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Crystal Structure and Molecular Geometry of an Octahedral Oxo–Alkylidene Complex of Tungsten(VI), $W({=}O)({=}CHCMe_3)(PMe_3)_2Cl_2$

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The species $W({=}O)({=}CHCMe_3)(PMe_3)_2Cl_2$, active as a catalyst in the metathesis of olefins, has been subjected to a single-crystal X-ray diffraction study. This complex crystallizes in the noncentrosymmetric orthorhombic space group $P2_12_12_1$ with $a = 11.3557$ (24) Å, $b = 11.8447$ (26) Å, $c = 14.8764$ (37) Å, $V = 2001.0$ (7) Å^3, and $Z = 4$. Diffraction data ($2\theta_{max} = 45°$; Mo Kα) were collected on a Syntex P2$_1$ automated four-circle diffractometer. Despite high-thermal motion exhibited by all atoms in the complex, the structure has been solved and refined to $R_F = 10.1\%$ and $R_{wF} = 8.6\%$ for all 2629 point-group independent reflections ($R_F = 6.2\%$ and $R_{wF} = 6.7\%$ for those 1237 independent reflections with $2\theta < 35°$). The tungsten(VI) atom is in a distorted octahedral coordination environment with the trimethylphosphine ligands in mutally trans sites [W–P(1) = 2.535 (7) Å, W–P(2) = 2.509 (8) Å] and the chloride ligands in mutually cis sites [W–Cl(1) = 2.482 (7) Å, W–Cl(2) = 2.513 (8) Å]. The neopentylidene ligand (W=C(1) = 1.986 (21) Å) and the oxo ligand (W=O = 1.697 (15) Å) are mutually cis, with an interligand angle of O=W=C(1) = 102.0 (9)°. The geometry of this octahedral $W({=}O)({=}CHCMe_3)(PMe_3)_2Cl_2$ molecule is compared to that of the trigonal-bipyramidal species $W({=}O)({=}CHCMe_3)(PEt_3)Cl_2$.

Introduction

There are now many reported alkylidene (and, particularly, neopentylidene, I) complexes of the group 5 elements tantalum

$$M{=}CHCMe_3$$

I

and niobium,[1] but this chemistry has not been carried over extensively to other transition metals. Schrock and co-workers[2] have, however, shown that alkylidene groups can be transferred from tantalum to tungsten, as indicated by eq 1.

$$Ta({=}CHCMe_3)(PR_3)_2Cl_3 + W({=}O)(OCMe_3)_4 \rightarrow W({=}O)({=}CHCMe_3)(PR_3)_2Cl_2 + Ta(OCMe_3)_4Cl \quad (1)$$

The resulting octahedral oxo–alkylidene–tungsten(VI) complexes are catalysts for the metathesis of terminal and internal olefins.[3]

We have previously reported[4] results of an X-ray diffraction study on a trigonal-bipyramidal oxo–alkylidene–tungsten(VI) complex, $W({=}O)({=}CHCMe_3)(PEt_3)Cl_2$, obtained by removal of one PEt$_3$ ligand from the octahedral species $W({=}O)({=}CHCMe_3)(PEt_3)_2Cl_2$. Attempts to determine the structure of the *octahedral* precursor $W({=}O)({=}CHCMe_3)(PEt_3)_2Cl$ were not successful due to a complex disorder problem.[5] We now report the results of a single-crystal X-ray diffraction study of the PMe$_3$ analogue $W({=}O)({=}CHCMe_3)(PMe_3)_2Cl_2$. Although the structural analysis suffers from some substantial problems (principally due to very high thermal motion and poor crystal quality), it does allow a comparison of interatomic parameters in closely related 5-coordinate and 6-coordinate oxo–alkylidene complexes of tungsten(VI).

