Half-field lines were further evidence for binuclearity. According to Bleany and Bowers,[22] a dimeric $S = {}^1/_2$ system with axial symmetry has four allowed transitions in the solid state, two for $g_\parallel$ separated by $2D$ and two for $g_\perp$ separated by $3E + D$. Thus, the zero-field splitting parameters D and E are 0.0299 ± 0.0009 cm^{-1} and 0.0004 ± 0.0003 cm^{-1} respectively. The internuclear Cu–Cu distance was estimated to be 4.85 Å from the equation of Chasteen and Belford.[23]

$$R = (0.650g_\parallel{}^2/D)^{1/3} \qquad (9)$$

This distance is consistent with a Na-bridged structure such as that illustrated in Figure 5. The tetrahydrofurfuroxide **15** also exhibited a seven-line hyperfine pattern in frozen THF solution ($3E + D = 0.0363$ cm^{-1}) indicating a structure similar to **13**. However, the copper centers must be further apart at room temperature since the spectra displayed a poorly resolved four-line pattern despite solution molecular weight measurements indicating a dimer.

Benzene solutions of $M[Cu(OR)_3]_n$ complexes **11**, **12**, and **17** had broad ESR lines with no resolution of $g_\perp$, $g_\parallel$, or hyperfine splitting. Broad lines could be caused by a number of things: molecular asymmetry, small unresolved hyperfine couplings, spin–spin relaxation, and the presence of multiple chemical species. All may be acting here. Both g values and a poorly resolved $A_\parallel$ (quartet) were identifiable for the frozen THF solutions of **11** and **12**; these peaks were assigned to a mononuclear THF adduct. Frozen solutions of **11** also had an extremely broad, barely discernible half-field resonance around $g = 4.5$ and a broad multiplet (see Figure 4c) that were attributable to dimers and/or higher oligomers. Frozen THF solutions of **17** had complex ESR spectra, which were not assigned (Figure 4d).

Acknowledgment. The contributions of Alan Berry for his helpful discussions, Dale Pace for his assistance with ESR spectroscopy, and Ramanathan Panayappan and Angela Ervin for ICP analyses are appreciated. We thank the ONR for financial support.

Supplementary Material Available: Tables of crystal data, anisotropic displacement coefficients, and complete bond lengths and angles for $Na_2Cu[HFIP]_4$, the log/log and van't Hoff plots for **11**, and tables of mass spectral data for **1–8**, **14**, **16**, and **17** (12 pages); a table of observed and calculated structure factors (6 pages). Ordering information is given on any current masthead page.

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[22] Bleany, B.; Bowers, K. D. *Proc. R. Soc. London* **1952**, *A214*, 451.

[23] Chasteen, D. R.; Belford, R. L. *Inorg. Chem.* **1970**, *9*, 169–175.

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Role of Solvent in Photoredox Reactions of Cobalt(III) Am(m)ine Complexes

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Charge-transfer (CT) excited states of transition-metal complexes arise from the radial movement of electron density between metal and ligands (or solvent), and this redistribution of charge should be inherently sensitive to changes in solvent medium. The commonly observed solvatochromism of CT absorption and luminescence bands, for example, results from dielectric (e.g. dipole–dipole, dipole–induced dipole, dispersion) and/or hydrogen-bonding interactions between the complex and solvent molecules.[1] Solvent viscosity also can influence CT luminescence through its effect upon the rate of solvent reorientation about the excited complex.[2]

A fundamental consideration in discussions of CT photochemical behavior is the role played by the environment. Does solvent participate chemically in the reactions of CT excited states, or is its influence confined to medium effects of the type noted above? This issue was addressed for ligand-to-metal charge-transfer (LMCT) states several years ago by Endicott et al. in their comprehensive studies of the $Co(NH_3)_5X^{2+}$ family (X is a uninegative ligand such as Cl$^-$ or Br$^-$).[3] It was concluded that the environmental influence on the photoredox chemistry of these complexes is appreciable. In particular, whether a LMCT state is bound or dissociative appeared to depend upon the solvent. Moreover, it was suggested that photooxidation of the solvent via a solvent-to-metal charge-transfer process is reasonably general for deep ultraviolet excitations of metal complexes.

