

dral geometry in the solid state with the alkyne orientated parallel to the OC-Ta-CO axis.

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$[\text{Et}_4\text{N}][\text{Ta}(\text{CO})_6]$, 67292-38-0; $\text{Ta}(\text{CO})_2(\text{PMe}_3)[\text{P}(\text{OMe})_3](\text{I})$ -(PhCCPh), 111690-51-8; $\text{Nb}(\text{CO})_2(\text{HCCH})(\text{Cl})(\text{PH}_3)_2$, 111690-52-9; $\text{PhC}\equiv\text{CPh}$, 501-65-5; $\text{PhC}\equiv\text{CH}$, 536-74-3; $\text{Ph}_2\text{PC}\equiv\text{CPh}_2$, 5112-95-8.

Supplementary Material Available: Tables of isotropic and anisotropic factors for 3, bond distances and angles for 3, mass spectral data for 3-5, calculated hydrogen positions, and planes calculations (10 pages); listing of observed and calculated structure factors for 3 (15 pages). Ordering information is given on any current masthead page.

Studies of Molybdenum Compounds. 6. Diaryl(2,2'-bipyridyl)dioxomolybdenum(VI) and Related Compounds

G. N. Schrauzer,* Xumu Zhang, Nan Hui Liu,[†] and E. O. Schlemper[‡]

Department of Chemistry, University of California, San Diego, La Jolla, California 92093

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The first σ -diaryl derivatives of the type $\text{R}_2\text{Mo}(\text{O})_2(\text{bpy})$ ($\text{R} = \text{C}_6\text{H}_5$, 4- $\text{CH}_3\text{OC}_6\text{H}_4$, 4- $\text{CH}_3\text{C}_6\text{H}_4$, 4- ClC_6H_4 , or 2- $\text{CH}_3\text{C}_6\text{H}_4$; bpy = 2,2'-bipyridyl) are described. The Mo-C bonds in these compounds are labilized through a combination of steric and electronic effects arising from interactions of the Mo=O moieties with the aromatic π -electron systems and overcrowding due to the presence of the coordinated bpy. In the complex with $\text{R} = 4\text{-CH}_3\text{OC}_6\text{H}_4$, the observed mean Mo-C_{sp²} bond lengths of 2.202 (3) Å are similar to the mean Mo-C_{sp³} bond lengths in the corresponding complex with $\text{R} = \text{C}_2\text{H}_5$. The aryl substituents adopt a conformation maximizing π -orbital overlap with one Mo=O moiety. This is possible only with distortions of the Mo-C-C bond angles. The π -orbital interactions affect the energies of first low-energy absorptions in the UV-vis spectra and of the IR $\nu_{\text{Mo=O}}$ absorptions. Crystal data for (4- $\text{CH}_3\text{OC}_6\text{H}_4$)₂Mo(O)₂(bpy): space group $P\bar{1}$, $a = 8.492$ (2) Å, $b = 11.340$ (2) Å, $c = 13.076$ (2) Å, $\alpha = 65.8$ (1)°, $\beta = 78.9$ (1)°, $\gamma = 71.4$ (1)°, $Z = 2$. The structure was solved and refined (2397 independent reflections above 2σ) to the final values of residual $R(F_o) = 0.025$ and $R_w(F_o) = 0.032$.