Experimental Section

An almost ellipsoidal crystal of rather poor quality and with maximum orthogonal dimensions 0.3 × 0.2 × 0.2 mm was sealed into a thin-walled glass capillary and mounted on our Syntex P2$_1$ automated four-circle diffractometer. Details of data collection are given in Table I.[6]

Table I. Experimental Data for the Diffraction Study on $W({=}O)({=}CHCMe_3)(PMe_3)_2Cl_2$

(A) Crystal Parameters at 24 °C

crystal class: orthorhombic	space group: $P2_12_12_1$
$a = 11.3557$ (24) Å	$V = 2001.0$ (7) Å
$b = 11.8447$ (26) Å	$Z = 4$
$c = 14.8764$ (37) Å	ρ(calcd) = 1.64 g cm^{-3}

(B) Data Measurement

diffractometer: Syntex P2$_1$
radiation: Mo Kα ($\bar{\lambda}$ 0.710730 Å)
monochromator: pyrolytic graphite
scan type: coupled θ(crystal)-2θ(counter)
scan speed: 3.0°/min
scan width: $[2.0 + \Delta(\alpha_2-\alpha_1)]°$
2θ range: 4.0–45.0°
reflctns measd: $+h,+k,\pm l$
total reflctns: 2629 independent (corresponding to point group D_2)
abs coeff: $\mu = 65.2$ cm^{-1}; empirical absorption correction was made, based on a series of ψ scans of close-to-axial reflections, and transmission factors varied by a factor of 1.12 (average)

Table II. Discrepancy Indices for Final Model

data restriction(s)	R_F, %	R_{wF}, %	no. of data
none	10.1	8.6	2629
$2\theta < 40°$	7.7	7.6	1870
$2\theta < 35°$	6.2	6.7	1237
$\lvert F_o \rvert > \sigma(F_o)$	9.3	8.5	2522
$\lvert F_o \rvert > 3.0\sigma(F_o)$	8.7	8.4	2374
$\lvert F_o \rvert > 5.0\sigma(F_o)$	8.3	8.4	2243
$\lvert F_o \rvert > 5.0\sigma(F_o)$ and $2\theta < 40°$	6.9	7.5	1693

All crystallographic calculations were carried out with the Syntex XTL system. Data were corrected for absorption and for Lorentz and polarization factors. The structure was solved by a combination of Patterson, difference-Fourier, and full-matrix least-squares refinement. Discrepancy indices are collected in Table II.

The following technical crystallographic points should be noted.

(1) As with our previous structural study of $W({=}O)({=}CHCMe_3)(PEt_3)Cl_2$,[4] there is a very substantial amount of thermal motion in the molecule.[7] Even $B_{equ}[W] = 5.0$ Å^2, while $B_{equ}[P(1)] = 4.8$ Å^2, $B_{equ}[P(2)] = 6.7$ Å^2, $B_{equ}[Cl(1)] = 7.0$ Å^2, and $B_{equ}[Cl(2)] = 9.9$ Å^2. Thermal motion in peripheral methyl groups is even more extreme, with $B = 10.3$–15.1 Å^2.

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(1) Schrock, R. R. *Acc. Chem. Res.* **1979**, *12*, 98.

(2) Schrock, R. R.; Rocklage, S.; Wengrovius, J. H.; Rupprecht, G. A.; Fellman, J. D. *J. Mol. Catal.* **1980**, *8*, 73.

(3) Wengrovius, J. H.; Schrock, R. R.; Churchill, M. R.; Missert, J. R.; Youngs, W. J. *J. Am. Chem. Soc.* **1980**, *102*, 4515.

(4) Churchill, M. R.; Missert, J. R.; Youngs, W. J. *Inorg. Chem.* **1981**, *20*, 3388–3391.

(5) Churchill, M. R.; Rheingold, A. L.; Youngs, W. J.; Schrock, R. R.; Wengrovius, J. H. *J. Organomet. Chem.* **1981**, *204*, C17.

(6) An exhaustive account of our experimental procedure has appeared previously: Churchill, M. R.; Lashewycz, R. A.; Rotella, F. J. *Inorg. Chem.* **1977**, *16*, 265.

(7) Equivalent isotropic thermal parameters are here calculated as $B_{equ} = (B_{11} + B_{22} + B_{33})/3$.

