$Cu_2L_2(CH_3COO)_2$ binuclear units. A portion of the chain structure is shown in Figure 1. The $Cu_2(CH_3COO)_4{\cdot}2C_2H_5OH$ dimer, shown in more detail in Figure 2, has a completely bridged structure similar to that of the copper acetate hydrate dimer. The Cu(2)···Cu(2)′ separation is 2.609 (1) Å. As shown in Figure 3, the other dimeric unit has a centrosymmetric structure with two acetate groups forming monoatomic oxygen bridges. Each bridging oxygen simultaneously occupies an in-plane coordination site on one copper(II) ion and an apical site of the other copper(II) ion. The Cu(1)···Cu(1)′ separation is 3.475 (1) Å. The Cu_2O_2 moiety is exactly planar owing to crystallographic inversion symmetry. The bridging angle at oxygen, Cu(1)–O(4)–Cu(1)′, is 98.2 (2)° and the long, out-of-plane Cu(1)–O(4)′ bond distance is 2.570 (7) Å. The second oxygen of each acetate ligand is hydrogen bonded to an apical ethanol oxygen of the next dimer in the chain. The O(5)···O(7) separation is 2.695 (15) Å, and the intermolecular Cu(1)···Cu(2) separation is 6.781 (1) Å. There are, therefore, three different Cu···Cu distances, corresponding to three distinct superexchange pathways, which alternate along the chains, according to a sequence ...*ABCBABC*..., where *A* = 2.609 (1) Å, *B* = 6.781 (1) Å, and *C* = 3.475 (1) Å.

From the interchain contacts, which are found to be always longer than the sums of van der Waals radii, it appears that the individual chains are effectively isolated.

Magnetically, the absence of any close contacts between chains that may be regarded as bonding interactions implies that interchain electron exchange between the copper atoms is essentially dipolar in character and therefore weak, on the order of 10^{-2} cm^{-1} or less.[12] Considerably stronger intrachain exchange interactions, leading to 1-D magnetic behavior, are suggested by existing structural and magnetic data for copper(II) systems involving single superexchange pathways of the same types of the distinct intrachain *A*,[3] *B*,[10] or *C*[11] superexchange pathways, respectively.

In conclusion, the present results and considerations suggest that $Cu_2L_2(CH_3COO)_2{\cdot}Cu_2(CH_3COO)_4{\cdot}2C_2H_5OH$ may be an appropriate candidate for observing a new type of 1-D, alternating-chain magnetism, characterized by three alternating exchange parameters and two *g* values, one for each type of dimer. Magnetic studies, aimed at verifying this hypothesis, are in progress.

Experimental Section

Synthesis. The new LH ligand was prepared by mixing a solution of *N*-methyl-1,3-propanediamine in absolute ethanol (0.01 mol in 30 mL) with a solution of 4,6-dimethoxysalicylaldehyde in the same solvent (0.01 mol in 30 mL). The resultant solution was heated at 50 °C for 4 h, and then it was evaporated under vacuum. A fluid residue was obtained, which was used without any further purification.

The copper complex was prepared as follows. A 0.02-mol (4.00-g) quantity of $Cu(CH_3COO)_2{\cdot}H_2O$ was added to a solution of LH (2.52 g, 0.01 mol) in absolute ethanol (50 mL). The addition was made over a period of 0.5 h, at 40 °C, under nitrogen and with constant stirring. A dark green solution was obtained, which was filtered and then allowed to cool to room temperature. Crystallization was allowed to continue for 12 h, in a dry nitrogen atmosphere. Blue-green crystals, suitable for X-ray analysis, separated and were decanted, washed twice with absolute ethanol, and dried under vacuum; yield 1.50 g (25%). An additional 2.5 g of smaller crystals of the compound was obtained after the residual solution stood at room temperature for 4 days: total yield 66%, mp 198–199 °C dec. Anal. Calcd for $C_{42}H_{68}O_{20}N_4Cu_4$: C, 41.93; H, 5.69; N, 4.66; Cu, 21.12. Found: C, 42.05; H, 5.77; N, 4.67; Cu, 21.21.

