relative to pertechnetate. Since this shift is outside the expected range for Tc(I) speices, we have remeasured the resonance frequency of [Tc(dmpe)$_3$]$^+$ as well as the closely related species [Tc(P(OMe)$_3$)$_6$]$^+$, also reported[5,11] as being at an anomalously low field. On the basis of our studies, it was postulated that the resonances of diamagnetic Tc(I) complex should be close to those of the [Tc(CNR)$_6$]$^+$ derivatives.

When a mixed sample of (NH$_4$)[TcO$_4$] and [Tc(dmpe)$_3$](PF$_6$) was studied there was observed only the resonance attributable to pertechnetate in the region ±500 ppm. A seven-line multiplet was observed, however, at a chemical shift of −1854 ppm. Indeed, when the spectrum (Figure 1) for a sample of [Tc(CNBu^t)6](PF$_6$) mixed with [Tc(dmpe)$_3$](PF$_6$) was obtained, resonances due to both species are observed. The technetium resonance at −1854 ppm, split by six equivalent phosphorus nuclei with $J_{\mathrm{Tc-P}} = 574$ Hz, is clearly due to the [Tc(dmpe)$_3$]$^+$ complex.[12] We also find the complex [Tc(P(OMe)$_3$)$_6$](BPh$_4$) to resonate at −1658 ppm as opposed to −422 ppm as previously reported.[5,11] The coupling constant derived from the observed septet agrees within experimental error with the data obtained from the ^{31}P spectrum.[13,14]

It is a common feature in the NMR of metal nuclei that the instrumentally imposed maximum spectral window is considerably smaller than the known chemical shift range. It seems probable that the previous workers have been measuring folded spectra that has led to their reporting incorrect chemical shifts. In all our measurements, careful consideration was given to this problem, and all spectra were obtained by using several different transmitter offsets.

If ^{99}Tc NMR is to be the useful tool for the study of technetium chemistry that we believe it to be, then it is essential that the chemical shifts for the already well-characterized species be known accurately.

Acknowledgment. This work was supported, in part, by a grant from the U.S. Department of Energy.

Registry No. [Tc(dmpe)$_3$](PF$_6$), 91491-92-8; [Tc(P(OMe)$_3$)$_6$](BPh$_4$), 97262-57-2; ^{99}Tc, 14133-76-7.

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(14) The previous incorrect ^{99}Tc NMR studies[5,11] reported the same coupling constants that we observed.

Contribution from the Dipartimento di Chimica
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Synthesis and X-ray Structure of Cu$_2$L$_2$(CH$_3$COO)$_2$·Cu$_2$(CH$_3$COO)$_4$·2C$_2$H$_5$OH (LH = *N*-Methyl-*N'*-(4,6-dimethoxysalicylidene)-1,3-propanediamine), a Novel Alternating-Chain Compound of Copper(II)[1]

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Spin–spin interaction phenomena in cluster compounds and extended systems (a topic that is relevant to several areas of chemistry, physics, and biology) have been the object of several recent reviews.[2]

Many carboxylate complexes of copper(II) display structures in which exchange-coupled dimers with four Cu–O–C–O–Cu bridges are magnetically isolated from the bulk of the sample.[3]

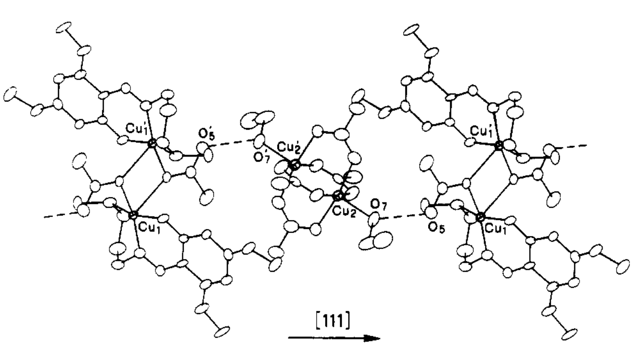

Figure 1. View of the 1-D chain structure of Cu$_2$L$_2$(CH$_3$COO)$_2$·Cu$_2$(CH$_3$COO)$_4$·2C$_2$H$_5$OH. Hydrogen atoms have been omitted for clarity.

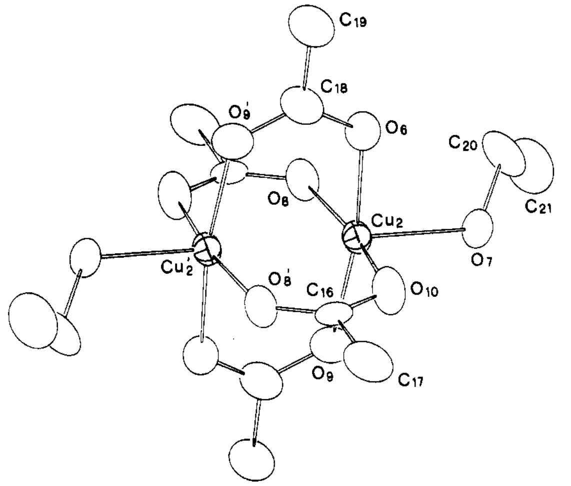

Figure 2. View of the Cu$_2$(CH$_3$COO)$_4$·2C$_2$H$_5$OH binuclear unit.