Our interest in base-generating photoinitiators for microlithographic applications led us to examine the ultraviolet spectroscopy and photochemistry of several $Co(NH_2R)_5X^{2+}$ complexes (R is methyl, ethyl, or *n*-propyl) in different solvents.[4] Unlike the results obtained previously for the $Co(NH_3)_5X^{2+}$ family, we find that the LMCT excited states of these alkylamine complexes undergo redox reactions with no discernible chemical involvement of solvent. This disparity suggests the need to revisit the issue of solvent influence on the reactivity of LMCT excited states. Reported below are spectral and photochemical data that facilitate some meaningful discussion along this line.

Experimental Section

Analytically pure samples of $[Co(NH_3)_5Br](ClO_4)_2$[5] and $[Co(NH_2CH_3)_5Br](ClO_4)_2$[6] were synthesized according to published procedures. Distilled water and commercially available acetonitrile (Burdick and Jackson spectral grade), methanol (Baker HPLC grade), and glycerol (Baker reagent grade) were used as solvents. Solutions of the complexes were acidified to pH ~3 with perchloric acid and maintained at 10.0 ± 0.5 °C in thermostated 1-cm quartz cells. Electronic absorption spectra were measured on a Varian DMS 300 spectrophotometer. Photolyses at selected wavelengths above 254 nm were conducted with a high-pressure mercury lamp in conjunction with a monochromator, while low-pressure mercury, cadmium, and zinc lamps were employed for 254-, 229-, and 214-nm excitations, respectively. Incident light intensities at wavelengths ≥254 nm were determined with a ferrioxalate actinometer,[7] while the redox decomposition of $Co(NH_3)_5Cl^{2+}$ was employed as an actinometer at shorter wavelengths.[3c] Photolyzed solutions were analyzed for Co^{2+}[8] and formaldehyde[9] by standard methods.

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[1] (a) Curtis, J. C.; Sullivan, B. P.; Meyer, T. J. *Inorg. Chem.* **1983**, *22*, 224. (b) Manuta, D. M.; Lees, A. J. *Inorg. Chem.* **1983**, *22*, 3825. (c) Caspar, J. V.; Meyer, T. J. *J. Am. Chem. Soc.* **1983**, *105*, 5583. (d) Kober, E. M.; Sullivan, B. P.; Meyer, T. J. *Inorg. Chem.* **1984**, *23*, 2098. (e) Kaim, W.; Kohlmann, S. *Inorg. Chem.* **1989**, *28*, 868. (f) Dodsworth, E. S.; Lever, A. B. P. *Coord. Chem. Rev.* **1990**, *97*, 271. (g) Suppan, P. *J. Photochem. Photobiol. A* **1990**, *50*, 293.

[2] Lees, A. J. *Chem. Rev.* **1987**, *87*, 711.

[3] (a) Endicott, J. F.; Ferraudi, G. J. *J. Am. Chem. Soc.* **1974**, *96*, 3681. (b) Endicott, J. F.; Ferraudi, G. J.; Barber, J. R. *J. Am. Chem. Soc.* **1975**, *97*, 219. (c) Endicott, J. F.; Ferraudi, G. J.; Barber, J. R. *J. Phys. Chem.* **1975**, *79*, 630. (d) Ferraudi, G. J.; Endicott, J. F.; Barber, J. R. *J. Am. Chem. Soc.* **1975**, *97*, 6406.

[4] (a) Weit, S. K.; Kutal, C. *Inorg. Chem.* **1990**, *29*, 1455. (b) Kutal, C.; Weit, S. K.; MacDonald, S. A.; Willson, C. G. *J. Coat. Technol.* **1990**, *62*, 63.

[5] Diehl, H.; Clark, H.; Willard, H. H. *Inorg. Synth.* **1939**, *1*, 186.

[6] Book, L. F.; Hui, K. Y.; Lau, O. W.; Li, W.-K. *Z. Anorg. Allg. Chem.* **1976**, *426*, 215.

[7] Hatchard, C. G.; Parker, C. A. *Proc. R. Soc. London, A* **1956**, *235*, 518.

[8] Vydra, F.; Pribil, R. *Talanta* **1960**, *5*, 44.

[9] Schmidt, S. A.; Antloga, M. F.; Markelov, M. *Formaldehyde: Analytical Chemistry and Toxicology*; American Chemical Society: Washington, DC, 1985; Chapter 3.