Table III. Final Parameters for the $W(=O)(=CHCMe_3)(PMe_3)_2Cl_2$ Molecule

atom	*x*	*y*	*z*	B_{iso}, Å^2
W	0.084 69 (10)	0.305 50 (9)	0.042 04 (7)	
P(1)	0.221 19 (66)	−0.124 75 (57)	−0.011 89 (41)	
P(2)	−0.059 79 (80)	0.137 01 (63)	0.137 36 (53)	
Cl(1)	−0.030 49 (68)	−0.131 98 (57)	0.100 12 (56)	
Cl(2)	0.196 23 (77)	0.004 66 (92)	0.186 48 (49)	
O	0.1729 (15)	0.1410 (12)	0.0149 (12)	
C(1)	−0.0248 (21)	0.0252 (22)	−0.0620 (13)	
C(2)	−0.0502 (23)	0.0793 (23)	−0.1478 (22)	
C(3)	−0.1755 (36)	0.1106 (35)	−0.1485 (26)	10.4 (11)
C(4)	−0.0323 (40)	0.0029 (38)	−0.2269 (29)	12.5 (13)
C(5)	0.0318 (45)	0.1821 (41)	−0.1701 (31)	14.0 (15)
C(6)	0.3623 (32)	−0.0679 (30)	−0.0361 (24)	10.3 (10)
C(7)	0.2606 (35)	−0.2319 (31)	0.0621 (24)	10.6 (10)
C(8)	0.1898 (39)	−0.1957 (38)	−0.1100 (28)	12.1 (12)
C(9)	−0.0606 (41)	0.1019 (35)	0.2557 (27)	11.9 (12)
C(10)	−0.0366 (46)	0.2815 (42)	0.1222 (34)	15.1 (16)
C(11)	−0.2130 (36)	0.1188 (33)	0.1114 (24)	10.4 (11)

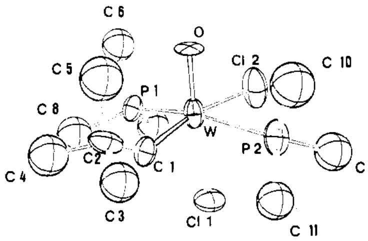

Figure 1. Labeling of atoms in the $W(=O)(=CHCMe_3)(PMe_3)_2Cl_2$ molecule. Hydrogen atoms are omitted for clarity (ORTEP-II diagram, 30% ellipsoids).

(2) Our refinement consisted of 109 variables against all 2629 point-group-independent reflections to $2\theta = 45°$ (NO:NV = 24.1:1), leading to $R_F = 10.1\%$ and $R_{wF} = 8.6\%$. Clearly, with the large degree of thermal motion, we would have obtained cosmetically more pleasing residuals if we had restricted our attention to a smaller range of data (e.g., $R_F = 6.2\%$ and $R_{wF} = 6.7\%$ for all 1237 data with $2\theta < 35°$).

(3) Reversal of atomic coordinates (x, y, z to $x, y, -z$) gave higher discrepancy indices so the crystal chirality originally chosen is correct.

(4) Despite all the above intrinsic problems, this material is the only octahedral alkylidene yet to be found in ordered crystalline form.

Discussion

The crystal consists of discrete molecular units separated by normal van der Waals distances; there are no abnormally short intermolecular contacts.

Final atomic parameters are given in Table III. The labeling of atoms is illustrated in Figure 1. Interatomic distances and angles are collected in Table IV.

The molecule consists of a central tungsten(VI) atom which has a rather distorted octahedral coordination geometry. The angles between mutually trans ligands (in increasing order) are P(1)—W—P(2) = 160.6 (3)°, Cl(2)—W═C(1) = 167.9 (7)°, and Cl(1)—W═O = 172.7 (6)°. Angles between the trimethylphosphine groups and the oxo or alkylidene ligands are all substantially increased from 90° [P(1)—W═O = 97.1 (6)°, P(2)—W═O = 97.7 (6)°, P(1)—W═C(1) = 96.5 (7)°, and P(2)—W═C(1) = 92.7 (7)°].

