dissolved in toluene (10 mL) and the solvent was removed, $Y_2(OC_6H_3Me_2\text{-}2,6)_6(THF)_2$ (**3**)[17] was isolated as a white powder (441 mg, 82%) according to eq 2.

$$\mathrm{YCl_3 + 3NaOC_6H_3Me_2\text{-}2,6 \xrightarrow{THF} \underset{\mathbf{2}}{Y(OC_6H_3Me_2\text{-}2,6)_3(THF)_3}} \tag{1}$$

$$\mathrm{YCl_3 + 3NaOC_6H_3Me_2\text{-}2,6 \xrightarrow{THF} \xrightarrow{toluene} \underset{\mathbf{3}}{[Y(\mu\text{-}OC_6H_3Me_2\text{-}2,6)(OC_6H_3Me_2\text{-}2,6)_2(THF)]_2}} \tag{2}$$

The $^1H$ and $^{13}C$ NMR spectra of **2**[16] in THF-$d_8$ indicated that a single phenoxide ligand environment was present. This was confirmed by a preliminary single-crystal X-ray diffraction study,[18] which revealed the structure shown in Figure 1. The $^1H$ NMR spectrum of **3** in THF-$d_8$ was identical with that of **2**. However, in $C_6D_6$[17] two methyl resonances in a ratio of 2:1 were observed. White crystals of **3** were grown from toluene at −34 °C and were shown by single-crystal X-ray diffraction to have the bimetallic structure shown in Figure 2.[19] The solid-state structure is consistent with the $^1H$ NMR spectrum, assuming that the methyl groups of the bridging ligands resonate at lower field than those of the terminal ligands. *tert*-Butoxide ligands attached to yttrium exhibit this same trend in resonance shifts.[7,8]

Extraction of **3** with THF and removal of the solvent by rotary evaporation regenerated **2** (eq 3). Extraction of the regenerated sample of **2** with toluene quantitatively regenerated **3**. This cycle could be traversed repeatedly; i.e., loss of THF from **2** to form phenoxide-bridged **3** is facile and reversible.

$$\mathrm{\underset{\mathbf{3}}{[Y(\mu\text{-}OC_6H_3Me_2\text{-}2,6)(OC_6H_3Me_2\text{-}2,6)_2(THF)]_2} \underset{toluene}{\overset{THF}{\rightleftarrows}} \underset{\mathbf{2}}{Y(OC_6H_3Me_2\text{-}2,6)_3(THF)_3}} \tag{3}$$

The structure of **2** contains a distorted *fac*-octahedral arrangement of ligands around yttrium. Six has been the common coordination number observed in all structurally characterized yttrium alkoxides in the literature except for the sterically crowded three-coordinate **1**.[11] Unfortunately, the quality of the structure of **2** does not allow a detailed discussion of bond distances and angles.[18]

In **3**, the yttrium atoms are five-coordinate. The geometry is best described as distorted square pyramidal with O(3) occupying the axial position. The bridging ligands are situated symmetrically between the two yttrium atoms (Y(1)–O(1) and Y(1)–O(1′) are 2.277 (5) and 2.275 (6) Å, respectively). As is typical,[6-10,22] these bridging Y–O distances are longer than the terminal Y–O distances (Y(1)–O(2) = 2.075 (6) Å, Y(1)–O(3) = 2.046 (6) Å). The terminal Y–O distances are similar to the 2.00 (2)-Å Y–O distances in **1**[11] and are in the 1.97–2.08-Å range of terminal Y–O distances in *tert*-butoxide complexes.[7,8] The Y(1)–O(2)–C(9) angle of 162.6 (5)° is smaller than the 171(1)–175(1)° range of Y–O–C angles in **1**, whereas the Y(1)–O(3)–C(17) angle of 168.7 (6)° is closer to this range. The 2.348 (6)-Å Y–O(THF) distance is in the 2.33–2.42-Å range observed for such distances in yttrium *tert*-butoxide complexes.[7,8]

Complexes **2** and **3** and their facile interconversion demonstrate that the 2,6-dimethylphenoxide ligand can provide a versatile $[Y(OR)_3(\text{solvent})_a]_b$ unit with yttrium in which *a* and *b* are variable. The sterically unsaturated "$Y(OR)_3(THF)$" moiety formed in the **2** to **3** conversion may be quite useful in synthesis, and its utility in the formation of heteropolymetallic alkoxide complexes is under study.