Following the discovery of dialkyl derivatives of oxomolybdate(VI) of the type $\text{R}_2\text{Mo}(\text{O})_2(\text{bpy})$,¹ we extended our study to the corresponding diaryl derivatives not only because few arylmolybdenum(VI) compounds are known but also for theoretical reasons: As M=O bonds in some complexes exhibit ketone-like behavior,² compounds of the type (aryl)₂Mo(O)₂(bpy) would offer an opportunity to obtain evidence for or against interactions of aromatic π -electron systems with Mo(VI)=O moieties. Reacting Mo(O)₂Cl₂·2THF with organomagnesium halides, Heyn and Hoffmann³ previously reported that organomolybdenum compounds are formed but implied that isolation of pure products is difficult. With use of this method, dimesityldioxomolybdenum(VI) (1) could at least be obtained, but no (σ -aryl)molybdenum(VI) species with aryl residues other than mesityl have been described in the literature. Reacting Br₂Mo(O)₂(bpy) (2) with arylmagnesium halides, we were able to obtain complexes of the type (aryl)₂Mo(O)₂(bpy) with aryl = C₆H₅ (3), 4-CH₃C₆H₄ (4), 2-CH₃C₆H₄ (5), 4-ClC₆H₄ (6), or 4-CH₃OC₆H₄ (7). Since spectroscopic measurements suggested π -orbital interactions between the aryl and the Mo=O moieties, the structure of one representative compound—7—was determined to establish the orientation of the $p\text{-CH}_3\text{OC}_6\text{H}_4$

rings with respect to the Mo(O)₂ group and other relevant structural features.

Experimental Section

Reagents and Chemicals. Solutions of the aryl magnesium halides in THF either were commercially available or were prepared by the usual methods and were employed without further purification. Tetrahydrofuran was dried over sodium/benzophenone and distilled immediately prior to use. Mo(O)₂Br₂(bpy) was prepared according to Hull and Stiddard⁴ and dried for 8 h at 100 °C prior to use.

General Synthesis of Complexes (Aryl)₂Mo(O)₂(bpy) (3-7). To stirred suspensions of 4.4 g (10 mmol) of 2 in 50 cm³ of dry tetrahydrofuran (THF) was added 22 mmol of the respective aryl magnesium bromides in 60 cm³ of THF dropwise under argon at 0 °C over a period of 1 h. The reaction mixtures were allowed to warm to room temperature for 2 h, and the THF solvent was evaporated in vacuo at room temperature. The residues were treated with 150 cm³ of cold water containing 12 drops of concentrated NH₄OH solution and transferred into separatory funnels of 1-L capacity. After the addition of 200 cm³ of CH₂Cl₂, the mixtures were shaken repeatedly for 2 h. During this time the organic phase turned orange. The aqueous phases were discarded,

*Department of Chemistry, Lanzhou University, Lanzhou, Gansu, China.

[†]Department of Chemistry, University of Missouri, Columbia, Missouri 65211.

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Table I. Analytical Data for Complexes 3-7

no.	mp, (dec)	compositn	anal. calcd				found			
			C	H	Mo	mol wt	C	H	Mo	mol wt
3	175	C ₂₂ H ₁₈ MoN ₂ O ₂	60.28	4.14	21.89	438.34	59.50	4.22	21.63	440
4	182	C ₂₄ H ₂₂ MoN ₂ O ₂	61.81	4.75	20.57	466.39	61.65	4.65	20.05	
5	180	C ₂₄ H ₂₂ MoN ₂ O ₂	61.81	4.75	20.57	466.39	62.05	4.85	20.10	
6	154	C ₂₂ H ₂₀ MoN ₂ O ₂ Cl ₂	52.10	3.18	18.91	507.23	51.97	3.21		
7	182	C ₂₄ H ₂₂ MoN ₂ O ₄	57.84	4.45	19.25	498.39	57.52	4.41	19.47	498

Table II. Optical and IR Spectral Data of Complexes 3-7

no.	R	UV-vis (CH ₂ Cl ₂) λ _{max} , nm (ε)			IR (KBr) ν _{Mo=O} , cm ⁻¹		
3	C ₆ H ₅	328 (6272)	309 (20 100)	301 (20 300)	249 (22 910)	929, 898	
4	4-MeC ₆ H ₄	337 (6761)	306 (22 755)	299 (23 666)	253 (23 442)	930, 892	
5	2-MeC ₆ H ₄	325 (6761)	310 (22 500)	295 (24 100)	260 (24 000)	931, 893	
6	4-ClC ₆ H ₄	335 (6310)	307 (20 100)	295 (20 640)	269 (25 120)	933, 898	
7	4-MeOC ₆ H ₄	360 (5643) 325 (7080)	309 (23 785)	302 (18 486)	252 (22 390)	924, 889	

