perturbations occur early on in the interaction. Thus, geometry optimizations indicate that one C–O bond is nearly entirely formed before the second C–O bond begins to form. Since the reaction is a nucleophilic attack by the olefin on the oxo, it is not surprising that a positive charge builds up on the unbound C. In order to take advantage of the greater overlap between the O p$\pi$ AO's in the frontier orbital of the Ru–oxo system and the ethylene $\pi$ system, the olefin cants. This canting motion agrees with the observation that *cis*-stilbene is epoxidized at a slower rate than the trans analogue. Eventually a symmetrically bound epoxide minimum on the potential energy surface is attained, as one would predict from simple electrostatic considerations, with a concomitant two-electron reduction of the metal. The 5% isomerized product formed in the epoxidation of *cis*-stilbene is consistent with the fact that some C–C bond rotation can occur before the formation of the second C–O bond. The relief of steric strain in *cis*-stilbene is obviously sufficient enough to provide a driving force for this rotation. The preference for bound epoxides over oxometallacycles found by INDO/1 is supported by partial charge, frontier MO, and simple electronic structural considerations.

**Note Added in Proof.** Since the acceptance of this paper for publication, an independent experimental study (Ostovic, D.; Bruice, T. C. *J. Am. Chem. Soc.* **1989**, *111*, 6511) on the related iron–porphyrin systems has presented conclusions similar to those given here.

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# Cation Effect on the Quenching of the Photoexcited State of Tetrakis($\mu$-pyrophosphito-$P,P'$)diplatinate(II) by Octacyanomolybdate(IV) in Aqueous Solution

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The photoquenching reaction of $[Pt_2(pop)_4]^{4-}$ ($pop^{2-}$ = pyrophosphite(2–)) with $[Mo(CN)_8]^{4-}$ was studied in 0.01 M HCl or $HClO_4$ in the presence of various added electrolytes (0.49 M) (LiCl, NaCl, KCl, CsCl, $NH_4Cl$, $(C_2H_5)_4NCl$, and $NaClO_4$) at 25 °C. The quenching rate constants ($k_q$) differ by nearly 1 order of magnitude from $1.17 \times 10^7$ (LiCl) to $9.90 \times 10^7$ $M^{-1}$ $s^{-1}$ (CsCl). The phosphorescence lifetime ($\tau_0$) in the absence of the quencher is practically constant (8.3–8.6 $\mu$s) regardless of the type of electrolyte added. $k_q$ shows a linear correlation with the reciprocal of the cation Stokes radii except for the case of $(C_2H_5)_4N^+$. The cation effect was interpreted by considering a precursor ion triplet among two negatively charged reactants and a foreign cation. The role of the cation is to reduce the Coulombic barrier of the two reactants in order to form an encounter complex.

In a recent work,[1,2] we showed that the rate of emission quenching of $[(Mo_6Cl_8)Cl_6]^{2-}$ with $[IrCl_6]^{2-}$ in acetonitrile is significantly affected by the nature of added cations ($M^+$). At $[M^+]$ = 0.1 M, the second-order rate constant ($M^{-1}$ $s^{-1}$) ranges from $2.7 \times 10^7$ for $M^+$ = $n$-$Bu_4N^+$ to $1.0 \times 10^9$ for $Na^+$ at 25.0 °C.[1,2] This fact was interpreted by considering that cations assist the encounter complex formation between the two negatively charged reactants. Namely, the ion triplet involving a foreign cation is considered as a precursor to the reaction. A similar drastic cation effect was not observed for the same reaction in aqueous hydrochloric acid media.[2] It is unlikely that the different behavior in the two solvents is directly related to the solvent properties, although water has a much higher dielectric constant and is less favorable for ion-pair or ion-triplet formation. It may be more important that the quenching rate is much faster in aqueous hydrochloric acid and in approaching the diffusion-controlled limit.[2] Significant cation effects have been reported for some thermal electron-transfer reactions in aqueous media.[3-5] A similar cation effect could be observed for a photoquenching reaction in aqueous media, if an appropriate reaction system were chosen.

We have used $[Pt_2(pop)_4]^{4-}$ ($pop^{2-}$ = pyrophosphite(2–))[6,7] as a sensitizer and $[Mo(CN)_8]^{4-}$ as a quencher for the study of the cation effect in aqueous solution. The rate of this quenching reaction is known to be much smaller than the diffusion-controlled limit.[8] With such highly negatively charged reactants, a significant influence of cations is expected on the quenching rate, so far as the reaction proceeds through encounter complex formation and not through a long-distance mechanism.