Table IV

(A) Intramolecular Distances (Å)			
W=C(1)	1.986 (21)	W=O	1.697 (15)
W–P(1)	2.535 (7)	W–Cl(1)	2.482 (7)
W–P(2)	2.509 (8)	W–Cl(2)	2.513 (8)
C(1)–C(2)	1.458 (38)	C(2)–C(4)	1.499 (54)
C(2)–C(3)	1.471 (48)	C(2)–C(5)	1.568 (57)
P(1)–C(6)	1.775 (37)	P(2)–C(9)	1.808 (41)
P(1)–C(7)	1.739 (37)	P(2)–C(10)	1.747 (51)
P(1)–C(8)	1.721 (43)	P(2)–C(11)	1.795 (42)
(B) Angles around the Tungsten Atom (Deg)			
P(1)–W–P(2)	160.63 (25)	P(2)–W=C(1)	92.66 (70)
P(1)–W–Cl(1)	82.53 (24)	Cl(1)–W–Cl(2)	82.73 (27)
P(1)–W–Cl(2)	82.77 (26)	Cl(1)–W=O	172.72 (58)
P(1)–W=O	97.06 (57)	Cl(1)–W=C(1)	85.23 (69)
P(1)–W=C(1)	96.52 (69)	Cl(2)–W=O	90.01 (58)
P(2)–W–Cl(1)	81.29 (26)	Cl(2)–W=C(1)	167.93 (70)
P(2)–W–Cl(2)	84.70 (28)	O=W=C(1)	102.02 (87)
P(2)–W=O	97.65 (58)		
(C) Other Angles (Deg)			
W–C(1)–C(2)	142.4 (19)	W–P(1)–C(6)	109.9 (12)
C(1)–C(2)–C(3)	108.0 (26)	W–P(1)–C(7)	119.1 (13)
C(1)–C(2)–C(4)	113.3 (27)	W–P(1)–C(8)	119.7 (15)
C(1)–C(2)–C(5)	114.2 (27)	W–P(2)–C(9)	116.0 (14)
C(3)–C(2)–C(4)	106.1 (29)	W–P(2)–C(10)	108.7 (17)
C(3)–C(2)–C(5)	112.2 (30)	W–P(2)–C(11)	116.9 (12)
C(4)–C(2)–C(5)	102.9 (30)		

Table V. Distances (Å) in Octahedral $W(=O)(=CHCMe_3)_2Cl_2$ and Trigonal-Bipyramidal $W(=O)(=CHCMe_3)(PEt_3)Cl_2$

bond	OCT[a]	TBP[b]	bond	OCT[a]	TBP[b]
W=O	1.697 (15)	1.661 (11)	W–Cl	2.482 (7)	2.379 (5)
W=C(1)	1.986 (21)	1.882 (14)		2.513 (8)	2.389 (5)
			W–P	2.509 (8)	2.518 (4)
				2.535 (7)	

[a] Octahedral species (this work). [b] Trigonal-bipyramidal species (see ref 4).

Of particular interest in the present molecule are the tungsten–alkylidene bond length [W═C(1) = 1.986 (21) Å] and the W═C(1)—C(2) angle of 142.4 (19)°. The tungsten–alkylidene distance may be compared to values of 1.882 (14) Å in the trigonal-bipyramidal species $W(=O)(=CHCMe_3)(PEt_3)Cl_2$[4] and 1.942 (9) Å in the square-pyramidal species $W(\equiv CCMe_3)(=CHCMe_3)(CH_2CMe_3)(dmpe)$.[8] The present W–C(α)–C(β) angle of 142.4 (19)° may be compared to values of 140.6 (11) and 150.4 (8)° in these same two molecules.

This range of angles seems to be rather lower than those found in neopentylidene complexes of tantalum [range 150.4 (5)–170.2 (2)°: see Table VII of ref 4]. This observation may require modification as more structural studies of tungsten neopentylidene complexes are carried out. We should note, however, that W(VI) complexes are less likely to be very "electron poor" (i.e., far short of an 18-electron configuration) than are Ta(V) complexes.[9] Similar arguments apply to W(IV) vis à vis Ta(III).

One peculiar result of the present analysis is as follows. The *cis* C(1)═W═O angle is *increased* from the regular octahedral value of 90 to 102.0 (9)°. The analogous cis-diequatorial angle in $W(=O)(=CHCMe_3)(PEt_3)Cl_2$[4] is similar, with a value of 106.7 (6)°; here, though, the cis angle is *decreased* from the ideal trigonal angle of 120°. It appears, therefore, that the C═W═O fragment in these complexes has a more-or-less fixed geometry, regardless of the detailed coordination about the tungsten atom.