0020-1669/91/1330-2819$02.50/0

Table I. LMCT Spectral Data for Cobalt(III) Complexes in Different Solvents[a]

	$Co(NH_3)_5Br^{2+}$ Br(σ) → Co[b]		$Co(NH_2CH_3)_5Br^{2+}$			
			Br(σ) → Co		N → Co	
solvent[c]	ν_{max}[d]	ΔE[e]	ν_{max}[d]	ΔE[e]	ν_{max}[d]	ΔE[e]
H_2O	39.52	0.00	37.87	0.00	44.52	0.00
50% glycerol	39.31	0.21	37.59	0.28	44.36	0.16
>99.9% CH_3OH	38.96	0.56	37.40	0.47	44.06	0.46
80% CH_3CN	39.35	0.17	37.59	0.28	44.74	−0.22
>99.9% CH_3CN	38.90	0.62	37.54	0.33	45.14	−0.62

[a] Measured at 10.0 ± 0.5 °C in solvents acidified to pH ~3 with $HClO_4$. [b] σ refers to an electron in an orbital having σ symmetry with respect to the metal–ligand bond. [c] Water is the other component in the mixed-solvent systems. [d] Energy of absorption maximum; units are 10^3 cm^{-1}. Estimated accuracy is 70–100 cm^{-1}. [e] Difference between ν_{max} values measured in water and the indicated solvent; units are 10^3 cm^{-1}.

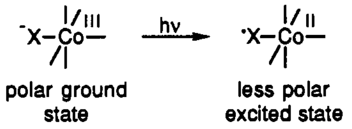
Table II. Properties of Pure Solvents

solvent	D_s[a]	Z, kcal/mol[b]	DN[c]	η, mP[d]
methanol	32.6	83.6	19.0	5.45
acetonitrile	36.2	71.3	14.1	3.45
glycerol	42.5			9450
water	78.5	94.6	≥18.0	8.90

[a] Static dielectric constant at 25 °C; from ref 10. [b] Kosower Z value; from ref 11. [c] Gutmann donor number; from ref 12. [d] Viscosity at 25 °C; from ref 10.

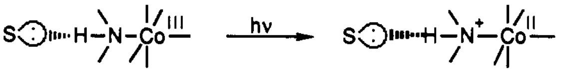
Results and Discussion

LMCT absorption maxima for $Co(NH_3)_5Br^{2+}$ and $Co(NH_2CH_3)_5Br^{2+}$ in several solvents are listed in Table I. Relevant solvent properties are summarized in Table II; the static dielectric constant, D_s,[10] and the Kosower Z value[11] are measures of medium polarity, while the Gutmann donor number, DN,[12] is related to the Lewis basicity of a molecule. The LMCT bands of both complexes exhibit solvatochromism, although the magnitudes of the spectral shift, ΔE in Table I, are rather modest. Thus, the percentage change in transition energy, defined as $\Delta E/\nu_{max} \times 100$, typically is less than 2%. Changing from water to less polar solvents lowers the energy of the Br → Co CT band by similar increments for the two complexes. This red shift reflects the different charge distributions of the ground and excited states. As depicted by the simplified structures in eq 1, the CT transition



(1)

occurs with an inward flow of electron density that decreases the molecular dipole moment. Consequently, switching to a less polar solvent destabilizes the ground state (decreases its solvation energy) more than the excited state and thereby lowers the transition energy. Solvent polarity considerations do not suffice, however, to explain why the N → Co CT transition in $Co(NH_2CH_3)_5Br^{2+}$ undergoes a red shift (relative to water) in hydroxylic solvents (50% glycerol, methanol) and a blue shift in predominantly nonhydroxylic media (80% CH_3CN, CH_3CN).[13] Some specific association between the complex and solvent must be occurring, and we propose a hydrogen-bonding interaction of the type described by eq 2 (S denotes solvent).[2a] The increased positive charge on the amine nitrogen atom(s) following the N → Co transition enhances the acidity of the directly attached hydrogen atoms. Accordingly, the stabilization effect of electron-pair donation from a solvent molecule to one of these hydrogens should be greater in the excited state than in the ground state. In accord with this proposal, the N → Co transition moves to lower energy with increasing Lewis basicity (larger DN value) of the solvent.[14]



(2)

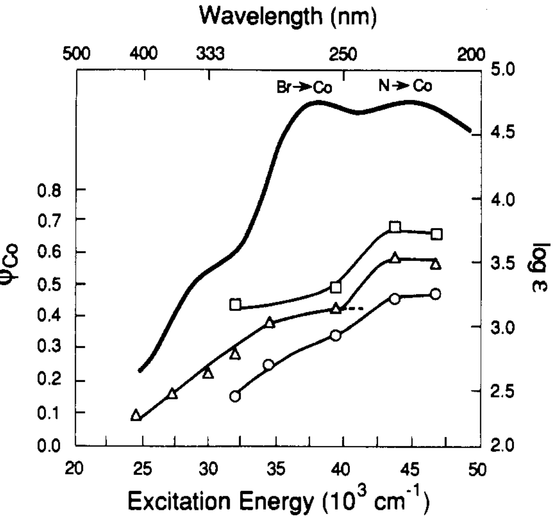
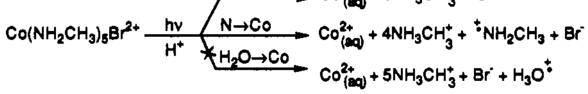


