybdenum–Flavine Systems. While study of the activity of Mo in biological systems has been the subject of many recent investigations, little is known about the structural properties and detailed function of the metal center. Evidence obtained primarily from ESR experiments indicates that an oxomolybdenum species is involved in the electron transfer sequence of various enzymes.²² In milk xanthine oxidase electron transfer occurs between a poorly defined Mo species and the quinoid isoalloxazine ring of an oxidized flavine.²³ During this process ESR signals characteristic of both flavine radicals and paramagnetic molybdenum centers are observed. This sequence is reversed for nitrate reductase where Mo(VI) is reduced by FADH_2 .²⁴ Xanthine oxidase and aldehyde reductase each contain two molybdenums and two flavines (FAD) which with evidence obtained from Mo–flavine model studies has led to the proposal that the biologically active Mo center may be dimeric.²⁵ The geometry of $\text{Mo}_2\text{O}_5(\text{PQ})_2$ is clearly pertinent to this proposal.

Table V. Dihedral Angles and Least-Squares Planes

Plane 1	Dihedral Angles Plane 2	Angle (deg)
C(2), C(4), C(6)	C(16), C(18), C(20)	19.3 (3)
O(2), O(3), O(6)	O(1), O(7), O(8)	172.4 (2)
O(4), O(5), O(9)	O(1), O(7), O(8)	172.2 (2)
O(2), O(3), O(6)	O(4), O(5), O(9)	169.5 (2)
Mo(1), O(7), O(8)	Mo(2), O(8), O(7)	106.3 (1)

Least-Squares Planes ^{a, b}			
Atom	Distance (Å)	Atom	Distance (Å)
Phenanthrenequinone I $12.72x - 1.18y + 1.34z = 4.69$			
Phenanthrenequinone I		Phenanthrenequinone II	
Mo(2)	0.057	O(6)	2.581
O(8)	−0.018	O(7)	2.250
O(9)	0.121	C(15)	2.788
C(1)	0.037 (6)	C(16)	2.606
C(2)	−0.045 (6)	C(17)	2.898
C(3)	0.003 (6)	C(18)	3.287
C(4)	0.038 (6)	C(19)	3.417
C(5)	−0.052 (6)	C(20)	3.171
C(6)	0.007 (6)	C(21)	2.774
C(7)	0.092	C(22)	3.036
C(8)	0.246	C(23)	3.415
C(9)	0.308	C(24)	3.564
C(10)	0.208	C(25)	3.324
C(11)	−0.036	C(26)	3.713
C(12)	−0.151	C(27)	3.954
C(13)	−0.221	C(28)	3.810
C(14)	−0.166		
Phenanthrenequinone II $12.95x - 2.50y - 1.44z = 1.70$			
Phenanthrenequinone II		Phenanthrenequinone I	
Mo(1)	0.000	O(8)	2.256
O(6)	−0.066	O(9)	2.572
O(7)	−0.001	C(1)	2.776
C(15)	−0.016 (6)	C(2)	2.598
C(16)	0.023 (6)	C(3)	2.935
C(17)	−0.014 (6)	C(4)	3.347
C(18)	−0.003 (6)	C(5)	3.371
C(19)	0.010 (6)	C(6)	3.318
C(20)	0.001 (6)	C(7)	2.920
C(21)	−0.035	C(8)	3.347
C(22)	−0.051	C(9)	3.793
C(23)	−0.028	C(10)	3.799
C(24)	−0.033	C(11)	3.186
C(25)	−0.021	C(12)	3.450
C(26)	−0.016	C(13)	3.672
C(27)	0.002	C(14)	3.645
C(28)	0.015		

^a Least-squares planes calculated according to W. C. Hamilton, *Acta Crystallogr.*, 14, 185 (1961). Equations given in triclinic coordinates. ^b Atoms listed without errors not included in the calculation of the plane.

While no discrete Mo–flavine complexes have yet been isolated, solution studies have indicated the formation of binuclear Mo(V)–flavine semiquinone complexes including examples reported to be ESR inactive.^{26,27} The structure and diamagnetism of $\text{Mo}_2\text{O}_5(\text{PQ})_2$ show that in simple Mo–flavine complexes diamagnetism does not preclude Mo(VI)–semiquinone coordination.

Crystal Structure of $\text{Mo}_2\text{O}_5(\text{PQ})_2 \cdot \text{CH}_2\text{Cl}_2$. Reduced and oxidized forms of benzoquinones and quinoid molecules are well known for their ability to form conductive donor–acceptor complexes.²⁸ Previous structural work on transition metal complexes containing 1,2-benzoquinone ligands indicates a similar trend. The complex $\text{Mo}(\text{PQ})_3$ prepared from $\text{Mo}_2\text{O}_5(\text{PQ})_2$ is a striking example of this behavior.⁷ Phenanthrenequinone ligands in this molecule differ significantly in oxidation state and coordination geometry. Adjacent molecules in the unit cell are related by strong interligand charge-transfer interactions. Separations between li-

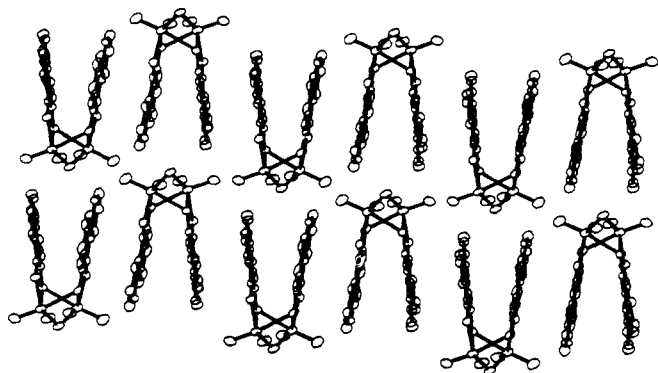


Figure 4. Crystal structure of $\text{Mo}_2\text{O}_5(\text{PQ})_2$ with methylene chloride molecules omitted for clarity. View contains unit cells related by three translations along a in the horizontal direction and one translation along c in the vertical direction.