Table III. Summary of ¹H NMR Data of Complexes 3-7

proton type	chem shifts, ppm relative to CDHCl ₂ (intensities), in CD ₂ Cl ₂ soln ^a				
	3	4	5	6	7
aromatic	7.43 (6) ^m 7.20 (4) ^m	6.68 (4) ^d 7.07 (4) ^d	6.45 (2) ^m 6.86 (2) ^m	7.43 (4) ^d 7.52 (4) ^d	6.41 (4) ^m 7.09 (4) ^d
CH ₃		2.06 (6) ^s	2.53 (6) ^s		3.57 (6) ^s
bpy					
H(1),(10)	9.99	9.96	9.58	9.96	9.95
H(2),(9)	7.74	7.74	7.58	7.74	7.74
H(3),(8)	8.05	8.04	8.07	7.83	8.06
H(4),(7)	8.06	8.06	8.14	8.06	8.11

^a Superscripts denote signal multiplicities, where observable (m, multiplets; d, doublets; s, singlets).

and the CH₂Cl₂ phases were washed with water and dried with MgSO₄. The CH₂Cl₂ phases were finally isolated and evaporated to a volume of 5 cm³. Upon the addition of hexane, the products precipitated, were collected by vacuum filtration, washed with hexane, and dried. For purification, the crude products were recrystallized from 1:1 CH₂Cl₂-ethanol. Yields of products generally were in the order of 50-76% as partial decomposition occurred during recrystallization, especially in the case of complexes 3, 5, and 6. Analytical data and physical properties are summarized in Tables I and II.

Thermolysis. Thermolysis of the solid complexes was conducted in rubber serum-capped test tubes at 200 °C under argon for 30 min. The soluble organic thermolysis products were identified by ¹H NMR measurements of filtered extracts of the thermolysis residues in CD₂Cl₂. These consisted essentially of mixtures of arenes and the corresponding biphenyls derived from the aryl moieties. The biphenyls were the main aryl group derived from pyrolysis products; the observed R-R/RH ratios depend somewhat on the pyrolysis conditions chosen and were typically 1.61 for 3, 6.25 for 4 and 5, and 2.0 for 6 and 7.

Structural Analysis. Crystals of 7 as obtained from CH₂Cl₂ were suitable for X-ray structural analysis. The structure was solved by conventional Patterson and difference Fourier techniques. An outline of crystallographic and data collection parameters is given in Table IV. A crystal of approximate dimensions 0.2 × 0.25 × 0.3 mm was mounted on an Enraf-Nonius CAD4 automated diffractometer for data collection. The triclinic P1 unit cell dimensions were determined by least-squares fit of 25 reflections obtained by automatic centering on the diffractometer. Intensity data (294 K) were measured by the θ-2θ step-scan technique with Mo Kα radiation (λ = 0.71073 Å) from a graphite monochromator. A total of 3849 Bragg reflections were examined out to 2θ = 45°. The intensities of three standard reflections measured after each 8000 s of X-ray exposure showed no systematic change during data collection. Orientation was maintained by checking the centering of three reflections after every 200 reflections and recentering 25 reflections and calculating a new orientation matrix if any of the three were significantly off center. Empirical absorption correction based on ψ scans gave

Table IV. Crystallographic and Data Collection Parameters

space group	P1
a, Å	8.492 (2)
b, Å	11.340 (2)
c, Å	13.076 (2)
α, deg	65.8 (1)
β, deg	78.9 (1)
γ, deg	71.4 (1)
T, K	295 (1)
V, Å ³	1086 (1)
ρ, g/cm ³	1.524 (1)
Z	2
fw	498.4
total no. of observns	3849
no. of indepnd observns	2820
no. of indepnd observns above 2σ	2397
diffractometer	Enraf-Nonius CAD-4
cryst size, mm	0.2 × 0.25 × 0.3
λ, Å (Mo Kα)	0.71073
μ, cm ⁻¹	6.20
absorptn correctn	none
transmissn range	0.94-1.00
scan speed	variable to maintain 3% counting statistics to max time of 90 s/scan
θ/2θ scan	96 steps/scan
scan width (θ)	(0.60 + 0.35 tan θ)°
R(F _o)	0.025
R _w (F _o)	0.032
S	1.10
max ρ (e/Å ³) on final diff Fourier	0.60