Figure 1. Electronic absorption spectrum (uppermost curve) of $Co(NH_2CH_3)_5Br^{2+}$ in water at 10 °C and quantum yield profiles (lower curves) for photoredox decomposition of $Co(NH_2CH_3)_5Br^{2+}$ in different solvent media: water, △; 80% acetonitrile–water, □; 50% glycerol–water, ○. All solutions were deoxygenated and acidified to pH ~3 with $HClO_4$.

Photochemical studies provide further insights concerning the interaction of solvent with LMCT excited states. Ultraviolet excitation of $Co(NH_2CH_3)_5Br^{2+}$ in deaerated, acidified water causes bleaching of its intense LMCT bands with concurrent production of Co^{2+}, free methylamine, and $Br^{\bullet}$.[4a] As seen from the quantum yield profile in Figure 1, $\Phi_{Co^{2+}}$ increases smoothly with decreasing excitation wavelength within the range 405–254 nm and appears to approach a limiting value (dashed line) characteristic of reaction from a bound state. The observed stoichiometry under these conditions (eq 3a) indicates that this state is Br → Co CT in character. Below 254 nm, however, a second redox process increases in importance, as evidenced by the sudden rise in $\Phi_{Co^{2+}}$ to a new plateau. Since the appearance of this process coincides with the population of the higher energy N → Co CT excited state (Figure 1), we conclude that redox decomposition is an intramolecular process occurring directly from this upper state (eq 3b) in competition with internal conversion to the Br → Co CT state. Oxygen scavenging[4a] and product analysis (formaldehyde, an expected hydrolysis product of the photogenerated $^{\bullet+}NH_2CH_3$ radical cation,[15] appears in the photolyte) results are in complete accord with this interpretation.



(3a)
(3b)
(3c)

As an alternative explanation for the rise in $\Phi_{Co^{2+}}$ at short excitation wavelengths, consider a pathway in which an initially populated LMCT state crosses to a dissociative CT state involving the transfer of an electron from a solvent molecule to the complex (eq 3c). This type of intermolecular solvent-to-complex CT process has been proposed to explain the deep-UV photoredox chemistry

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(10) (a) Gordon, A. J.; Ford, R. A. *The Chemist's Companion*; Wiley-Interscience: New York, 1972. (b) *Handbook of Chemistry and Physics*; Weast, R. C., Ed.; CRC Press: Cleveland, OH, 1972–1973.

(11) Kosower, E. M. *An Introduction to Physical Organic Chemistry*; John Wiley: New York, 1968; p 301.

(12) Gutmann, V.; Resch, G.; Linert, W. *Coord. Chem. Rev.* **1982**, *43*, 133.

(13) Unfortunately, competitive absorption by solvent precludes an accurate assessment of the solvatochromic behavior of the high-energy N → Co CT band in $Co(NH_3)_5Br^{2+}$.

(14) This type of hydrogen-bonding interaction also has been invoked to explain trends in the redox thermodynamics of metal am(m)ine complexes: (a) Chang, J. P.; Fung, E. Y.; Curtis, J. C. *Inorg. Chem.* **1986**, *25*, 4233. (b) Lay, P. A.; McAlpine, N. S.; Hupp, J. T.; Weaver, M. J.; Sargeson, A. M. *Inorg. Chem.* **1990**, *29*, 4322.

(15) Mann, C. K.; Barnes, K. K. *Electrochemical Reactions in Nonaqueous Systems*; Marcel Dekker: New York, 1970; Chapter 9.

of $Co(NH_3)_5X^{2+}$ complexes, especially in mixed-solvent media.[3] Data in Figure 1, however, clearly militate against this possibility for $Co(NH_2CH_3)_5Br^{2+}$. Thus, unlike the behavior of the ammine family, the qualitative features of the quantum yield profile in water are retained in both 80% acetonitrile and 50% glycerol solutions. In particular, there is no evidence for the development of photodissociative character (i.e. a continuous rise in $\Phi_{Co^{2+}}$ toward unity) at high excitation energies in these mixed solvents. It should be noted that the values of $\Phi_{Co^{2+}}$ at a specific wavelength vary with solvent in the order 80% acetonitrile > water > 50% glycerol. This trend tracks solution viscosities (η in Table II) and suggests that solvent influences the photoredox behavior of $Co(NH_2CH_3)_5Br^{2+}$ primarily through its effect upon the separation/recombination kinetics of primary (solvent caged) and secondary (solvent separated) radical-pair photoproducts.[16,17]