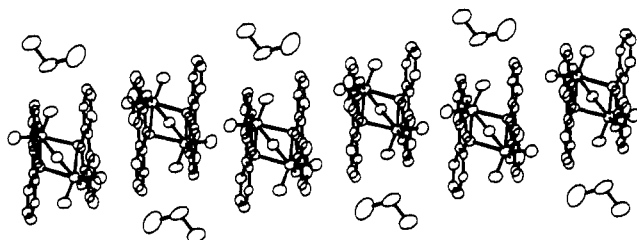


Figure 5. Crystal structure of $\text{Mo}_2\text{O}_5(\text{PQ})_2 \cdot \text{CH}_2\text{Cl}_2$. Each CH_2Cl_2 interacts weakly with O(5). The view is approximately down the c axis.

gand planes are in the 3.2–3.3 Å range. The benzene solvate of $\text{Mo}_2(\text{O}_2\text{C}_6\text{Cl}_4)_6$ contains solvent molecules sandwiched interstitially between quinone ligands of adjacent molecules.¹⁹ Planar separations of 3.4–3.5 Å are observed in this structure with each complex linked to four neighboring molecules by benzene bridges. In solid, $\text{Mo}_2\text{O}_5(\text{PQ})_2 \cdot \text{CH}_2\text{Cl}_2$ exists as chains of molecules joined by donor–acceptor interactions between ligands (Figure 4 and 5). Methylene chloride molecules interact weakly with O(5) through one hydrogen. Within the unit cell centrosymmetrically related molecules pair by overlap of the central quinone benzene ring of one ligand, carbons C(15) to C(20), with one outer aromatic ring of the adjacent phenanthrenequinone ligand, carbons C(17)', C(18)', C(21)', C(22)', C(23)', C(24)'. Adjacent unit cells are related along the crystallographic a axis by similar donor–acceptor interactions between central quinone carbons and neighboring aromatic rings. Interatomic contacts are within the range of 3.4–3.5 Å while interplanar separations between adjacent molecules are approximately 3.37 Å (Table VI). These values are slightly longer than separations between ligands in $\text{Mo}(\text{PQ})_3$ and longer than those found in strong benzoquinone–hydroquinone donor–acceptor complexes which are within the range of 3.15–3.20 Å.²⁹ Nevertheless, the intermolecular separations in $\text{Mo}_2\text{O}_5(\text{PQ})_2$ are on the order of stacked organic molecules found to exhibit conductive behavior.²⁸

Supplementary Material Available. A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 24X reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Business Office, Books and Journals Division, American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036. Remit check or money order for \$4.50 for photocopy or \$2.50 for microfiche, referring to code number JACS-75-6450.

Table VI. Inter- and Intramolecular Contacts between Adjacent Phenanthrenequinone Ligands

Atoms	Distance (Å)	Atoms	Distance (Å)
Intramolecular			
O(7)...O(8)	2.648 (6)	C(3)...C(15)	3.028 (9)
C(2)...C(15)	2.766 (9)	O(8)...C(15)	3.073 (7)
C(1)...C(16)	2.781 (9)	O(7)...C(1)	3.079 (7)
O(6)...O(8)	2.805 (6)	C(6)...C(17)	3.193 (9)
O(7)...O(9)	2.828 (6)	C(3)...C(20)	3.221 (9)
O(6)...C(2)	2.966 (7)	C(6)...C(18)	3.295 (9)
O(7)...C(2)	2.994 (8)	C(4)...C(20)	3.355 (9)
O(8)...C(16)	2.998 (8)	C(5)...C(19)	3.414 (9)
O(9)...C(16)	3.005 (8)	C(5)...C(18)	3.537 (9)
C(1)...C(17)	3.026 (9)	C(4)...C(19)	3.632 (9)
		C(2)...C(16)	3.778 (9)
Intermolecular ^a			
Center of Inversion ^b			
C(15)...C(23)'	3.47 (1)	C(17)...C(19)'	3.48 (1)
C(15)...C(24)'	3.43 (1)	C(19)...C(21)'	3.50 (1)
C(16)...C(18)'	3.51 (1)	C(20)...C(21)'	3.46 (1)
C(16)...C(24)'	3.53 (1)	C(20)...C(22)'	3.47 (1)
C(17)...C(18)'	3.53 (1)	C(22)...C(25)'	3.41 (1)
Translation Along a^c			
C(1)...C(5)''	3.41 (1)	C(3)...C(12)''	3.56 (1)
C(1)...C(14)''	3.56 (1)	C(4)...C(6)''	3.43 (1)
C(2)...C(13)''	3.67 (1)	C(4)...C(11)''	3.41 (1)
C(2)...C(14)''	3.67 (1)	C(5)...C(6)''	3.48 (1)
C(3)...C(11)''	3.45 (1)	C(6)...C(6)''	3.67 (1)

^a Closest contacts between atoms of one ligand with the least-squares plane of the adjacent ligand related by inversion or translation–inversion are 3.37 Å in each case. ^b Prime notation refers to atoms related to those in Table I by $\bar{x}, \bar{y}, \bar{z}$. ^c Double prime refers to atoms related to those in Table I by $1 - x, \bar{y}, \bar{z}$.

References and Notes

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