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**Synthesis and Characterization of a Novel
Oxovanadium(IV) Complex and
Conductometric Studies with
N,N'-bis(Salicylidene)-1,2-bis-
(*p*-aminophenoxy)ethane**

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ABSTRACT

A new oxovanadium complex of the Schiff base obtained by the condensation of 1,2-bis(*p*-aminophenoxy)ethane with salicylaldehyde was synthesized. The complex has been characterized by elemental analyses, magnetic measurements, UV-VIS and IR spectra. Stability constants and thermodynamic values for complexation between Cu(NO₃)₂, Zn(NO₃)₂·6H₂O, and VOSO₄·5H₂O salts and the ligand synthesized by the method described in the literature in 80% dioxane–water and

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pure methanol were determined by conductance measurements. The stability constants ($\log K_c$) in 80% dioxane/water decrease in the order $\text{Cu(II)} > \text{Zn(II)}$. However, just the opposite behavior has been obtained for these metal complexes with the ligand in methanol ($\text{Zn(II)} > \text{Cu(II)}$). The magnitudes of these ion association constants are related to the nature of solvation of the cation and of the complexed cation. The mobility of the complexes is also dependent, in part, upon solvation effects. Since the mobility of the VO(IV)L complex has been found to be higher than that of VO(IV) ion, assuming that the complex–solvent interaction in the VO(IV)-L systems is comparatively weak. A major consequence of the complexation is the increase in the molar conductivity of the complex and a corresponding large decrease in κ values. For this reason, it was not obtained any stability constant values for VO(IV)-L systems in two type of solvents.

Key Words: Schiff base; Novel oxovanadium; Conductometric studies.

INTRODUCTION

In recent years, the complexes of oxovanadium(IV) have received considerable attention. Oxovanadium(IV) chelates containing tetradentate Schiff base ligands derived from 1,2-diamines have been the subject of several recent reports.^[1–7] These square-pyramidal complexes exhibit a strong tendency to remain five-coordinate in both donor and non-donor solvents.^[8,9]

In this paper, we report the synthesis of a new oxovanadium complex of a Schiff base derived from the condensation of 1,2-*bis*(*p*-aminophenoxy)ethane with salicylaldehyde (Fig. 1). The resulting complex was studied by elemental analyses, magnetic measurements, UV-VIS and IR spectra. Furthermore, we report the stability constants and thermodynamic values for the complexation of Cu(II) , Zn(II) , and VO(IV) in 80% dioxane–water and pure methanol as solvent with this ligand containing nitrogen and oxygen donor atoms.

RESULTS AND DISCUSSION

The analytical data for the ligand and VO(IV) complex are listed in Table 1. The ligand was prepared by the reaction of 1,2-*bis*(*p*-aminophenoxy)ethane with salicylaldehyde in absolute ethanol. The ligand, on interaction with $\text{VOSO}_4 \cdot 5\text{H}_2\text{O}$, yields a complex corresponding to the general formula VOL . The metal to ligand ratio of the VO(IV) complex was found to be 1 : 1.



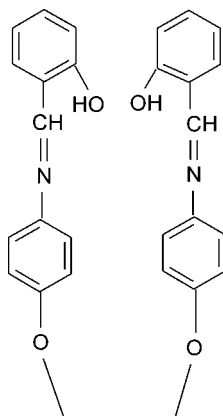


Figure 1. *N,N'*-bis(Salicylidene)-1,2-bis-(*p*-aminophenoxy)ethane.

IR Spectra

The tentative assignment of the important bands of the Schiff base under investigation and its corresponding metal complex are recorded in Table 2. The important features for the Schiff base and its complex maybe summarized as follows:

The broad band that appeared in the IR spectrum of the Schiff base at 2892 cm^{-1} is assigned to the stretching vibration of the intramolecular hydrogen bonded OH in the molecule. Similar bands were observed at the same frequency in the IR spectra of salicylideneanilines.^[1,10-12] This band disappeared in the IR spectrum of the complex. The band at 1282 cm^{-1} in the IR spectrum of the ligand is ascribed to the phenolic C–O stretching vibration. This band is found

Table 1. The colors, formulas, formula weight, yields, melting points, and elemental analyses results of the ligand and the vanadium complex.