We have found that members of the $Co(NH_2R)_5Cl^{2+}$ family undergo photoredox chemistry in mixed-solvent media very similar to that just described for $Co(NH_2CH_3)_5Br^{2+}$. Quite generally, then, it appears that the replacement of NH_3 by NH_2R in the first coordination sphere of cobalt enhances the importance of intramolecular photodecomposition (eqs 3a,b) relative to the intermolecular pathway involving photooxidation of solvent (eq 3c). Such behavior cannot be attributed to the ability of the peripheral alkyl groups to shield the complex from the solvent, since comparable solvatochromatic shifts occur for the Br → Co CT band in $Co(NH_3)_5Br^{2+}$ and $Co(NH_2CH_3)_5Br^{2+}$ (note ΔE values in Table I). We would expect diminished solvent sensitivity of this band for the methylamine complex if such shielding were important.[18] A more attractive explanation is that the LMCT excited states in alkylamine complexes possess weaker Co–N bonds than their ammine analogues. While both types of complex experience photoinduced bond labilizations in their LMCT states resulting from the population of σ-antibonding orbitals, it is quite likely that additional bond weakening occurs in the alkylamine systems owing to nonbonding repulsions between the bulky alkyl groups.[19] Weaker bonding should favor intramolecular decomposition from the initially populated LMCT state over crossing to the potential energy surface of the solvent → Co CT state.

Finally, let us return to the question posed at the outset concerning the role of solvent in the LMCT photochemistry of metal complexes. The present results, in conjunction with earlier work,[3] suggest the following answer: solvent may participate chemically in the reactions of LMCT excited states, but such behavior is not universal. In retrospect, this should not be surprising, since the reactivity of LMCT states undoubtedly is determined by several factors (metal, ligand type, excitation wavelength, nature of solvent), the relative importance of which will vary from system to system.

Summary

The solvatochromism exhibited by the LMCT absorption bands of $Co(NH_3)_5Br^{2+}$ and $Co(NH_2CH_3)_5Br^{2+}$ can be understood in terms of conventional solvent polarity and hydrogen-bonding interactions. Unlike their ammine counterparts, complexes belonging to the $Co(NH_2R)_5X^{2+}$ family undergo redox chemistry from LMCT excited states without accompanying oxidation of the solvent. This disparity has been ascribed to weaker Co–N bonding in the LMCT states of the alkylamine complexes.

Acknowledgment. We thank the U.S. National Science Foundation (Grant DMR-8715635) and the IBM Corp. for financial support.

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(16) Scandola, F.; Bartocci, C.; Scandola, M. A. *J. Am. Chem. Soc.* **1973**, *95*, 7898.

(17) Harris, A. L.; Brown, J. K.; Harris, C. B. *Annu. Rev. Phys. Chem.* **1988**, *39*, 341.

(18) Examples of pendant group shielding effects on metal-to-ligand CT excited states: (a) Reitz, G. A.; Demas, J. N.; DeGraff, B. A.; Stephens, E. M. *J. Am. Chem. Soc.* **1988**, *110*, 5051. (b) Sacksteder, L.; Demas, J. N.; DeGraff, B. A. *Inorg. Chem.* **1989**, *28*, 1787.

(19) (a) Buckingham, D. A.; Foxman, B. M.; Sargeson, A. M. *Inorg. Chem.* **1970**, *9*, 1790. (b) Foxman, B. M. *J. Chem. Soc., Chem. Commun.* **1972**, 515. (c) Swaddle, T. W. *Can. J. Chem.* **1977**, *55*, 3166. (d) Foxman, B. M. *Inorg. Chem.* **1978**, *17*, 1932.