**Acknowledgment.** For support of this research we thank the Division of Chemical Sciences of the Office of Basic Energy Sciences of the Department of Energy. Funds for the purchase of the Nicolet R3m/V diffractometer system were made available from the National Science Foundation under Grant CHE-85-14495.

**Supplementary Material Available:** Tables of crystal data, bond distances and angles, thermal parameters, and hydrogen atom coordinates and thermal parameters and a summary of the structure solution method (10 pages); a listing of structure factor amplitudes (13 pages). Ordering information is given on any current masthead page.

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[17] $^1H$ NMR ($C_6D_6$): δ 7.09 (d, $J_{HH}$ = 7.1 Hz, 6 H, $C_6\mathit{H}_3Me_2$-2,6); 6.79 (br m, 3 H, $C_6\mathit{H}_3Me_2$-2,6), 3.18 (s, 4 H, THF), 2.52 (s, 6 H, μ-$OC_6H_3\mathit{Me}_2$-2,6), 2.23 (s, 12 H, $OC_6H_3\mathit{Me}_2$-2,6), 0.66 (s, 4 H, THF). $^{13}C\{^1H\}$ NMR (THF-$d_8$): δ 159.66 (s, $\mathit{C}_6H_3Me_2$-2,6), 153.85 (s, $\mathit{C}_6H_3Me_2$-2,6), 128.30 (s, $\mathit{C}_6H_3Me_2$-2,6), 128.20 (d, $J_{CH}$ = 163 Hz, $\mathit{C}_6H_3Me_2$-2,6), 127.68 (d, $J_{CH}$ = 158 Hz, $\mathit{C}_6H_3Me_2$-2,6), 124.77 (s, $\mathit{C}_6H_3Me_2$-2,6), 120.37 (d, $J_{CH}$ = 156 Hz, $\mathit{C}_6H_3Me_2$-2,6), 116.73 (d, $J_{CH}$ = 157 Hz, $\mathit{C}_6H_3Me_2$-2,6), 69.73 (t, $J_{CH}$ = 155 Hz, THF), 23.75 (t, $J_{CH}$ = 133 Hz, THF), 17.26 (q, $J_{CH}$ = 125 Hz, $C_6H_3\mathit{Me}_2$-2,6). Anal. Calcd for $Y_2C_{56}H_{70}O_8$: Y, 17.0. Found: Y, 16.8. IR (KBr): 3000 w, 2940 w, 2900 s br, 1590 m, 1440 s br, 1270 s, 1235 m, 1195 m, 1090 m, 1010 w, 915 w, 860 s br, 755 s, 740 s, 700 m, 670 w $cm^{-1}$.

[18] **2** crystallized from THF/hexane at −34 °C in space group *P3c1* with unit cell parameters at 183 K of $a$ = 18.340 (5) Å, $c$ = 17.772 (5) Å, $V$ = 5177 (2) Å$^3$, and $D_{calcd}$ = 1.29 g cm$^{-3}$ for $Z$ = 6. The asymmetric unit consists of three independent molecules, each located on a crystallographic 3-fold rotation axis, Y(1) at ($^2/_3$, $^1/_3$, $z$), Y(2) at ($^1/_3$, $^2/_3$, $z$) and Y(3) at (0, 0, $z$). The poor quality of the data set prevented an accurate refinement of the model. Attempts to refine the structure by using the centrosymmetric space group *P*3*c*1 proved unsuccessful.