Compounds	F.W. (g mol^{-1})	M.p. ($^{\circ}\text{C}$)	Yield (%)	Elemental analyses found (calcd), %			μ_{eff} (B.M.)
				C	H	N	
Ligand (yellow) $\text{C}_{28}\text{H}_{24}\text{N}_2\text{O}_4$	452.00	214.0	68.0	74.36 (74.34)	4.95 (5.31)	6.25 (6.19)	—
VO(IV)L (orange yellow) $\text{C}_{28}\text{H}_{22}\text{N}_2\text{O}_5\text{V}$	516.94	285.0	66.0	65.15 (64.94)	4.20 (4.26)	5.65 (5.42)	1.74



Table 2. Characteristic IR bands (cm^{-1}) of the ligand and vanadium complex in KBr pellets.^a

Ligand	VOL	Assignment
2892 m	—	Intramolecular H-bonded –OH
1627 s	1621 s	C=N stretching
1282 m	1284 m	Phenolic C–O stretching
3041 m	3049 m	C–H aromatic
2892 s	2893 s	C–H aliphatic
—	983 w	V=O stretching
—	480 w	M–N stretching
—	430 w	M–O stretching

^as, strong; m, medium; w, weak.

in the region 1284 cm^{-1} in the IR spectrum of the complex. These changes suggest that the *o*-OH group of this Schiff base moiety has taken part in complex formation. The solid state IR spectrum (in KBr pellet) of the complex compared with that of the ligand indicates that the C=N band 1627 cm^{-1} is shifted to lower frequency values for the complex of VO(IV). This band is found in the region 1621 cm^{-1} in the IR spectra of the complex.^[13,14] The $\nu(\text{V}=\text{O})$ band appears in the range^[2–9] 983 cm^{-1} . The bands between 480 and 430 cm^{-1} are assigned to vibrations that are probably due to M–N and M–O.^[10–15]

Electronic Spectra

The electronic spectral data of the synthesized compounds were recorded in dimethyl formamide (DMF) solutions. The absorption spectrum of the Schiff base is characterized mainly by two absorption bands in the region 275–560 nm. In the spectrum of the Schiff base ligand, the aromatic bands at 210–302 nm ($\epsilon = 14,427\text{ L mol}^{-1}\text{ cm}^{-1}$) are attributed to a benzene $\pi \rightarrow \pi^*$ transition. The band at 420 nm ($\epsilon = 6710\text{ L mol}^{-1}\text{ cm}^{-1}$) is assigned to the imino $\pi \rightarrow \pi^*$ transition. Compared to the free ligand, the imine $\pi \rightarrow \pi^*$ transitions of the complex were shifted to some extent (13 nm), probably because of coordination of the nitrogen atom of the ligand imine group to the metal ion.^[15–18]

Magnetic Properties

The magnetic moment of the VO(IV)L complex of the ligand is 1.74 B.M.^[2–9] Since the VO(IV)L complex is paramagnetic, its ^1H NMR spectrum could not be obtained.

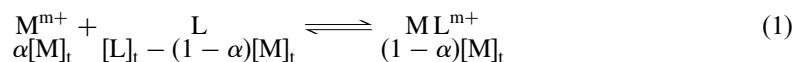


Conductivity

The complex is a non-electrolyte as shown by its molar conductivity (Λ_M) in DMF,^[15-18] which is $5 \Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$. Its structure is presumably based on the familiar square pyramid^[2-9] (Fig. 2).

The Conductometric Study of Ligand with Cu(II), Zn(II), and VO(VI) Salts

When the Schiff base ligand (L) forms a 1 : 1 complex with the metal ion (M^{m+}), the equilibrium equation may be written as in Eq. (1)



where M^{m+} , L, $[M]_t$, $[L]_t$, and α are the cation, the ligand, the total concentration of the metal salt and the Schiff base ligand, and fraction of free cations, respectively. Thus, the complex formation constant (K_{ML}) is defined by

$$\begin{aligned} K_{ML} &= \frac{[ML^{m+}]}{[M^{m+}][L]} \\ &= 1 - \frac{\alpha}{\alpha[L]} \end{aligned} \quad (2)$$

The apparent conductivity (κ_{app}) of the metal nitrate/sulfate (MA_m) solution in the presence of ligand L is given by

$$\kappa_{app} = \kappa_{MA_m} + \kappa_{MLA_m} \quad (3)$$

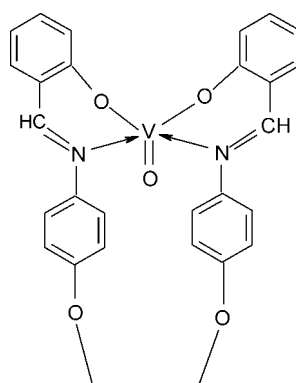


Figure 2. Suggested structure of the square-pyramidal VO(IV)L complex of the ligand.