The intradimer spin coupling in these compounds is antiferromagnetic in nature, characterized by a singlet–triplet splitting ($-2J$) of about 300 cm^{-1}. Since 1951, more than 60 paper have appeared in the literature concerning the magnetic exchange problem for the copper acetic hydrate dimer,[4,5] which has become a "reference" compound for discussing exchange concepts.

Intercluster exchange interactions are, in general, far more difficult to measure and interpret than intracluster exchange interactions. To our knowledge, only one compound involving binuclear copper acetate units, Cu$_2$(CH$_3$COO)$_4$, has been designed for studying interdimer magnetic exchange, i.e. copper acetate–pyrazine.[6,7] In this compound, the binuclear acetate units are linked by the bidentate pyrazine ligand, C$_4$H$_4$N$_2$, and a linear chain is formed.

We have recently investigated the structural and magnetic properties of several copper(II) compounds containing one-atom acetate-bridged Cu$_2$O$_2$ moieties.[8-11] Both structures built of

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Table I. Fractional Atomic Coordinates in $Cu_2L_2(CH_3COO)_2 \cdot Cu_2(CH_3COO)_4 \cdot 2C_2H_5OH$

atom	*x/a*	*y/b*	*z/c*
Cu(1)	−0.0033 (1)	−0.1081 (1)	0.0901 (1)
Cu(2)	0.5422 (1)	0.5586 (1)	0.6269 (1)
O(1)	−0.0040 (8)	0.0337 (8)	0.2314 (8)
O(2)	0.1226 (9)	0.3117 (8)	0.6427 (8)
O(3)	0.2529 (9)	−0.1157 (9)	0.5919 (9)
O(4)	−0.1319 (7)	−0.0273 (8)	−0.0283 (8)
O(5)	−0.2438 (9)	−0.1755 (10)	−0.0216 (10)
O(6)	0.5394 (9)	0.3814 (8)	0.6538 (9)
O(7)	0.5964 (10)	0.6438 (12)	0.8386 (9)
O(8)	0.3682 (8)	0.5669 (9)	0.6194 (9)
O(9)	0.5348 (10)	0.7016 (9)	0.5654 (10)
O(10)	0.7018 (8)	0.5085 (10)	0.5983 (9)
N(1)	0.0874 (9)	−0.2059 (9)	0.2097 (10)
N(2)	0.0326 (10)	−0.2260 (10)	−0.0744 (10)
C(1)	0.0630 (11)	0.0512 (10)	0.3571 (11)
C(2)	0.0652 (11)	0.1689 (11)	0.4390 (11)
C(3)	0.1288 (11)	0.1949 (11)	0.5732 (12)
C(4)	0.1915 (11)	0.1000 (11)	0.6284 (12)
C(5)	0.1947 (10)	−0.0135 (12)	0.5505 (12)
C(6)	0.1287 (11)	−0.0455 (11)	0.4085 (11)
C(7)	0.2145 (16)	0.3498 (15)	0.7694 (13)
C(8)	0.3097 (15)	−0.0976 (15)	0.7345 (13)
C(9)	0.1342 (11)	−0.1693 (11)	0.3365 (12)
C(10)	0.0946 (14)	−0.3383 (12)	0.1534 (13)
C(11)	0.1746 (15)	−0.3671 (13)	0.0549 (14)
C(12)	0.1600 (12)	−0.2717 (13)	−0.0317 (14)
C(13)	−0.0538 (14)	−0.3246 (14)	−0.1670 (13)
C(14)	−0.2293 (11)	−0.0864 (12)	−0.0653 (13)
C(15)	−0.3325 (15)	−0.0478 (17)	−0.1734 (18)
C(16)	0.7127 (12)	0.4589 (12)	0.4813 (14)
C(17)	0.8453 (13)	0.4415 (16)	0.4718 (17)
C(18)	0.5029 (13)	0.2935 (14)	0.5567 (16)
C(19)	0.5044 (16)	0.1725 (15)	0.5847 (17)
C(20)	0.5422 (17)	0.6142 (22)	0.9292 (17)
C(21)	0.4914 (15)	0.7262 (20)	1.0055 (18)

Table II. Bond Distances (Å) and Angles (deg) for the $Cu_2(CH_3COO)_4 \cdot 2C_2H_5OH$ Binuclear Unit[a]