## Experimental Section

**Potassium Tetrakis($\mu$-pyrophosphito-$P,P'$)diplatinate(II) Dihydrate, $K_4[Pt_2(pop)_4]\cdot 2H_2O$.** This compound was prepared by the reported me-

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thod[6,9] and recrystallized from water upon slow evaporation in a desiccator containing $P_2O_5$. The absorption spectrum and emission lifetime (8.7 μs) in water at 25 °C are in reasonable agreement with the reported ones.[10,11]

**Other Materials.** $K_4[Mo(CN)_8]\cdot 2H_2O$ was prepared as described in the literature[12] and recrystallized three times from water. Hydrochloric and perchloric acids for microanalytical use and a special grade reagent of CsCl were used as received. Special grade reagents of NaCl, LiCl, KCl, $NH_4Cl$, $NaClO_4$, and $(C_2H_5)_4NCl$ were recrystallized from water.

**Measurements.** A solution of ca. $1 \times 10^{-5}$ M $K_4[Pt_2(pop)_4]$, (0–5.0) $\times 10^{-3}$ M $K_4[Mo(CN)_8]$, 0.01 M HCl or $HClO_4$, and a 0.49 M concentration of the appropriate salt was degassed under vacuum with at least three freezing cycles. Light (337.1 nm) from an $N_2$ laser with ca. 5-ns pulse width (JS-1000L of NDC) was used for the excitation. Luminescence decay was monitored at 520 nm and analyzed as described elsewhere.[2] The flash photolysis was carried out by the use of a Molectron UV 400 $N_2$ laser (10-ns pulse width, pulse energy 2.6 mJ) for the excitation and an FX 200U flash lamp for the analysis. Absorption spectra were recorded with a Hitachi 330 automatic recording spectrophotometer.

## Results and Discussion

**Preliminary Observations.** Six chloride salts, LiCl, NaCl, KCl, CsCl, $NH_4Cl$, and $(C_2H_5)_4NCl$, were chosen for the investigation of the cation effect. $NaClO_4$ was used to determine the effect of anionic species. The absorption spectra of each of the reactants in 0.01 M HCl did not show appreciable change on addition of up to 0.49 M MCl ($M^+ = Li^+, Na^+, K^+$, and $Cs^+$). The spectrum of $[Pt_2(pop)_4]^{4-}$ showed appreciable change, however, on addition of $(C_2H_5)_4NCl$ (intensity of the peak at 368 nm ($\epsilon = 35\,300$ $M^{-1}$ $cm^{-1}$)[9,13] decreased by ca. 10% at $[(C_2H_5)_4N^+] = 0.49$ M). Since other physical properties such as the phosphorescence lifetime is not affected by the addition of $(C_2H_5)_4NCl$, the change should be due to simple ion-pair formation between $[Pt_2(pop)_4]^{4-}$ and $(C_2H_5)_4N^+$. A smaller change was observed on addition of $NH_4Cl$. A significant spectral change was also observed for the solution of $[Mo(CN)_8]^{4-}$ on addition of $(C_2H_5)_4NCl$. Both $[Mo(CN)_8]^{4-}$ and $[Pt_2(pop)_4]^{4-}$ can interact with solvent water molecules through hydrogen bonds. Such bonds may be destroyed by adding hydrophobic alkylammonium ions to cause the change in the spectra. Alkali-metal ions would not work in such a way.

Absorption spectra of the solutions for the quenching experiments did not show appreciable change on standing except for the following case: In the presence of both $[Mo(CN)_8]^{4-}$ and $(C_2H_5)_4NCl$ (0.49 M), the absorption spectrum of $[Pt_2(pop)_4]^{4-}$ slowly changed under the laboratory light. A new peak appeared at ca. 400 nm. The emission lifetime decreased considerably during the change (after 60 min, the lifetime decreased to one-third). Because of this change the photoquenching rate was measured quickly after the preparation of the solution.