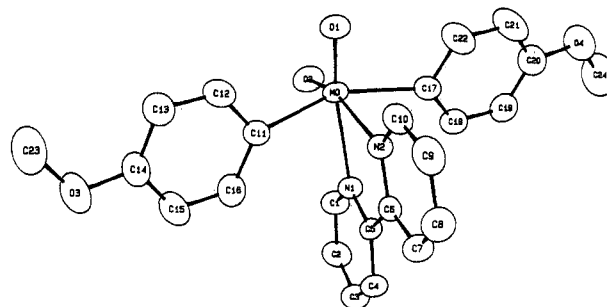


Figure 1. Perspective view of 7 with atoms numbered.

a relative transmission range from 94 to 100% (μ = 6.2 cm⁻¹). The structure was solved by Patterson and Fourier methods. Least-squares refinement, minimizing Σw(|F_o| - |F_c|)², where w = 4F_o/(σ_{counting}² + (0.040F_o)²), converged with R = Σ(|F_o| - |F_c|)/ΣF_o = 0.025 and R_w = {Σw(|F_o| - |F_c|)²/ΣwF_o²}^{1/2} = 0.032.

Hydrogen atoms were located and held in near "ideal" X-ray positions. The largest shift on the last cycle was 0.10 times its estimated standard deviation, and the error in an observation of unit weight was 1.10. Atomic scattering factors were taken from

Table V. Positional Parameters and Their Estimated Standard Deviations

atom	x	y	z
Mo	0.04862 (3)	0.26323 (2)	0.24988 (2)
O(1)	-0.0559 (3)	0.2100 (2)	0.3778 (2)
O(2)	-0.0784 (3)	0.3049 (2)	0.1464 (2)
O(3)	0.4067 (4)	-0.3391 (2)	0.2866 (2)
O(4)	-0.1563 (4)	0.8522 (2)	0.2528 (2)
N(1)	0.2643 (3)	0.3166 (2)	0.1208 (2)
N(2)	0.2839 (3)	0.2272 (2)	0.3387 (2)
C(1)	0.2450 (4)	0.3590 (3)	0.0115 (3)
C(2)	0.3737 (4)	0.3785 (3)	-0.0701 (3)
C(3)	0.5294 (4)	0.3538 (3)	-0.0373 (3)
C(4)	0.5502 (4)	0.3138 (3)	0.0752 (3)
C(5)	0.4157 (3)	0.2946 (3)	0.1530 (2)
C(6)	0.4255 (3)	0.2476 (3)	0.2755 (2)
C(7)	0.5686 (4)	0.2250 (3)	0.3241 (3)
C(8)	0.5670 (4)	0.1804 (3)	0.4389 (3)
C(9)	0.4226 (5)	0.1611 (3)	0.5032 (3)
C(10)	0.2832 (4)	0.1847 (3)	0.4507 (3)
C(11)	0.1944 (4)	0.0614 (3)	0.2572 (2)
C(12)	0.1402 (4)	-0.0435 (3)	0.3421 (3)
C(13)	0.2044 (5)	-0.1771 (3)	0.3544 (3)
C(14)	0.3316 (5)	-0.2110 (3)	0.2812 (3)
C(15)	0.3912 (6)	-0.1117 (4)	0.1974 (4)
C(16)	0.3221 (5)	0.0218 (3)	0.1843 (4)
C(17)	0.0202 (4)	0.4647 (3)	0.2469 (2)
C(18)	0.0615 (4)	0.5722 (3)	0.1588 (3)
C(19)	0.0054 (4)	0.7035 (3)	0.1564 (3)
C(20)	-0.0919 (5)	0.7283 (3)	0.2461 (3)
C(21)	-0.1299 (6)	0.6226 (4)	0.3368 (3)
C(22)	-0.0743 (6)	0.4941 (4)	0.3373 (3)
C(23)	0.3482 (7)	-0.4451 (4)	0.3724 (4)
C(24)	-0.1260 (7)	0.9644 (4)	0.1591 (4)