X-ray Data and Structure Solution. A crystal with dimensions 0.20 × 0.14 × 0.10 mm^3 was mounted on a computer-controlled Philips PW1100 single-crystal diffractometer equipped with graphite-monochromatized Mo Kα radiation (λ = 0.71069 Å). The crystals are triclinic. The cell dimensions, determined by a least-squares calculation based on the setting angles of 25 selected reflections, are *a* = 11.470 (3) Å, *b* = 11.202 (3) Å, *c* = 10.771 (3) Å, α = 104.33 (2)°, β = 106.32 (2)°, γ = 85.38 (2)°, and *V* = 1286.8 Å^3. The space group is $P\bar{1}$ (from intensity statitics and structural analysis). The calculated density for a cell content of two units of $C_{21}H_{34}N_2O_{10}Cu_2$ (M_r = 602) is 1.576 g·cm^{-3}. The absorption coefficient for Mo Kα is μ = 16.84 cm^{-1}. The intensities were collected at room temperature up to 2θ = 50° and the ω–2θ scan technique was employed, the scan range being 1.4° and the speed 0.05° s^{-1}. A total of 4552 independent reflections were measured, of which 2596 having $I < 3\sigma(I)$ were considered as "unobserved" and excluded from the refinement. Three standard reflections were measured periodically and showed no variation in intensity during data collection. The data were corrected for Lorentz and polarization factors. An empirical absorption correction was applied during the refinement, according to the method of Walker and Stuart.[13] Absorption correction factors are in the range 1.05–0.77.

The structure was solved by the Patterson method and refined by the full-matrix least-squares method with the SHELX-76 package of programs.[14] The hydrogen atoms (except for those of the methyl groups) were included at the calculated positions (C–H = 1.08 Å), with overall isotropic parameter *U* = 0.05 Å^2. Anisotropic thermal parameters were refined for the Cu, O, N, and C atoms. The refinement converged at *R*(unweighted) = 0.062 and *R*(weighted) = 0.062, for 318 parameters and 1926 observed reflections [$R_w = (\sum w(|F_0| - |F_c|)^2)^{1/2}/(\sum wF_0{}^2)^{1/2}$; $w = (\sigma^2(F_0))^{-1}$]. The atomic scattering factors were taken from ref 14 for O, N, C, and H and from ref 15 for the Cu atom. The correction for anomalous dispersion of Cu was applied.

Registry No. $Cu_2L_2(CH_3COO)_2{\cdot}Cu_2(CH_3COO)_4{\cdot}2C_2H_5OH$, 115160-72-0; LH, 115095-66-4.

Supplementary Material Available: Tables SI and SII, listing thermal parameters and the derived hydrogen positions (2 pages); tables of calculated and observed structure factors (11 pages). Ordering information is given on any current masthead page.

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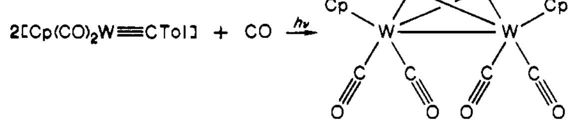
Contribution from the Departments of Chemistry,
The Pennsylvania State University,
University Park, Pennsylvania 16802,
and University of Delaware, Newark, Delaware 19716

Preparation and Structural Characterization of $[\{Cp(CO)_2W\}_2(\mu\text{-}TolCC(OH)CTol)][BF_4]{\cdot}CH_2Cl_2$

John B. Sheridan,[†] Kevin Garrett,[†] Gregory L. Geoffroy,*[,†] and Arnold L. Rheingold[‡]

Received February 16, 1988

We recently reported the photochemical preparation of a binuclear complex **1** possessing the unusual μ-TolC–C(O)–CTol ligand, which was formed by the photoinduced coupling of two carbyne ligands with a metal carbonyl[1] (eq 1). A diplatinum



(1)

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[†] The Pennsylvania State University.

[‡] University of Delaware.

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Table I. Crystallographic Parameters for **2**

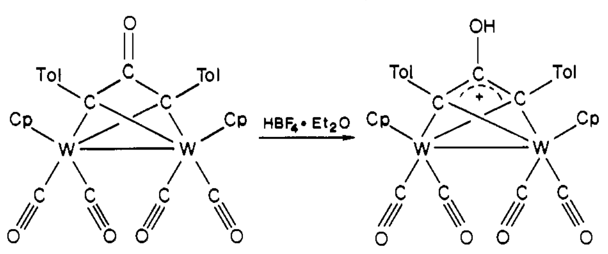
formula	$W_2C_{31}H_{25}O_5BF_4{\cdot}CH_2Cl_2$
fw	1016.91
space group	triclinic, $P\bar{1}$
a, Å	9.832 (3)
b, Å	11.540 (4)
c, Å	16.114 (6)
α, deg	75.40 (4)
β, deg	79.14 (3)
γ, deg	69.09 (2)
V, Å^3	1643 (1)
Z	2
D(calcd), g cm^{-3}	2.056
cryst size, mm	0.25 × 0.25 × 0.36
μ(Mo Kα), cm^{-1}	76.2
diffractometer	Nicolet R3m/μ
radiation	Mo Kα (λ = 0.71073 Å)
monochromator	graphite
scan method	$\theta/2\theta$
scan limits, deg	$4 \leq 2\theta \leq 52$
octants collcd	$\pm h,\pm k,\pm l$
no. of reflcns collcd	7826
no. of indep reflcns	7532
R(int),[a] %	1.8
no. of obsd reflcns ($3\sigma F_o$)	5051
std reflcns	3 std/197 reflcns (decay <2%)
$R(F)$,[b]	3.67
$R(wF)$,[b] %	3.89
GOF[c]	1.028
Δ/σ	0.162
$\Delta(\rho)$, e Å^{-3}	1.01 [0.89 Å, W(1)]
N_o/N_v	12.1