[19] Crystal data for $C_{56}H_{70}O_8Y_2\cdot 2(C_7H_8)$: The crystals belong to the monoclinic system with unit cell parameters at 183 K of $a$ = 10.8392 (15) Å, $b$ = 13.896 (2) Å, $c$ = 22.065 (3) Å, $\beta$ = 101.891 (11)°, and $V$ = 3252.1(8) Å$^3$. The space group is $P2_1/c$ with $Z$ = 2 dimers/unit cell and $D_{calcd}$ = 1.26 g cm$^{-3}$. Intensity data (4517 total; $2\theta_{max}$ = 45.0°) were collected on the Nicolet R3m/V diffractometer system using monochromatized Mo Kα radiation (λ = 0.710730 Å) via the $\theta$–$2\theta$ scan technique.[20] The structure was solved by direct methods and refined by full-matrix least-squares techniques[21] using those 2851 data with $|F_0| > 3.0\sigma(|F_0|)$. At convergence, $R_F$ = 7.8%, $R_{wF}$ = 7.7%, and GOF = 1.41 for 326 variables. A final difference-Fourier map yielded $\rho(\max)$ = 1.27 e Å$^{-3}$ at a distance of 0.75 Å from C(30).

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## Oxidation Photochemistry of Dimolybdenum(II) Diaryl Phosphate Promoted by Visible Light

Quadruply bonded metal–metal dimers are promising multielectron photoreagents. The lowest energy $^1(\delta\delta^*)$ excited state is long-lived[1] and is a two-electron source or sink in oxidation–reduction reactions.[2] Nevertheless the development of $\mathrm{M}\overset{4}{-}\mathrm{M}$ dimers for multielectron photochemical schemes has been hindered by the fact that $^1(\delta\delta^*)$ excited-state chemistry is an exceptional reaction pathway for these dimers. The photoredox chemistry of quadruply bonded metal–metal ($\mathrm{M}\overset{4}{-}\mathrm{M}$) dimers has generally been confined to photooxidation reactions derived from high-energy electronic excited states. Ultraviolet irradiation of acidic solutions containing $\mathrm{M}\overset{4}{-}\mathrm{M}$ dimers leads to one- or two-electron oxidation of the metal–metal core with the concomitant production of hydrogen.[3,4] Recent studies in our laboratories of dimolybdenum phosphates have shown that photochemical conversion of $Mo_2$-

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[1] (a) Miskowski, V. M.; Goldbeck, R. A.; Kliger, D. S.; Gray, H. B. *Inorg. Chem.* **1979**, *18*, 86–89. (b) Hopkins, M. D.; Gray, H. B. *J. Am. Chem. Soc.* **1984**, *106*, 2468–2469.

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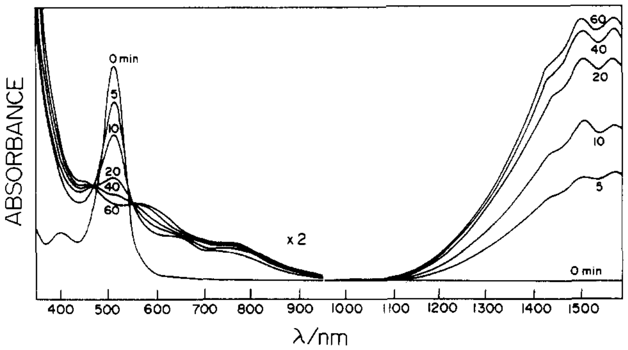

**Figure 1.** Electronic absorption spectral changes during visible irradiation ($\lambda_{exc} \geq 530$ nm) of $Mo_2(O_2P(OC_6H_5)_2)_4$ in 1,2-dichloroethane at 23 °C. The solution was subject to seven freeze–pump–thaw cycles prior to irradiation. The visible absorption scale is twice that of the near-infrared absorbance scale.

$(HPO_4)_4{}^{4-}$ to its two-electron oxidized congener, $Mo_2(HPO_4)_4{}^{2-}$, proceeds by sequential one-electron steps via the high-energy $\pi\pi^*$ excited state.[3] The photochemical reaction mechanism of this dimolybdenum phosphate system appears to be completely general and conforms well with observations that the lowest energy $\delta\delta^*$ excited state is not responsible for the photooxidation chemistry of $M\overset{4}{-}M$ complexes in aqueous solutions. Our observation that the lifetime of the $^1(\delta\delta^*)$ excited state is significantly attenuated in aqueous solutions by efficient proton quenching reactions suggested to us that the chemistry of the $^1(\delta\delta^*)$ excited state, which should be effective in promoting multielectron photoprocesses driven by visible light, could be exploited by undertaking studies of dimolybdenum phosphates in aprotic environments. Thus we have investigated the photochemical activity of the newly synthesized $Mo_2(O_2P(OC_6H_5)_2)_4$ quadruply bonded metal–metal complex with halocarbon substrates.[5] We now report the two-electron photoreduction of 1,2-dichloroethane (DCE) promoted by the $^1(\delta\delta^*)$ excited state of $Mo_2(O_2P(OC_6H_5)_2)_4$.