A comparison of metal–ligand distances in the present octahedral species, $W(=O)(=CHCMe_3)(PMe_3)_2Cl_2$, with those in the trigonal-bipyramidal species $W(=O)(=CHCMe_3)(PEt_3)Cl_2$ is provided in Table V. As can clearly be seen, most bond distances in the octahedral species are substantially longer than those in the trigonal-bipyramidal complex [W—Cl by

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(8) (a) Churchill, M. R.; Youngs, W. J. *Inorg. Chem.* **1979**, *18*, 2454. (b) Churchill, M. R.; Youngs, W. J. *J. Chem. Soc., Chem. Commun.* **1979**, 321.

(9) For example, the metal atom in $TaMe_5$ is associated with 10 outer electrons, while that in WMe_6 is associated with 12 outer electrons. There is also a greater tendency for tungsten to expand its number of coordination sites beyond six.

~0.11 Å, W=C by ~0.10 Å, W=O by 0.036 Å, but W—P by only ~0.004 Å (average)].

Finally, we emphasize that these molecular oxo–alkylidene complexes of tungsten(VI) are closely related to certain heterogeneous tungsten(VI) and molybdenum(VI) catalysts used industrially for olefin metathesis reactions.[10-13]

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Registry No. W(=O)(=CHCMe$_3$)(PMe$_3$)$_2$Cl$_2$, 76603-92-4.

Supplementary Material Available: Listings of observed and calculated structure-factor amplitudes, anisotropic thermal parameters, calculated positions for hydrogen atoms, and data-processing formulas (18 pages). Ordering information is given on any current masthead page.

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(10) Calderon, N.; Lawrence, J. P.; Ofstead, E. A. *Adv. Organomet. Chem.* **1979**, *17*, 449 and references contained therein.

(11) Katz, T. J. *Adv. Organomet. Chem.* **1977**, *16*, 283 and references contained therein.

(12) Calderon, N.; Ofstead, E. A.; Judy, W. A. *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 401.

(13) Haines, R. J.; Leigh, G. J. *Chem. Soc. Rev.* **1975**, *4*, 155.

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Iridium Carbonyl Clusters. 5.[1-4] Crystal Structure and Molecular Geometry of $(\eta^5\text{-}C_5H_5)_2W_2Ir_2(CO)_{10}$

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The bimetallic species $(\eta^5\text{-}C_5H_5)_2W_2Ir_2(CO)_{10}$ has been examined via a single-crystal X-ray diffraction study. This complex crystallizes in the centrosymmetric triclinic space group $P\bar{1}$ [C_i^1, No. 2] with a = 8.503 (2) Å, b = 9.238 (2) Å, c = 15.659 (6) Å, α = 87.40 (3)°, β = 76.61 (3)°, γ = 73.12 (2)°, V = 1144.7 (5) Å^3, and ρ(calcd) = 3.37 g cm^{-3} for Z = 2 and mol wt 1162.4. Diffraction data were collected with a Syntex P2$_1$ diffractometer, and the structure was refined to R_F = 3.6% for 2998 reflections with 3° < 2θ < 45° (Mo Kα radiation). The molecule is based upon a closed tetrahedral W$_2$Ir$_2$ core. Each iridium atom is linked to three terminal carbonyl ligands, while the two tungsten atoms are each associated with an η^5-cyclopentadienyl ligand and with two carbonyl ligands. There are some indications that the two most bent carbonyl ligands may have some "semibridging" character [W(1)–WC(11)–WO(11) = 167.2 (13)° with W(2)···WC(11) = 2.794 (14) Å; W(2)–WC(21)–WO(21) = 171.8 (12)° with Ir(1)···WC(21) = 2.835 (14) Å]. The metal–metal distances (Å) are Ir(1)–Ir(2) = 2.722 (1), W(1)–W(2) = 2.991 (1), Ir(1)–W(1) = 2.796 (1), Ir(1)–W(2) = 2.863 (1), Ir(2)–W(1) = 2.833 (1), and Ir(2)–W(2) = 2.847 (1).