[a] $R(\text{int}) = \sum|F - \langle F\rangle|/F$. [b] $R(F) = \sum(|F_o| - |F_c|)/\sum|F_o|$; $R(wF) = \sum(w^{1/2}(|F_o| - |F_c|))/(w^{1/2}|F_o|)$. [c] $\mathrm{GOF} = [\sum w||F_o| - |F_c||/(N_o - N_v)]^{1/2}$.

complex with a similar μ-C(Ph)–C(O)–CPh ligand has been reported,[2] but there have been no investigations of the chemistry of this unusual ligand. We accordingly explored the reactivity of complex **1** with several unsaturated organic substrates under various reaction conditions, but in no case was tractable chemistry observed. However, one reaction that this complex undergoes with great facility is protonation, a reaction of interest because of the several sites available for H$^+$ addition. Protonation could occur at the W–W bond to give a μ-hydride complex, at one of the Tol–C carbons to form a ligand tethered to one metal by a CH(Tol) center, or at the carbonyl oxygen to form a hydroxy group. A priori, there is no basis to choose between these, but as described herein protonation occurs exclusively at the last site to form a novel μ-CTol–C(OH)–CTol ligand.

Results and Discussion

Complex **1** reacts rapidly with HBF_4 to yield a single product, which has been spectroscopically and crystallographically characterized as the binuclear complex **2**, which results from protonation of the carbonyl oxygen (eq 2). This reaction can be



(2)

1 **2**

conveniently followed by IR where the most noticeable change is the immediate disappearance of the ketonic ν_{CO} band of **1** at 1568 cm^{-1}. The ^{1}H NMR spectrum of **2** shows no obvious metal hydride resonances, but a broad singlet is apparent at δ 10.45 that is attributed to the hydroxy proton. If protonation had occurred across the metal–metal bond, a distinctive hydride resonance at approximately δ −16 to −20 would be expected. Such a peak is seen at δ −17 (J_{WH} = 33 Hz) for the hydride resonance of a related complex $[Cp(CO)_2W]_2(\mu\text{-H})(\mu\text{-CTol}{\equiv}\text{CTol})]^+$ (**3**), formed by protonation of $[Cp(CO)_2W]_2(\mu\text{-CTol}{\equiv}\text{CTol})$ or via addition of $HBF_4{\cdot}Et_2O$ to $Cp(CO)_2W{\equiv}CTol$ at low temperature.[3] Complex **2** shows a weak O–H stretch at 3116 cm^{-1} in its KBr IR spectrum, providing additional evidence that protonation occurred on the carbonyl oxygen.

Table II. Atomic Coordinates (×10^4) and Isotropic Thermal Parameters (Å^2 × 10^3)