The electronic absorption spectrum of $Mo_2(O_2P(OC_6H_5)_2)_4$[6] is analogous to that of the $Mo_2(HPO_4)_4{}^{4-}$ ion and is dominated by the prominent $\delta^2 \rightarrow \delta\delta^*$ band ($\lambda_{abs,max} = 515$ nm, $\epsilon = 156$ M$^{-1}$ cm$^{-1}$). However, in contrast to $Mo_2(HPO_4)^{4-}$, solids and solutions of the dimolybdenum(II) diphenyl phosphate complex exhibit weak luminescence ($\lambda_{em,max} = 560$ nm, $\Phi_e \leq 10^{-4}$) upon excitation into the $\delta^2 \rightarrow \delta\delta^*$ absorption band. The 68-ns lifetime of the $^1(\delta\delta^*)$ excited state, measured from transient absorption kinetics,[9] is comparable to the longest $^1(\delta\delta^*)$ lifetimes of $M\overset{4}{-}M$ complexes and is sufficiently long-lived to permit reaction of the $^1(\delta\delta^*)$ excited state with substrate molecules.[10] This is the case. Whereas halocarbon solutions of dimolybdenum(II) diphenyl phosphate are stable indefinitely in the absence of light, reaction of these solutions is immediate upon visible irradiation. Figure 1 displays the spectral changes for photolysis ($\lambda_{exc} \geq 530$ nm) of dimolybdenum(II) diphenyl phosphate in DCE. The decrease of the $\delta^2 \rightarrow \delta\delta^*$ absorption band is accompanied by increased absorption in the near-infrared spectral region. Isosbestic points are observed in the visible spectral region during the initial stages of photolysis; however, the establishment of equilibrium between axially coordinated mixed-valence $Mo^{II}Mo^{III}Cl$ and uncoordinated $Mo^{II}Mo^{III}$ leads to degradation of the isosbestic points during the latter stages of photolysis.

The appearance of the vibrationally-structured absorption band in the near-infrared region is consistent with the formation of the mixed-valence dimolybdenum(II,III) diphenyl phosphate dimer. Fast atom bombardment mass spectra[11] of the isolated photoproduct display a parent ion peak at *m/z* 1225, which is consistent with the photoproduction of $Mo_2(O_2P(OC_6H_5)_2)_4Cl$. Indeed, the final photolysis spectrum is nearly identical with that of $Mo_2(O_2P(OC_6H_5)_2)_4Cl$ ($\lambda_{abs,max} = 1494$ nm, $\epsilon = 362$ M$^{-1}$ cm$^{-1}$).[12] Toepler pumping of the photochemical reaction mixture gave 0.51 ± 0.03 equiv of ethylene (as determined by mass spectrometry and gas chromatography). Thus the overall photoreaction is consistent with

$$2Mo_2(O_2P(OC_6H_5)_2)_4 + ClCH_2CH_2Cl \xrightarrow{h\nu \geq 530\ \mathrm{nm}} 2Mo_2(O_2P(OC_6H_5)_2)_4Cl + CH_2CH_2 \quad (1)$$

The maximum quantum yield for the photoreaction, $\Phi_p = 0.040 \pm 0.004$, is observed for 510-nm irradiation, which is consistent with the photoreaction originating directly from the $^1(\delta\delta^*)$ excited state. Cyclic voltammograms of dimolybdenum(II) diphenyl phosphate in $CH_2Cl_2$ exhibit two reversible one-electron-oxidation processes at +0.07 and +1.00 V vs SCE corresponding to the dimolybdenum(II,II)/dimolybdenum(II,III) and dimolybdenum(II,III)/dimolybdenum(III,III) diphenyl phosphate couples, respectively. Because the reduction potential of DCE is ca. −2.13 V vs SCE,[13] the $^1(\delta\delta^*)$ excited state is sufficiently energetic to directly reduce DCE to form $Cl^-$ and the radical fragment $^\bullet CH_2CH_2Cl$. Indeed, the photochemical reduction of halocarbons have been demonstrated to proceed via one-electron reduction of the halocarbon to form $RX^{\bullet -}$ followed by subsequent rapid unimolecular decomposition to $R^\bullet$ and $X^-$.[14-16]