O(6)–Cu(2)	1.969 (7)	C(16)–C(17)	1.546 (15)
O(7)–Cu(2)	2.198 (9)	C(16)–O(8)″	1.201 (16)
O(8)–Cu(2)	1.971 (7)	C(18)–C(19)	1.457 (18)
O(9)–Cu(2)	1.972 (8)	C(18)–O(9)″	1.277 (19)
O(10)–Cu(2)	1.940 (8)	C(20)–C(21)	1.493 (25)
O(6)–C(18)	1.245 (17)	Cu(2)···Cu(2)″	2.609 (1)
O(7)–C(20)	1.408 (20)	O(5)···O(7)	2.695 (15)
O(10)–C(16)	1.282 (16)		

O(6)–Cu(2)–O(7)	96.7 (4)	C(18)–O(6)–C1(2)	120.1 (8)
O(6)–Cu(2)–O(8)	89.0 (3)	C(20)–O(7)–Cu(2)	120.5 (9)
O(6)–Cu(2)–O(9)	169.6 (4)	C(16)–O(10)–Cu(2)	120.4 (7)
O(6)–Cu(2)–O(10)	87.7 (3)	O(10)–C(16)–C(17)	114.6 (9)
O(7)–Cu(2)–O(8)	92.2 (3)	O(10)–C(16)–O(8)″	126.7 (10)
O(7)–Cu(2)–O(9)	93.7 (4)	C(17)–C(16)–O(8)″	118.5 (9)
O(7)–Cu(2)–O(10)	99.4 (3)	O(6)–C(18)–C(19)	116.9 (11)
O(8)–Cu(2)–O(9)	90.1 (3)	O(6)–C(18)–O(9)″	126.5 (11)
O(8)–Cu(2)–O(10)	168.2 (3)	C(19)–C(18)–O(9)″	116.6 (11)
O(9)–Cu(2)–O(10)	91.1 (3)	O(7)–C(20)–C(21)	111.4 (13)

[a] Double prime indicates atom of equivalent position $1 - x$, $1 - y$, $1 - z$.

Table III. Bond Distances (Å) and Angles (deg) for the $Cu_2L_2(CH_3COO)_2$ Binuclear Unit[a]

Cu(1)–O(1)	1.910 (8)	N(2)–C(12)	1.492 (14)
Cu(1)–O(4)	1.980 (7)	N(2)–C(13)	1.507 (16)
Cu(1)–N(1)	1.925 (9)	C(1)–C(2)	1.391 (14)
Cu(1)–N(2)	2.054 (10)	C(1)–C(6)	1.407 (13)
Cu(1)–O(4)′	2.570 (7)	C(2)–C(3)	1.393 (16)
O(1)–C(1)	1.331 (14)	C(3)–C(4)	1.397 (14)
O(2)–C(3)	1.344 (13)	C(4)–C(5)	1.343 (15)
O(2)–C(7)	1.460 (16)	C(5)–C(6)	1.471 (16)
O(3)–C(5)	1.385 (13)	C(6)–C(9)	1.418 (14)
O(3)–C(8)	1.459 (16)	C(10)–C(11)	1.550 (19)
O(4)–C(14)	1.258 (12)	C(11)–C(12)	1.555 (17)
O(5)–C(14)	1.244 (14)	C(14)–C(15)	1.528 (20)
N(1)–C(9)	1.291 (16)	Cu(1)···Cu(1)′	3.475 (1)
N(1)–C(10)	1.462 (13)	O(5)···O(7)	2.695 (15)