	x	y	z	U[a]
W(1)	5467.2 (3)	4577.7 (3)	2420.1 (2)	34.4 (1)
W(2)	7332.2 (4)	2093.3 (3)	3244.6 (2)	41.2 (1)
Cl(1)	1923 (5)	3607 (4)	494 (3)	109 (2)
Cl(2)	3394 (7)	1550 (5)	−400 (4)	147 (3)
Cs	2547 (19)	1944 (15)	625 (10)	115 (9)
O(1)	8223 (9)	5467 (8)	2047 (6)	94 (5)
O(2)	4914 (10)	5643 (7)	4114 (4)	80 (4)
O(3)	10125 (9)	2786 (11)	2304 (6)	100 (5)
O(4)	7547 (9)	3498 (10)	4629 (5)	86 (5)
O(5)	4530 (8)	1758 (7)	2242 (4)	59 (3)
C(1)	7271 (11)	5100 (9)	2186 (6)	56 (4)
C(2)	5170 (11)	5224 (9)	3528 (5)	52 (4)
C(3)	9075 (10)	2577 (11)	2640 (6)	63 (5)
C(4)	7408 (9)	3020 (10)	4115 (6)	55 (4)
C(5)	5182 (9)	2420 (7)	2473 (5)	38 (3)
C(6)	4934 (8)	2914 (7)	3235 (5)	33 (3)
C(7)	6421 (9)	2757 (8)	1988 (5)	72 (5)
C(11)	7633 (14)	18 (10)	3108 (8)	72 (5)
C(12)	8994 (13)	87 (11)	3153 (7)	77 (5)
C(13)	9033 (13)	356 (12)	3950 (8)	89 (6)
C(14)	7646 (13)	428 (10)	4438 (7)	78 (5)
C(15)	6786 (13)	231 (9)	3899 (8)	76 (5)
C(16)	4191 (10)	5058 (9)	1205 (6)	55 (4)
C(17)	3142 (11)	5221 (10)	1939 (7)	63 (4)
C(18)	3220 (11)	6174 (10)	2311 (7)	64 (4)
C(19)	4329 (11)	6657 (8)	1787 (6)	59 (4)
C(20)	4900 (11)	5946 (9)	1112 (6)	57 (4)
C(21)	8137 (10)	3102 (10)	658 (6)	55 (4)
C(22)	8906 (10)	2808 (10)	−121 (6)	57 (4)
C(23)	8748 (10)	1883 (10)	−455 (5)	56 (4)
C(24)	7744 (12)	1292 (9)	−9 (6)	64 (5)
C(25)	6971 (11)	1570 (9)	778 (6)	59 (4)
C(26)	7177 (8)	2472 (7)	1148 (5)	39 (3)
C(27)	9620 (12)	1538 (12)	−1295 (6)	84 (6)
C(31)	3916 (10)	2847 (9)	4793 (5)	50 (4)
C(32)	2727 (10)	2977 (9)	5434 (5)	49 (4)
C(33)	1350 (10)	3108 (9)	5279 (6)	53 (4)
C(34)	1172 (10)	3107 (10)	4453 (6)	62 (5)
C(35)	2352 (9)	3008 (9)	3799 (6)	53 (4)
C(36)	3733 (8)	2913 (8)	3949 (5)	39 (3)
C(37)	67 (11)	3259 (11)	5985 (7)	74 (5)
B	6695 (1)	695 (1)	7006 (1)	60 (5)
F(1)	6860 (2)	−292 (1)	6663 (1)	141 (6)
F(2)	5272 (2)	1306 (4)	7175 (5)	175 (8)
F(3)	7320 (7)	1479 (4)	6449 (2)	148 (6)
F(4)	7330 (7)	287 (2)	7735 (3)	175 (8)

[a] Equivalent isotropic U defined as one-third of the trace of the orthogonalized $\mathbf{U}_{ij}$ tensor.

The ^{13}C NMR resonance of the ketonic carbon of **1** at δ 177.3 shifts to δ 160.9 in **2** upon protonation. Similarly, the Tol*C* NMR signals also shift upfield by 14 ppm from δ 78.0 in **1** to δ 64.2 in **2**. These changes not only confirm the site of protonation as the oxygen atom of the μ-CTol-C(O)-CTol ligand, but also suggest that all three carbons of the new bridging ligand in **2** are affected by protonation of the ketonic oxygen. In order to fully define the nature of this new bridging ligand, a crystallographic study was undertaken. (Tables I and II.)

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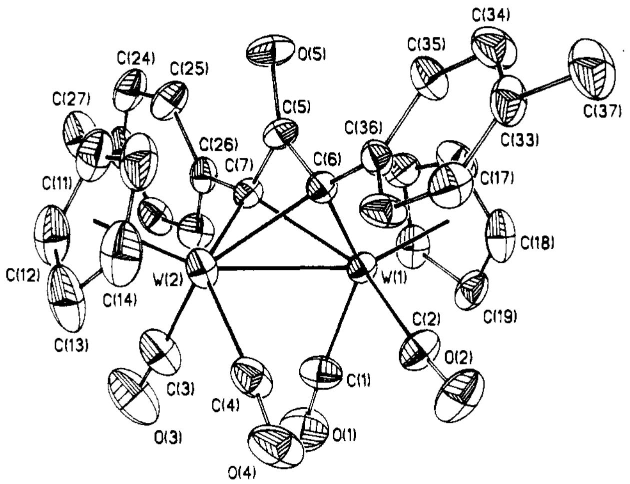

Figure 1. Molecular structure and labeling scheme for the cation of $2\cdot CH_2Cl_2$. Thermal ellipsoids are drawn at the 40% probability level.