The results described above are consistent with the photochemical reaction mechanism shown in Figure 2. Electron transfer from the $^1(\delta\delta^*)$ excited state of dimolybdenum(II) diphenyl phosphate yields the mixed-valence $Mo^{II}Mo^{III}Cl$ complex and $^\bullet CH_2CH_2Cl$. Subsequent reaction of the radical with another equivalent of the $Mo^{II}{}_2$ starting complex directly yields the observed photoproducts. In this case, the photogenerated radical escapes the solvent cage of the primary photoproducts. Alternatively, if the organic radical intermediate is scavenged by the

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(5) A preliminary report of the synthesis and photochemistry of the dimolybdenum(II) diphenyl phosphate system was presented at the 194th National Meeting of the American Chemical Society, New Orleans, LA, Aug 30–Sept 4, 1987.

(6) The dimolybdenum(II) diphenyl phosphate was prepared by addition of 0.5 g of $(NH_4)_5Mo_2Cl_9{\cdot}H_2O$[7] (0.81 mmol) to a 60-mL deaerated MeOH solution containing 3.25 g of the anion $(C_6H_5O)_2PO_2{}^-$, which was generated upon stirring $(C_6H_5O)_2P(O)OH$ with a molar equivalence of NaOMe. The solution was heated to reflux for 3 h. The pink precipitate, formed during reflux, was recrystallized from tetrahydrofuran/cyclohexane solution. Yield: 76%. Anal. Calcd (found) for $Mo_2(O_2P(OC_6H_5)_2)_4{\cdot}2THF$: Mo, 14.4 (14.87); P, 9.29 (9.17); C, 50.46 (49.93); H, 4.24 (4.15). During the review of this manuscript, the synthesis of the dimolybdenum(II) diphenyl phosphate was reported by a similar method.[8] The analytical and spectroscopic results of the compound prepared by these authors are consistent with the compound reported herein.

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(11) FABMS was performed on a JEOL HX 110 double-focusing mass spectrometer housed in the National Institutes of Health/Michigan State University Mass Spectrometry Facility; samples were dissolved in 2-(octyloxy)nitrobenzene matrices.

(12) Addition of 0.5 equiv of dichloroiodobenzene to a dichloromethane solution of $Mo_2(O_2P(OC_6H_5)_2)_4$ leads to the quantitative production of $Mo_2(O_2P(OC_6H_5)_2)_4Cl$. The product was identified by FABMS and electronic absorption spectroscopy.

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(16) Whereas the reduction of alkyl halides by transition-metal donors can occur by an outer-sphere electron transfer,[17,18] recent studies have shown that $S_N2$ electron transfer is especially important for coordinatively unsaturated transition-metal reductants.[19,20] In view of the vacant axial coordination sites of the metal–metal core, the latter reaction pathway should play a significant role in the photoreduction of alkyl halides by electronically excited $Mo^{II}{}_2$ complexes.

(17) Bakac, A.; Espenson, J. H. *J. Am. Chem. Soc.* **1986**, *108*, 713–719.

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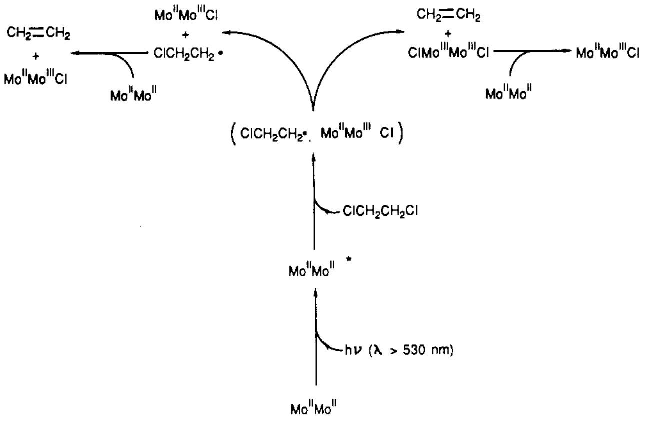