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An Unsymmetrical Binuclear Ruthenium(II) Complex of Tris(2-pyridyl)-1,3,5-triazine and Its Identification by ^1H NMR Spectroscopy

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Introduction

The past several years have seen an explosive growth in the field of ruthenium polypyridine chemistry. These complexes are of interest because of photochemical and electrochemical properties that predicate their utility in a variety of photoredox applications.[1] Since many of these applications require the transfer of more than one electron, there has been considerable recent interest in the design of polynuclear ruthenium complexes that might satisfy this requirement. These complexes typically involve a bridging ligand (BL) which has two or more bidentate sites into which are incorporated $Ru(bpy)_2^{2+}$ subunits, where bpy = 2,2′-bipyridine.[2–6] Nearly all of these bridging ligands are symmetrical, so that the ruthenium atoms occupy identical, chemically equivalent binding sites. For systems designed to investigate mixed-valence states, binding of two different metals, or sites with different auxiliary ligands, unsymmetrical bridging ligands might prove useful.

In this work we describe how tris(2-pyridyl)-1,3,5-triazine (TPT) can be used as a bridging ligand that incorporates two ruthenium(II) atoms in nonequivalent sites such that one Ru is bound in a bidentate fashion and the other is bound in a tridentate fashion. The same ligand has previously been used to form a dicobalt[7] or dimercury[8] complex using both a bidentate and a tridentate site. There has also been a recent report of TPT binding two $Ru(CO)_2Cl_2$ moieties in a symmetric fashion utilizing two equivalent bidentate sites.[9] It is, in fact, possible that TPT could bind three metals using three equivalent bidentate sites.

A principal problem associated with the preparation of unsymmetrical binuclear polypyridyl complexes is their characterization by ^1H NMR spectroscopy due to the complexity found in the aromatic region of their spectra. Detailed analysis is often impossible even with the assistance of a variety of sophisticated techniques. We recently demonstrated the utility of bpy-d_8 as an auxiliary ligand in the formation and characterization of an unsymmetrical mononuclear ruthenium(II) complex.[10] In this paper we extend the technique to include the perdeuterio analogue of 2,2′;6′,2″-terpyridine (tpy) and demonstrate its utility in the characterization of a binuclear complex of TPT.

Experimental Section

Nuclear magnetic resonance spectra were recorded on a General Electric QE-300 spectrometer in CD_3CN or $CDCl_3$ with chemical shifts reported in parts per million downfield from $(CH_3)_4Si$. FAB mass spectra were obtained on a VG 70-SEQ mass spectrometer using *m*-nitrobenzyl alcohol as a matrix. Tris(2-pyridyl)-1,3,5-triazine was pur-

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(1) For an excellent review see: Juris, A.; Balzani, V.; Barigelletti, F.; Campagna, S.; Belser, P.; von Zelewsky, A. *Coord. Chem. Rev.* **1988**, *84*, 85. See also: Kalyanasundaram, K. *Coord. Chem. Rev.* **1982**, *46*, 159. Krause, R. A. *Struct. Bonding* (*Berlin*) **1987**, *67*, 1.

(2) (a) Fuchs, Y.; Lofters, S.; Dieter, T.; Shi, W.; Morgan, R.; Strekas, T. C.; Gafney, H. D.; Baker, A. D. *J. Am. Chem. Soc.* **1987**, *109*, 2691. (b) Braunstein, C. H.; Baker, A. D.; Strekas, T. C.; Gafney, H. D. *Inorg. Chem.* **1984**, *23*, 857.

(3) Thummel, R. P.; Chirayil, S. *Inorg. Chim. Acta* **1988**, *154*, 77.

(4) (a) Barigelletti, F.; De Cola, L.; Balzani, V.; Hage, R.; Haasnoot, J. G.; Reedijk, J.; Vos, J. G. *Inorg. Chem.* **1989**, *28*, 4344. (b) Hage, R.; Dijkhuis, A. H. J.; Haasnoot, J. G.; Prins, R.; Reedijk, J.; Buchanan, B. E.; Vos, J. G. *Inorg. Chem.* **1988**, *27*, 2185.

(5) Sahai, R.; Baucom, D. A.; Rillema, D. P. *Inorg. Chem.* **1986**, *25*, 3843.

(6) Ernst, S. D.; Kaim, W. *Inorg. Chem.* **1989**, *28*, 1520.

(7) Vagg, R. S.; Warrener, R. N.; Watton, E. C. *Aust. J. Chem.* **1969**, *22*, 141.

(8) Halfpenny, J.; Small, R. W. H. *Acta Crystallogr.* **1982**, *B38*, 939.

(9) Thomas, N. C.; Foley, B. L.; Rheingold, A. L. *Inorg. Chem.* **1988**, *27*, 3426.

(10) Chirayil, S.; Thummel, R. P. *Inorg. Chem.* **1989**, *28*, 813.