Table III. Selected Bond Distances, Bond Angles, Dihedral Angles, and Torsion Angles for **1** and **2**

	1	**2**
(a) Bond Distances (Å)		
W(1)–W(2)	2.956 (1)	2.9311 (7)
W(1)–CNT(1)[a]	2.01 (1)	1.998 (7)
W(2)–CNT(2)	1.99 (1)	2.002 (8)
W(1)–C(1)	2.021 (14)	2.012 (12)
W(1)–C(2)	1.998 (18)	2.036 (10)
W(1)–C(6)	2.221 (14)	2.208 (8)
W(1)–C(7)	2.229 (14)	2.205 (9)
W(2)–C(3)	2.017 (14)	1.994 (11)
W(2)–C(4)	2.020 (15)	1.992 (12)
W(2)–C(6)	2.193 (13)	2.207 (7)
W(2)–C(7)	2.219 (15)	2.214 (9)
W(1)···C(5)	2.639 (14)	2.582 (10)
W(2)···C(5)	2.648 (15)	2.518 (9)
C(5)–C(6)	1.516 (19)	1.426 (12)
C(5)–C(7)	1.526 (17)	1.440 (12)
C(5)–O(5)	1.222 (18)	1.311 (14)
(b) Bond Angles (deg)		
C(6)–W(1,2)–C(7) av	62.2 (5)	61.5 (3)
W(1)–C(6,7)–W(2) av	83.7 (4)	83.2 (3)
C(6)–C(5)–C(7)	97.5 (11)	104.0 (9)
CNT(1,2)–W(1,2)–W(2,1) av	155.5 (7)	158.1 (4)
CNT–W(1,2)–C(7,6) av	116.4 (7)	117.1 (4)
CNT–W(1,2)–C(6,7) av	109.8 (7)	112.2 (4)
W(1,2)–C(6,7)–C(26,36) av	104.7 (11)	103.9 (5)
O(5)–C(5)–C(6,7) av	131.2 (11)	128.0 (6)
W(1,2)–C(6,7)–C(5) av	88.0 (8)	86.2 (6)
C(5)–C(6,7)–C(26,36) av	123.6 (12)	128.0 (9)
Dihedral Angle		
[W(1)–W(2)–C(6)]–[W(1)–W(2)–C(7)]	87.5 (5)	86.2 (3)
Torsion Angle		
CNT(1)–W(1)–W(2)–CNT(2)	29.6 (5)	22.6 (3)

[a] CNT(1) and CNT(2) are the centroids of the η^5-C_5H_5 rings.

An ORTEP drawing of complex **2** is shown in Figure 1, and Table III presents a comparison of the structural parameters of the W_2C_3 cores of both complexes **1** and **2**. The only distances and angles that show an appreciable change in going from **1** to **2** are the average W(1)–C(5) and W(2)–C(5) distances ($\Delta = -0.095$ Å), C(5)–C(6) ($\Delta = -0.09$ Å), C(5)–C(7) ($\Delta = -0.086$ Å), C(5)–O(5) ($\Delta = +0.089$ Å), and the angle C(6)–C(5)–C(7) ($\Delta = +6.5°$). The increase in the C(5)–O(5) distance is consistent with the conversion of the carbonyl group of the bridging ligand in **1** to a hydroxy group in **2** via H^+ addition to the oxygen atom. The decrease in the metal–C(5) distances, although significant, still leaves C(5) as an average nonbonding distance of 2.55 Å from W(1) and W(2). The changes in the C(5)–C(6) and C(5)–C(7) distances and the extension of the C(6)–C(5)–C(7) angle by 6.5° are most significant and indicate that the positive charge in **2** is delocalized over the three-carbon bridging unit making it similar to a cationic allyl ligand. Indeed, the C(5)–C(6) and C(5)–C(7) bond lengths in **2** of 1.426 and 1.440 Å, respectively, are typical of those found in allyl ligands.[4]