Table VI. Selected Bond Distances (Å)

Mo-O(1)	1.700 (2)	C(5)-C(6)	1.478 (4)
-O(2)	1.712 (2)	C(11)-C(12)	1.389 (5)
-C(11)	2.199 (3)	C(11)-C(16)	1.389 (5)
-C(17)	2.205 (3)	C(12)-C(13)	1.387 (5)
-N(1)	2.291 (2)	C(13)-C(14)	1.367 (5)
-N(2)	2.343 (2)	C(14)-C(15)	1.365 (6)
N(1)-C(1)	1.334 (4)	C(15)-C(16)	1.387 (6)
N(1)-C(5)	1.345 (4)	C(17)-C(18)	1.380 (5)
N(2)-C(5)	1.350 (4)	C(17)-C(28)	1.393 (5)
N(2)-C(6)	1.341 (4)	C(18)-C(19)	1.399 (5)
N(2)-C(10)	1.343 (4)	C(19)-C(20)	1.373 (5)
av N-C	1.343 (4)	C(20)-C(21)	1.372 (6)
C(1)-C(2)	1.376 (5)	C(21)-C(22)	1.378 (6)
C(2)-C(3)	1.376 (5)	av phenyl C-C	1.382 (5)
C(3)-C(4)	1.379 (5)	O(3)-C(14)	1.365 (4)
C(4)-C(5)	1.385 (4)	O(3)-C(23)	1.418 (6)
C(6)-C(7)	1.382 (5)	O(4)-C(20)	1.369 (4)
C(7)-C(8)	1.372 (5)	O(4)-C(24)	1.413 (7)
C(8)-C(9)	1.382 (5)		
av bpy C-C	1.378 (5)		

ref 5 and included anomalous scattering effects in F_o . Final atomic positional parameters are included in Table V, general displacement parameters are deposited in the supplementary material. Selected interatomic distances and angles are given in Tables VI and VII. A perspective view of the structure of 7 is shown in Figure 1.

Results and Discussion

Properties and Reactions of Complexes (Aryl)₂Mo(O)₂(bpy). Complexes 3-7 are stable to air in the solid state and in inert organic solvents under anaerobic conditions. They are also remarkably resistant to attack by protic solvents but are decomposed by strong

Table VII. Selected Interatomic Bond Angles (deg)

O(1)-Mo-O(2)	109.7 (1)	Mo-N(1)-C(1)	119.7 (2)
N(1)-Mo-N(2)	68.92 (9)	Mo-N(1)-C(15)	121.4 (2)
C(11)-Mo-C(17)	153.6 (1)	Mo-N(2)-C(6)	119.2 (2)
		Mo-N(2)-C(10)	122.1 (2)
O(1)-Mo-N(2)	89.6 (1)	Mo-C(11)-C(12)	115.0 (2)
O(2)-Mo-N(1)	91.9 (1)	Mo-C(11)-C(16)	130.4 (3)
O(1)-Mo-N(1)	158.5 (1)	Mo-C(17)-C(18)	127.7 (2)
O(2)-Mo-N(2)	160.8 (1)	Mo-C(17)-C(22)	116.1 (3)
O(1)-Mo-C(11)	95.7 (1)	N(1)-C(5)-C(6)	114.9 (3)
O(1)-Mo-C(17)	94.7 (1)	N(2)-C(6)-C(15)	115.4 (3)
O(2)-Mo-C(11)	99.5 (1)	C(14)-O(3)-C(23)	118.5 (3)
O(2)-Mo-C(17)	99.7 (1)	C(20)-O(4)-C(24)	118.0 (4)
N(1)-Mo-C(11)	80.2 (1)		
N(1)-Mo-C(17)	81.2 (1)		
N(2)-Mo-C(11)	77.6 (1)		
N(2)-Mo-C(17)	78.2 (1)		
N(2)-Mo-C(11)-C(12)		101.35 (0.25)	
N(2)-Mo-C(11)-C(16)		-85.60 (0.35)	
N(2)-Mo-C(17)-C(18)		99.15 (0.31)	
N(2)-Mo-C(17)-C(22)		-92.12 (0.29)	
C(11)-Mo-C(17)-C(18)		74.73 (0.40)	
C(17)-Mo-C(17)-C(22)		-116.53 (0.33)	
C(17)-Mo-C(11)-C(12)		125.82 (0.28)	
C(17)-Mo-C(11)-C(16)		-61.13 (0.45)	
N(1)-C(5)-C(6)-N(2)		2.95 (0.38)	