**Figure 2.** Proposed mechanism for the photochemical reduction of 1,2-dichloroethane by $Mo_2(O_2P(OC_6H_5)_2)_4$.

mixed-valence complex within the solvent cage, then the fully oxidized $Mo^{III}{}_2\,Cl_2$ complex is produced. The energetics of the dimolybdenum(II,II)/dimolybdenum(II,III) and dimolybdenum(II,III)/dimolybdenum(III,III) couples are such that comproportionation of $Mo^{III}{}_2Cl_2$ and unreacted dimolybdenum(II) diphenyl phosphate will yield the mixed-valence species. Thus, the stoichiometry of the photoreaction is independent of whether the radical reacts within or external to the solvent cage of the primary photoproducts.

Multielectron photochemistry has been observed from the lowest energy excited state of $M\overset{4}{-}M$ complexes. The dimolybdenum(II) diphenyl phosphate complex effectively promotes the two-electron reduction of halocarbon substrates upon excitation into the $\delta\delta^*$ excited state with the absorption of a single visible photon. Reaction from low-energy excited states of $M\overset{4}{-}M$ complexes has not been heretofore achieved, and hence the photoreduction of alkyl halides by $Mo_2(O_2P(OC_6H_5)_2)_4$ from its $\delta\delta^*$ excited state is a unique reaction pathway of $M\overset{4}{-}M$ complexes. The overall multielectron photochemistry is achieved by coupling the one-electron-oxidation-reduction chemistry of independent metal-metal cores. Efforts are currently underway to effect the multielectron transformation at a discrete electronically excited $M\overset{4}{-}M$ center.

**Acknowledgment.** We express our gratitude to Dr. Jay Winkler for providing us with access to the picosecond laser facilities at Brookhaven National Laboratory. Financial support of this work was provided by the National Science Foundation (Grant CHE-8705871). D.G.N. also gratefully acknowledges a Presidential Young Investigator Award administered by the National Science Foundation.

**Registry No.** DCE, 107-06-2; $Mo_2(O_2P(OC_6H_5)_2)_4$, 118494-24-9; $Mo_2(O_2P(OC_6H_5)_2)_4Cl$, 122967-43-5; $CH_2{=}CH_2$, 74-85-1; $(NH_4)_5Mo_2Cl_9$, 61583-95-7; $(C_6H_5O)_2PO_2^-$, 48168-03-2.

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## Facile Regioselective Ligand Substitution for the In-Plane Bridging Acetates in Octakis(μ-acetato-*O*,*O'*)tetraplatinum(II)

Platinum(II) with $d^8$ electronic configuration has strong tendency to take a square-planar structure. Octakis(μ-acetato)tetraplatinum(II) (**1**), which was reported over a decade ago,[1,2] is unique in that each platinum(II) ion has an octahedral geometry and metal-metal direct bonds (metal-metal distance of 2.50 Å) exist. A complex with such unusual structural characteristics certainly deserves further investigations such as its electronic structure, route of formation, and various other properties, in view of recent extensive progress in studies of the chemistry of cluster complexes.

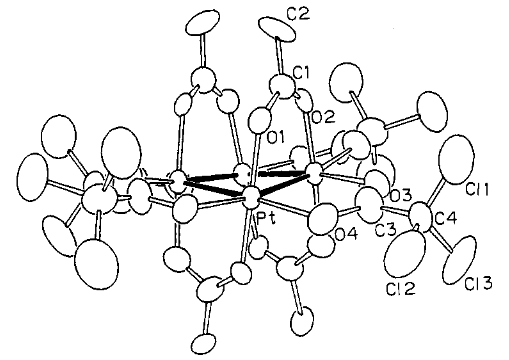

**Figure 1.** Structure of $Pt_4(\mu\text{-}CCl_3COO)_4(\mu\text{-}CH_3COO)_4$ showing the 50% probability thermal ellipsoids. Selected interatomic distances (Å) and angles (deg) are as follows: Pt-Pt = 2.49 (1); Pt-O($CH_3COO$) = 2.01 (3), 2.00 (3); Pt-O($CCl_3COO$) = 2.14 (5), 2.20 (4); Pt-Pt-Pt = 89.6 (1); Pt-Pt-O($CCl_3COO$) = 85 (1), 84 (1); Pt-Pt-O($CH_3COO$) = 82 (2), 84 (1), 94 (1), 97 (1).