were A denotes an anion, κ_{MAm} and κ_{MLAm} refer to the conductivities of the electrolyte and the ligand–electrolyte complex, respectively. The molar conductivities are

$$\begin{aligned}\Lambda_{MAm} &= \frac{\kappa_{MAm}}{[M^{m+}]} \\ &= \frac{\kappa_{MAm}}{\alpha[M]_t}\end{aligned}\quad (4)$$

$$\begin{aligned}\Lambda_{MLAm} &= \frac{\kappa_{MLAm}}{[ML^{m+}]} \\ &= \frac{\kappa_{MLAm}}{(1 - \alpha)[M]_t}\end{aligned}\quad (5)$$

were Λ_{MAm} and Λ_{MLAm} designate the molar conductivities of the electrolyte and the ligand–electrolyte complex, respectively. The apparent molar conductivity of the metal salt, is defined as in Eq. (6).

$$\begin{aligned}\Lambda_{app} &= \frac{\kappa_{app}}{[M]_t} \\ &= \alpha\Lambda_{MAm} + (1 - \alpha)\Lambda_{MLAm}\end{aligned}\quad (6)$$

As a consequence of Eq. (6), Eq. (2) maybe transformed into Eq. (7)

$$K_e = \frac{(\Lambda_{MAm} - \Lambda_{app})}{(\Lambda_{app} - \Lambda_{MLAm})[L]}\quad (7)$$

where

$$[L] = [L]_t - \frac{[M]_t(\Lambda_{MAm} - \Lambda_{app})}{(\Lambda_{MAm} - \Lambda_{MLAm})}$$

The differences in complexing ability using two types of solvents (80% dioxane/water and methanol) between the Schiff base and metal ion can be determined based on the thermodynamic equation shown below

$$\Delta G_c^\circ = -2.303RT \log K_e\quad (8)$$

where ΔG_c° is the Gibbs free energy of complexation in these solvents.

In the complexation of Cu(II) with the Schiff base in 80% dioxane/water, the interactions with binary solvent mixture and the metal ion are greatly decreased by ionic bonding of the Schiff base oxygen and nitrogen to the metal ion. Therefore, ΔG_c° for Cu(II) ion is expected to be larger than the free energies for Zn(II) ion in 80% dioxane/water. However, for the complexations in methanol, the hydrogen bond interaction between methanol and the uncomplexed ligand is greater for synthesized Schiff base.



It can, therefore, be presumed that the main reasons for the higher complexing ability of the metal ions with the ligand in 80% dioxane/water are less effective shielding of the metal ion in the complexation and weaker hydrogen bonding of uncomplexed ligand in 80% dioxane/water.

The experimental molar conductance equations and all calculations for stability constant values have been published in our previous works.^[11,18–20,22] All of these experimental studies have been made for the 1:1 ratio of metal–ion and Schiff base ligand. The results show that the κ_{app} vs. $[M^{m+}]$ plots in Figs. 3–8 show a decrease of κ_{app} with an increase in M^{m+} concentration except for the Schiff base ligand–VO(IV) system. This indicates that complexation occurs between the Schiff base ligand and Cu(II) and Zn(II) metal ions, and that the Schiff base ligand–Cu(II) and Zn(II) metal ion complexes are less mobile than the free Cu(II) and Zn(II) metal ions. The κ_{app} vs. $[M^{m+}]$ plots of the L-VO(IV) system show an increase of κ_{app} as the M^{m+} concentration increases. Assuming that the complex–solvent interaction is comparatively weak, major consequences of this complexation are the increase in the molar conductivity of complex and a corresponding large decrease as κ values in Eqs. 3–5.

This indicates that Schiff base ligand forms a complex with VO(IV), and that the Schiff base ligand–VO(IV) complex is more mobile than the VO(IV) ion; consequently using two types of solvents (80% dioxane/water and methanol), the conductometric determination of the complex formation constant for the VO(IV)-L system was not possible.^[19] It can be seen from Table 3 no data are given for the VO(IV)-L systems.