Complex **2** displays a new type of bridging hydrocarbyl ligand, namely a μ-CR–CR′–CR ligand that formally donates 5 electrons to the ditungsten core. Recently, Stone and co-workers have reported the synthesis of a complex $Cp^*(CO)_2W(\mu$-C(Tol)C(O)C(Me)C(Me))$Fe(CO)_3$ from the reaction of a dinuclear carbyne complex and but-2-yne.[5] This species also undergoes protonation at the oxygen atom of the bridging ligand to form a cation with a bridging C(Tol)C(OH)C(Me)C(Me) moiety.[5a] Furthermore, a dinuclear MoFe complex possessing a μ-C(Tol)C(OH)C(H) ligand exactly analogous to the hydrocarbyl ligand found in complex **2** has been proposed as a reactive intermediate in a reaction sequence studied by Stone and co-workers involving the coupling of carbyne, methylene, and carbon monoxide ligands.[5b]

Treatment of complex **2** with carbanions such as MeLi gave intractable products, although hydride ion (BH_4^- in CH_2Cl_2) and **2** give **1** as the main product. Similarly, CH_2Cl_2 solutions of **2** can be converted cleanly and quantitatively to **1** by treatment with NEt_3 or 1,8-bis(dimethylamino)naphthalene.

Experimental Section

Complex **1** was prepared as described in ref 1. All manipulations were performed under N_2 using predried solvents and standard Schlenk techniques. The instruments used were previously described.[1b]

Protonation of 1 To Give [{Cp(CO)$_2$W}$_2$(μ-CTolC(OH)CTol)][BF$_4$] (2). A CH_2Cl_2 solution (10 mL) of **1** (0.068 g, 0.08 mmol) was treated with 1–2 drops of $HBF_4\cdot Et_2O$. The orange solution immediately turned deep red in color and slightly opaque. Removal of the solvent in vacuo followed by washing the residue with Et_2O (3 × 5 mL) gave an orange solid. Recrystallization from CH_2Cl_2/Et_2O gave $\mathbf{2}\cdot CH_2Cl_2$ as orange crystals (0.074 g, 0.08 mmol, 88%). $2\cdot CH_2Cl_2$. Anal. Calcd for $C_{32}H_{27}BCl_2F_4O_5W_2$: C, 37.75; H, 2.65. Found: C, 38.17; H, 2.19. IR: $\nu_{CO}(CH_2Cl_2) = 2045$ (s), 2010 (m), 1954 (m) cm^{-1}; ν_{O-H}(KBr) = 3116 cm^{-1}. ^{1}H NMR (acetone-d_6): δ 2.34 (6 H, s, CH_3), 5.78 (10 H, s, Cp), 7.15 (4 H, d, $J = 8.5$ Hz, Tol), 7.30 (4 H, d, $J = 8.5$ Hz, Tol), 10.45 (1 H, s, O*H*). ^{13}C NMR (acetone-d_6): δ 20.9 (*C*H$_3$), 64.2 (*C*–Tol), 92.9 (Cp), 129.9, 130.2, 136.6, 142.4 (Tol), 160.9 (*C*–OH), 210.9 (*C*O).

Crystallographic Characterization of $2\cdot CH_2Cl_2$. The crystal, data collection and refinement parameters are given in Table I. Crystals of $\mathbf{2}\cdot CH_2Cl_2$ for X-ray diffraction were obtained as red prisms from diffusion of hexane into a CH_2Cl_2 solution of **2**. All reduction routines failed to find a crystal symmetry higher than triclinic. The centrosymmetric space group $P\bar{1}$ was confirmed by the computationally stable and chemically rational structure obtained. An empirical correction for absorption was applied (ψ scans, 216 data, $T_{max}/T_{min} = 1.36$). The W atom positions were obtained from a Patterson synthesis. All non-hydrogen atoms were anisotropically refined, and all hydrogen atoms (except those of the solvent molecule and the O(5)-bound H atom) were incorporated as idealized isotropic and updated contributions (d(CH) = 0.96 Å). A molecule of CH_2Cl_2 cocrystallizes with **2**. All computations used the SHELXTL (5.1) program library (G. W. Sheldrick; distributed by Nicolet XRD, Madison, WI). Atomic coordinates are given in Table II.

Acknowledgment. We thank the National Science Foundation (Grant CHE 8501548) for support of this research and for providing funds for the partial support of the crystallographic facility at the University of Delaware.

Registry No. **1**, 101992-47-6; **2**, 115512-21-5; $\mathbf{2}\cdot CH_2Cl_2$, 115512-22-6.

Supplementary Material Available: Complete lists of bond distances and angles, anisotropic thermal parameters, and hydrogen atom coordinates (5 pages); a table of structure factors (30 pages). Ordering information is given on any current masthead page.

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