**Mechanism of the Quenching.** From the reported redox potentials of $[Mo(CN)_8]^{4-}/[Mo(CN)_8]^{3-}$ (−0.77 V vs NHE)[8,14] and photoexcited $[Pt_2(pop)_4]^{4-}/[Pt_2(pop)_4]^{5-}$ (1.34 V),[15] it is possible that electron-transfer quenching takes place. In order to determine whether the electron-transfer mechanism operated for the present quenching reaction, flash photolysis experiments were carried out at $[Pt_2(pop)_4{}^{4-}] = 1 \times 10^{-4}$ M, $[Mo(CN)_8{}^{4-}] = 5 \times 10^{-3}$ M, and $I = 0.5$ M (0.01 M $HClO_4$, 0.49 M $NaClO_4$). No absorbance change was observed when the experiment was monitored at 415 nm. At this wavelength, the one-electron-reduced species of the sensitizer, $[Pt_2(pop)_4]^{5-}$, has been reported to have significant absorption ($\lambda_{max}$ 420 nm ($\epsilon$ ca. 13 000 $M^{-1}$ $cm^{-1}$)).[16] The oxidized form of the quencher, $[Mo(CN)_8]^{3-}$, has relatively weak absorption ($\epsilon$ ca. 1000 $M^{-1}$ $cm^{-1}$) in this region.[17] Under a similar experimental setup, appearance of the photoexcited state of $[Pt_2(pop)_4]^{4-}$ was clearly observed at 465 nm, where it has significant absorption ($\epsilon$ $10^4$ $M^{-1}$ $cm^{-1}$).[11] Subsequently the absorption decreased at a rate corresponding to the quenching reaction. Since the products of the electron-transfer quenching were not detected, the quenching reaction must proceed mainly through an energy-transfer mechanism. Participation of the electron-transfer pathway, if any, should be less than 10% of the total quenching reaction.

**Table I.** Quenching Rate Constants ($k_q/M^{-1}$ $s^{-1}$) at 25 °C for Photoexcited $[Pt_2(pop)_4]^{4-\,a}$ with $[Mo(CN)_8]^{4-}$ in 0.01 M HCl in the Presence of 0.49 M Electrolyte.

| electrolyte | $\tau_0/\mu s^b$ | $10^{-7}k_q/M^{-1}$ $s^{-1}$ |
|---|---|---|
| none | 8.4 ± 0.1 | |
| LiCl | 8.5 ± 0.3 | 1.17 ± 0.16 |
| NaCl | 8.6 ± 0.2 | 2.00 ± 0.24 |
| $NaClO_4$ | 8.5 ± 0.1 | 1.86 ± 0.27 |
| | 8.5 ± 0.2 | 1.10 ± 0.20[c] |
| KCl | 8.6 ± 0.1 | 4.38 ± 0.22 |
| $NH_4Cl$ | 8.3 ± 0.2 | 3.83 ± 0.49 |
| CsCl | 8.5 ± 0.1 | 9.90 ± 0.71 |
| $(C_2H_5)_4NCl$ | 8.6 ± 0.1 | 5.58 ± 0.39 |

[a] $pop^{2-}$ = pyrophosphite(2−). [b] Phosphoresence lifetime without the quencher. Average of up to five independent measurements. [c] 0.09 M $NaClO_4$.

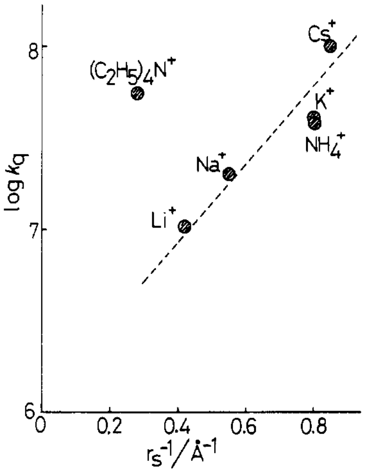

**Figure 1.** Plot of log $k_q$ against the reciprocal of the Stokes radii of $M^+$ for the photoquenching reaction of $[Pt_2(pop)_4]^{4-}$ with $[Mo(CN)_8]^{4-}$ in 0.01 M HCl in the presence of 0.49 M MCl ($M^+$ = alkali-metal ion) ($pop^{2-}$ = pyrophosphite(2−)).

**Effect of Cationic Species on Quenching with $[Mo(CN)_8]^{4-}$.** The phosphorescence lifetime ($\tau_0$) of $K_4[Pt_2(pop)_4]$ without the quencher was practically constant in the presence of the added salts (Table I). Thus the salts do not act as quenchers. The quenching reactions with $[Mo(CN)_8]^{4-}$ in the presence of various electrolytes were studied at a fixed ionic strength, 0.5 M. Stern–Volmer plots, $[Mo(CN)_8{}^{4-}]$ vs $(1/\tau - 1/\tau_0)$, were reasonably linear. From the slopes of these plots the quenching rate constant $k_q$ was obtained (Table I). The $k_q$ values vary over 1 order of magnitude. The chloride and perchlorate anions did not significantly influence the $k_q$ value. The $k_q$ values at ionic strengths 0.1 and 0.5 M ($NaClO_4$) (Table I) show the expected trend of ionic strength dependence. For the photoquenching reaction of $[(Mo_6Cl_8)Cl_6]^{2-}$ with $[IrCl_6]^{2-}$ in $CH_3CN$, $k_q$ shows linear dependence on the square root of ionic strength ($(C_2H_5)_4NClO_4$).[2]