O(1)–Cu(1)–O(4)	88.4 (3)	O(1)–C(1)–C(2)	118.0 (9)
O(1)–Cu(1)–N(1)	93.0 (3)	O(1)–C(1)–C(6)	122.0 (9)
O(1)–Cu(1)–N(2)	162.0 (3)	C(2)–C(1)–C(6)	120.0 (9)
O(1)–Cu(1)–O(4)′	82.9 (3)	C(1)–C(2)–C(3)	122.1 (9)
O(4)–Cu(1)–N(1)	165.6 (3)	O(2)–C(3)–C(2)	116.7 (9)
O(4)–Cu(1)–N(2)	89.5 (3)	O(2)–C(3)–C(4)	124.0 (9)
O(4)–Cu(1)–O(4)′	81.2 (2)	C(2)–C(3)–C(4)	119.3 (9)
N(1)–Cu(1)–N(2)	93.5 (4)	C(3)–C(4)–C(5)	119.8 (9)
N(1)–Cu(1)–O(4)′	113.2 (3)	O(3)–C(5)–C(4)	125.9 (9)
N(2)–Cu(1)–Cu(1)′	81.7 (3)	O(3)–C(5)–C(6)	111.3 (8)
N(2)–Cu(1)–O(4)′	79.1 (3)	C(4)–C(5)–C(6)	122.8 (9)
Cu(1)–O(1)–C(1)	127.1 (7)	C(1)–C(6)–C(5)	115.9 (9)
C(3)–O(2)–C(7)	117.2 (10)	C(1)–C(6)–C(9)	125.8 (9)
C(5)–O(3)–C(8)	115.9 (9)	C(5)–C(6)–C(9)	118.2 (9)
Cu(1)–O(4)–C(14)	110.4 (6)	N(1)–C(9)–C(6)	123.5 (9)
Cu(1)–N(1)–C(9)	127.2 (7)	N(1)–C(10)–C(11)	111.5 (10)
Cu(1)–N(1)–C(10)	116.5 (7)	C(10)–C(11)–C(12)	113.8 (11)
C(9)–N(1)–C(10)	116.1 (9)	N(2)–C(12)–C(11)	113.4 (9)
Cu(1)–N(2)–C(12)	106.9 (6)	O(4)–C(14)–O(5)	123.6 (9)
Cu(1)–N(2)–C(13)	123.3 (7)	O(4)–C(14)–C(15)	117.2 (9)
C(12)–N(2)–C(13)	113.4 (9)	O(5)–C(14)–C(15)	119.2 (9)

[a] Prime indicates atom of equivalent position $-x$, $-y$, $-z$.

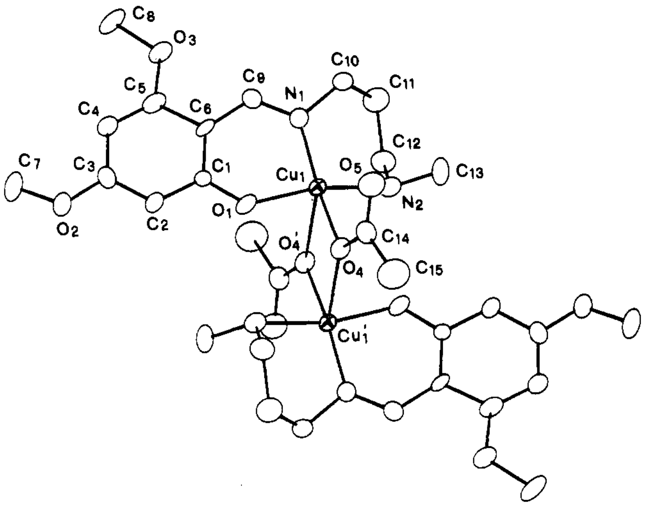

Figure 3. View of the $Cu_2L_2(CH_3COO)_2$ binuclear unit.

magnetically diluted dimers and structures involving extended magnetic interactions have been found.

We now report the synthesis and crystal structure of a novel extended system, $Cu_2L_2(CH_3COO)_2 \cdot Cu_2(CH_3COO)_4 \cdot 2C_2H_5OH$ (where LH = *N*-methyl-*N*′-(4,6-dimethoxysalicylidene)-1,3-propanediamine), in which two completely different dimeric units, involving one-atom acetate-bridged Cu_2O_2 moieties and completely bridged $Cu_2(CH_3COO)_4$ units, respectively, are connected by hydrogen-bonding interactions into an interesting and quite unusual structural chain.

Results and Discussion

The title compound was prepared by addition, at 40 °C, of $Cu(CH_3COO)_2 \cdot H_2O$ to the LH ligand dissolved in absolute ethanol, in a 1:2 ligand to metal molar ratio.

The crystal structure of the compound consists of chains parallel to the {111} direction of the triclinic space group $P\bar{1}$. Final positional parameters are listed in Table I, and the more important bond distances and angles are compiled in Tables II and III. The chains are formed by alternating $Cu_2(CH_3COO)_4 \cdot 2C_2H_5OH$ and

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$Cu_2L_2(CH_3COO)_2$ 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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Preparation and Structural Characterization of $[\{Cp(CO)_2W\}_2(\mu\text{-}TolCC(OH)CTol)][BF_4]{\cdot}CH_2Cl_2$

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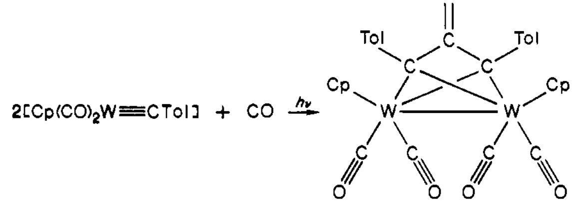
We recently reported the photochemical preparation of a binuclear complex **1** possessing the unusual μ-TolC–C(O)–CTol ligand, which was formed by the photoinduced coupling of two carbyne ligands with a metal carbonyl[1] (eq 1). A diplatinum



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