angle between phenyl planes = 13.87

Table VIII. Substituent Dependence of the First Low-Energy Optical Absorption of Complexes 3-7 and of the $n_{C=O} \rightarrow \pi^*$ Transition of Similarly Substituted Benzophenone Derivatives

	substituent in the 4-position			
	H	CH ₃	Cl	CH ₃ O
λ_{max} , nm Mo complexes	328	337	337	360
benzophenones	248	258	260	280

mineral acids, concentrated CH₃COOH, by aqueous HgCl₂, and on heating with strong alkalis. Their thermal stability is comparable to that of other organomolybdenum derivatives of this type. As thermolysis occurs without prior melting of the complexes, it is evidently initiated by homolytic Mo-C bond cleavage. The aryl radicals generated predominantly dimerize to the corresponding biphenyl derivatives although some arenes are formed through H-abstraction reactions. Thermolysis temperatures may be assumed to reflect primarily variations of Mo-C bond strengths; their substituent dependence suggests that electron-donating aryl substituents have a stabilizing effect (see Table I).

Spectroscopic Properties. Comparison of the IR spectra of complexes 3-7 reveals that electron-donating substituents cause a lowering of one or both of the IR $\nu_{Mo=O}$ stretching frequencies, as expected, especially in 7 (see Table II).

In the solution absorption spectra of the complexes, three maxima are observed at or around 309, 300, and 249-269 nm. These are composites of expected allowed electronic transitions of the Mo(O)₂(bpy)- and the Mo-aryl moieties and need not be discussed in detail. Of greater interest are the absorptions extending into the visible region. Although absorptions at similar energies have previously been observed in the spectra of the dialkyl derivatives of this type and assigned to transitions involving the Mo-C σ -bonds, in the spectra of 3-7, these transitions are obscured by more intense transitions which exhibit a substituent dependence not unlike that observed for the $n_{C=O} \rightarrow \pi^*$ transition in benzophenone derivatives (Table VIII) and thus are assigned to $n_{Mo=O} \rightarrow \pi^*$ transitions involving the (aryl)Mo(O)₂ moieties. In the spectrum of 7, two $n_{Mo=O} \rightarrow \pi^*$ transitions are resolved, suggesting that

(5) *International Tables for Crystallography*; Kynoch: Birmingham, England, 1974; Vol. IV.

(6) Schrauzer, G. N.; Hughes, L. A.; Strampach, N.; Ross, F.; Ross, D.; Schlemper, E. O. *Organometallics* 1983, 2, 481.

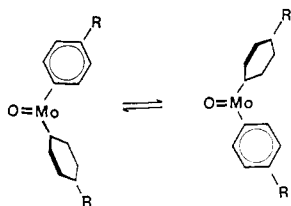


Figure 2. Proposed equilibrium conformations of complexes 3-7 in solution (coordinated bpy and second Mo=O bond not shown).

the two 4-CH₃OC₆H₄ residues are not equivalent and only one is coplanar with one Mo=O moiety. In solution, conformational equilibria such as shown in Figure 2 could exist, just as has been suggested for benzophenone derivatives on the basis of spectroscopic and dipole moment measurements.⁷ This proposal is supported by the results of the X-ray structural analysis of 7.