For thermal electron-transfer reactions where a significant cation effect was observed, linear relationships between the rate constant and either the polarizability or the hydrated radius (Stokes radius) of alkali-metal ions were noted.[3-5] The effect was interpreted by considering a kind of ion triplet among two neg-

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atively charged reactants and a foreign cation; namely, the role of the cation is to reduce the Coulombic barrier of the two reactants in order to form an encounter complex.[3-5] Alternatively, the correlation with the polarizability was taken as evidence for the electron-mediating role of the cation between reductant and oxidant.[3] Figure 1 shows the plot of the quenching rate constant of the present reaction against the Stokes radii of the cations. Linear correlation was observed except for the case of the $(C_2H_5)_4N^+$ ion. The present quenching rate constant displays good linear correlation with the thermal electron-self-exchange rate constants of $[Fe(CN)_6]^{3-/4-}$ [18] and $MnO_4^{-/2-}$ [5] and the oxidation rate of $[Mo(CN)_8]^{4-}$ with $S_2O_8^{2-}$ [4] for various alkali-metal ions, which indicates that alkali-metal ions play a similar role for both the photoquenching and the thermal electron-transfer reactions over the wide range of rate constants. The exceptional behavior of $(C_2H_5)_4N^+$ in the plot of Figure 1 may be related to the observation of the spectral change of the reactants in the presence of the cation (vide supra).

In conclusion, the cation effect seems to provide strong evidence that the photoquenching reaction proceeds through an encounter complex that is an ion triplet containing the foreign cation, which reduces the Coulombic barrier. A long-distance electron- or energy-transfer mechanism is clearly ruled out. The present results indicate that any quantitative treatment of photoquenching rate constants should take electrolyte effect into consideration, particularly for reactants with the same charge sign.

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# Mononuclear, Dinuclear, and Trinuclear Ruthenium(II) Complexes of a Tris(bipyridine) Bridging Ligand: Syntheses, Absorption Spectra, Redox Potentials, and Photophysical Properties

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The tris(bipyridine) ligand **1**, 1,3,5-tris[(((5-(ethoxycarbonyl)-2,2′-bipyridyl-5′-yl)carbonyl)benzylamino)methyl]benzene, previously used to prepare a hemicaged Ru(II) complex, $\mathbf{1}[Ru]^{2+}$, has now been employed as a ligand capable of coordinating one, two, or three $Ru(bpy)_2^{2+}$ units. The three new complexes $\mathbf{1}[Ru(bpy)_2]^{2+}$, $\mathbf{1}[Ru(bpy)_2]_2^{4+}$, and $\mathbf{1}[Ru(bpy)_2]_3^{6+}$ have been synthesized as $PF_6^-$ salts, and their spectroscopic and electrochemical properties have been investigated. The absorption spectra of the complexes exhibit bpy and **1** ligand-centered bands in the UV region and Ru → bpy and Ru → **1** charge-transfer bands in the visible region, with the latter being at lower energy. In acetonitrile solution, all the complexes exhibit a reversible oxidation wave, centered on the metal, at +1.36 V and a reversible reduction wave, centered on the coordinated bpy-type arm of **1**, at −0.91 V vs SCE. Other reduction waves are present at more negative potentials. All the waves are monoelectronic, dielectronic, and trielectronic for the mononuclear, dinuclear, and trinuclear complexes, respectively. The luminescence properties are the same for the three complexes, with a band at 654 nm ($\tau \sim 1.4$ μs) in a propionitrile–butyronitrile rigid matrix at 77 K and a band at 700 nm ($\tau = 60$ ns) in a fluid propionitrile–butyronitrile solution at room temperature, indicating that (i) the lowest MLCT excited state involves the bpy-type coordinated arm of **1** in each complex and (ii) there is no strong interaction between the metal-containing chromophoric units. However, the fluorescence of the bpy-type arms of **1**, observable in the free ligand **1** at 77 K, is no longer present in $\mathbf{1}[Ru(bpy)_2]^{2+}$, indicating the occurrence of an intramolecular quenching. Excitation spectra performed on $\mathbf{1}[Ru(bpy)_2]^{2+}$ confirm that there is an efficient mechanism for intramolecular energy transfer from the two noncoordinated arms of **1** and the metal-containing chromophoric unit.

## Introduction

Mononuclear Ru(II)–polypyridine complexes are highly luminescent species and powerful reactants for light-induced and light-generating electron-transfer processes.[2] For a variety of practical applications, however, *multicenter* luminescent and redox systems are expected to be more useful.[3] The synthesis and characterization of polynuclear coordination compounds having desired photophysical and electrochemical properties is currently an important research field, and a great number of papers have recently appeared on this subject.[4-23]

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