While investigating reactivity of this cluster in solution, we found that only one of the two chemically different acetate groups is easily replaced by free carboxylate ions in solution. This paper reports the crystal structure of the new complex prepared by this facile regioselective substitution and kinetics of the acetate-exchange reaction as well as the theoretical studies on this unusual reactivity.

The substitution reaction is unexpectedly fast for the bridging carboxylates and is completed within a minute.[3] The new complex, $Pt_4(\mu\text{-}CH_3COO)_4(\mu\text{-}CCl_3COO)_4$ (**2**), was precipitated immediately from the solution of **1** in $CHCl_3$ by adding $CCl_3COOH$.[4] Figure 1 shows the ORTEP drawing of the new complex **2**,[5,6] which clearly shows that the in-plane acetates are replaced. Except for the difference in the in-plane ligands, the main structural features are very similar to those of the parent complex **1**. The Pt-Pt distance (2.49 Å) in **2** is similar to that in **1**. Somewhat longer in-plane Pt-O($CCl_3COO$) distances than out-of-plane Pt-O($CH_3COO$) distances are observed.

Kinetics of reaction 1 in $CDCl_3$ for the in-plane acetate exchange was studied by the $^1H$ NMR saturation transfer method.[7]

$$Pt_4(CH_3COO)_8 + {}^*CH_3COOH \rightarrow Pt_4(CH_3COO)_4({}^*CH_3COO)_4 + CH_3COOH \quad (1)$$

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(1) Carrondo, M. A. A. F. de C. T.; Skapski, A. C. *J. Chem. Soc., Chem. Commun.* **1976**, 410-411.

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(3) An example of the fast bridging carboxylate exchange: Teramoto, K.; Sasaki, Y.; Migita, K.; Iwaizumi, M.; Saito, K. *Bull. Chem. Soc. Jpn.* **1979**, *52*, 446-452.

(4) Other carboxylic acids such as $C_6H_5COOH$ and $CF_3COOH$ also give similar complexes, e.g. $Pt_4(\mu\text{-}CH_3COO)_4(\mu\text{-}RCOO)_4$, on reacting with **1**.

(5) The complex **2** was recrystallized from $CH_2Cl_2$. Anal. Calcd for $Pt_4(\mu\text{-}CH_3COO)_4(\mu\text{-}CCl_3COO)_4\cdot 2CH_2Cl_2$: C, 11.78; H, 0.89; Cl, 30.90. Found: C, 11.67; H, 0.90; Cl, 29.68.

(6) X-ray analysis: The compound $Pt_4(\mu\text{-}CH_3COO)_4(\mu\text{-}CCl_3COO)_4\cdot 2CH_2Cl_2$ crystallizes in the tetragonal space group $I\bar{4}2d$, with $a$ = 13.909 (2) Å, $c$ = 23.372 (4) Å, $V$ = 4522 (1) Å$^3$, and $Z$ = 4. With the use of 626 unique reflections ($F_o > 3\sigma(F_o)$) collected at room temperature with Mo Kα (λ = 0.709 26 Å) radiation up to $2\theta$ = 50° on a Rigaku AFC-5R diffractometer, the structure was solved by the heavy-atom method and refined by block-diagonal least-squares techniques with anisotropic temperature factors for non-hydrogen atoms. The current $R$ value is 0.053. Atomic positional and thermal parameters are provided as supplementary material.

(7) (a) Forsen, S.; Hoffman, R. A. *J. Chem. Phys.* **1963**, *39*, 2892-2901. (b) Recent application of this method: Cho, H.; Iwashita, T.; Ueda, M.; Mizuno, A.; Mizukawa, K.; Hamaguchi, M. *J. Am. Chem. Soc.* **1988**, *110*, 4832-4834 and references cited therein.

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