Structure of 7. The X-ray structural analysis of 7 revealed one CH₃OC₆H₄Mo=O moiety to be coplanar and thus provides clear evidence for π -type interactions of one Mo=O bond with the substituted arene moiety. The other CH₃OC₆H₄ residue adopts a position in which steric repulsions are minimized and π -interactions are weakened. The steric obstructions are caused by the presence of the coordinated bpy and the distorted coordination geometry around the central molybdenum atom which is intermediate between tetrahedral (as observed in 1⁸) and octahedral. The C-Mo-C bond angle of 153.6° is wider than in the dialkyl derivatives and approaches that of the dibenzyl complex of 155.5°.⁹ This suggests that repulsive interactions of the axial organic substituent with the bpy ligand are even stronger in 7 than in the corresponding dineopentyl derivative, whose C-Mo-C angle is only 145.8°. Although the two CH₃OC₆H₄ residues in 7 adopt a nearly coplanar conformation with torsional angles of -3.5° and 13.06° with respect to the Mo=O(1) axis, this distortion would lead to only a slight diminution of the respective overlap integrals as compared to the fully coplanar structure. However, even the distorted arrangement still brings the hydrogen atoms on C₁₆ and C₁₈ very close

to the bipyridyl moiety. To minimize these repulsions, the angles Mo-C(11)-C(16) and Mo-C(17)-C(18) are widened to 130.4 (3) and 127.7 (2)°, while the angles Mo-C(11)-C(12) and Mo-C(17)-C(22) are closed to 115.0 (2) and 116.1 (3)°. A repulsive interaction is also evident between the H atom on C(22) and O(1), giving rise to the differences in the angles Mo-C(11)-C(12) and Mo-C(17)-C(22). In addition, the Mo-C bonds are lengthened. The observed average Mo-C bond lengths of 2.202 (3) Å are identical with those in (C₂H₅)Mo(O)₂(bpy)¹ and thus are too long for Mo-C_{sp²} bonds. In 1, the mean Mo-C bond lengths are 2.09 Å.⁸ The remaining structural features of 7 are not remarkable; the (bpy)Mo(O)₂ moiety is essentially planar, and the respective bond distances and angles are within previously observed ranges.

Attempted Syntheses of Related Compounds. Steric as well as electronic effects of aryl substituents limit the existence of compounds of this general type. Thus, utilizing analogous preparative techniques, no stable or isolable compounds of composition R₂Mo(O)₂(bpy) could be obtained, e.g. with R = 1-naphthyl-, 2-CH₃O-C₆H₄, or 2-(CH₃)₂NC₆H₄, although the existence of 4 indicates that one ortho CH₃ group on each phenyl residue is evidently still tolerated. It is of interest to note that the corresponding complex with R = 4-(CH₃)₂NC₆H₄ could also not be obtained, at least not by the method of synthesis employed. In this case, the reaction was accompanied by the formation of reduced molybdenum species, an undesirable side reaction obviously facilitated by the strongly electron-donating effects of the N(CH₃)₂ substituents. Last but not least, in efforts to obtain compounds analogous to 1, reactions of arylmagnesium halides RMgBr (R = 2-CH₃OC₆H₄, 4-CH₃O-C₆H₄, 2-CH₃C₆H₄) with Mo(O)₂-Cl₂·2THF were explored but these failed to produce compounds of stabilities comparable to 1, illustrating the importance of the shielding effect of the ortho methyl groups.

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Supplementary Material Available: Tables of crystal data, least-squares planes, torsional angles, and general displacement parameter expressions (9 pages); a listing of observed and calculated structure factors (14 pages). Ordering information is given on any current masthead page.

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