Introduction

The "mixed-metal" cluster complexes $(\eta^5\text{-}C_5H_5)WIr_3(CO)_{11}$ and $(\eta^5\text{-}C_5H_5)_2W_2Ir_2(CO)_{10}$ have recently been synthesized by Shapley and co-workers[5] as a prelude to studying the properties of bimetallic clusters as alumina-supported catalysts. We have previously reported the detailed molecular structure of the 1:3 (W:Ir) species $(\eta^5\text{-}C_5H_5)WIr_3(CO)_{11}$[1] and now report the results of a full X-ray structural analysis of the 2:2 species, $(\eta^5\text{-}C_5H_5)_2W_2Ir_2(CO)_{10}$.[5] While these complexes are normal "60-electron" tetrahedral clusters, they are among the most crowded of the known tetranuclear derivatives.

Experimental Section

Collection of X-ray Diffraction Data for $(\eta^5\text{-}C_5H_5)W_2Ir_2(CO)_{10}$. The compound was provided by Professor J. R. Shapley in the form of opaque dark crystals. The crystal selected for data collection was close to spherical with an average dimension of 0.2 mm. The crystal was mounted and aligned on our Syntex P2$_1$ diffractometer, and intensity data were collected as described previously.[6] Details are given in Table I. The intensities were corrected for absorption with an empirical method based upon twofold interpolation (in 2θ and ϕ) between ψ-scans of close-to-axial reflections.

Table I. Experimental Data for the X-ray Diffraction Study of $(\eta^5\text{-}C_5H_5)_2W_2Ir_2(CO)_{10}$

(A) Crystal Parameters at 24 °C[a]

crystal system: triclinic	V = 1144.7 (5) Å^3
space group: $P\bar{1}$ (C_i^1, No. 2)	Z = 2
a = 8.5028 (24) Å	mol wt = 1162.4
b = 9.2377 (24) Å	ρ(calcd) = 3.37 g cm^{-3}
c = 15.6588 (64) Å	μ(Mo Kα) = 228.9 cm^{-1}
α = 87.40 (3)°	
β = 76.61 (3)°	
γ = 73.12 (2)°	

(B) Measurement of Data

diffractometer	Syntex P2$_1$
radiation	Mo Kα (λ 0.710 73 Å)
monochromator	highly oriented graphite, equatorial mode (2θ_{mono} = 12.2°)
reflctns measd	$h, \pm k, \pm l$
2θ range	3–45°
scan type	θ(crystal)–2θ(counter)
scan width	[2θ(Kα_1) – 0.8]° to [2θ(Kα_2) + 0.8]°
scan speed	3.0°/min
bkgd measmnt	stationary crystal and counter at beginning and end of 2θ scan, each for half of the scan time
reflctns collected	3346 total, merged to 2998 independent reflections
std reflctn	three measured after each 97 reflections (006, 070, 411); no decay was observed over the period of data collection

[a] Unit cell parameters were derived by a least-squares fit to the setting angles of the unresolved Mo K$\bar{\alpha}$ components of 24 reflections with 2θ between 20° and 30°. Reflections used were {620}, {$54\underline{0}$}, {533}, {504}, {457}, {439}, {$4\bar{1}9$}, {356}, {462}, {361}, {$26\bar{4}$}, and {163}.

Analysis of the standard reflections showed no evidence for crystal decay. All absorption-corrected intensities were therefore converted

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(1) Part 4, $(\eta^5\text{-}C_5H_5)WIr_3(CO)_{11}$: Churchill, M. R.; Hutchinson, J. P. *Inorg. Chem.* **1981**, *20*, 4112.

(2) Part 3, Ir$_4$(CO)$_{10}$(diars): Churchill, M. R.; Hutchinson, J. P. *Inorg. Chem.* **1980**, *19*, 2765.

(3) Part 2, Ir$_4$(CO)$_{11}$[CN(*t*-Bu)]: Churchill, M. R.; Hutchinson, J. P. *Inorg. Chem.* **1979**, *18*, 2451.

(4) Part 1, Ir$_4$(CO)$_{12}$: Churchill, M. R.; Hutchinson, J. P. *Inorg. Chem.* **1978**, *17*, 3528.

(5) Shapley, J. P.; Hardwick, S. J.; Foose, D. S.; Stucky, G. D.; Churchill, M. R.; Bueno, C.; Hutchinson, J. P. *J. Am. Chem. Soc.* **1981**, *103*, 7383.

(6) Churchill, M. R.; Lashewycz, R. A.; Rotella, F. J. *Inorg. Chem.* **1977**, *16*, 265.

0020-1669/82/1321-1359$01.25/0