The stability constants ($\log K_c$) in 80% dioxane/water decrease in the order Cu(II) > Zn(II). However, the opposite behavior has been observed for these

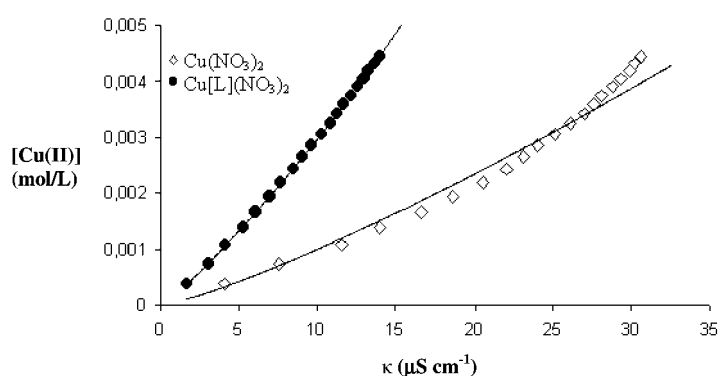


Figure 3. The plots of $[Cu(II)]$ (mol L^{-1}) vs. observed conductivity, κ ($\mu\text{S cm}^{-1}$) of $\text{Cu}(\text{NO}_3)_2$ with L in 80% dioxane/water mixtures at 25 °C.



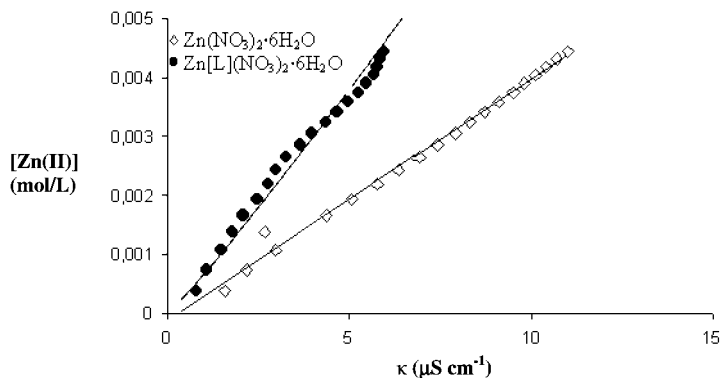


Figure 4. The plots of $[Zn(II)]$ (mol L^{-1}) vs. observed conductivity, κ ($\mu\text{S cm}^{-1}$) of $Zn(NO_3)_2 \cdot 6H_2O$ with L in 80% dioxane/water mixtures at 25 °C.

metal complexes with the ligand in methanol (see Table 3): $Zn(II) > Cu(II)$. The synthesized Schiff base ligand forms a more stable complex with $Cu(II)$ than with $Zn(II)$ in 80% dioxane/water. On the other hand, in methanol, the complex of $Zn(II)$ unexpectedly is more stable than the corresponding $Cu(II)$ complex. There are some differences in the complexation constant values of the metal complexes in methanol compared to 80% dioxane/water. We observed that the stabilities of the complexes are affected not only by the Schiff base and various metal ions but also by the physical properties of the solvent. Eighty percentage of dioxane/water mixtures have a lower dipole moment than methanol, and this

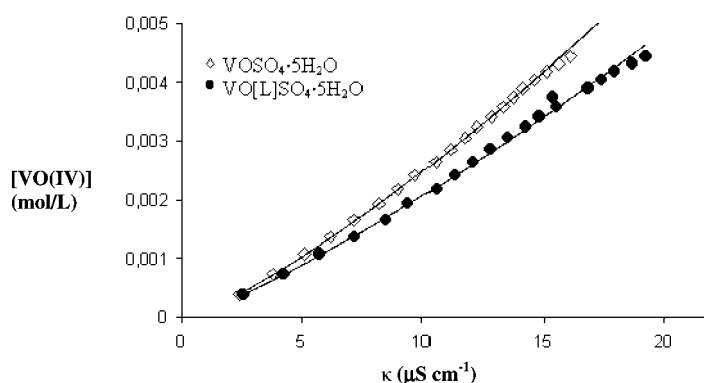


Figure 5. The plots of $[VO(IV)]$ (mol L^{-1}) vs. observed conductivity, κ ($\mu\text{S cm}^{-1}$) of $VOSO_4 \cdot 5H_2O$ with L in 80% dioxane/water mixtures at 25 °C.



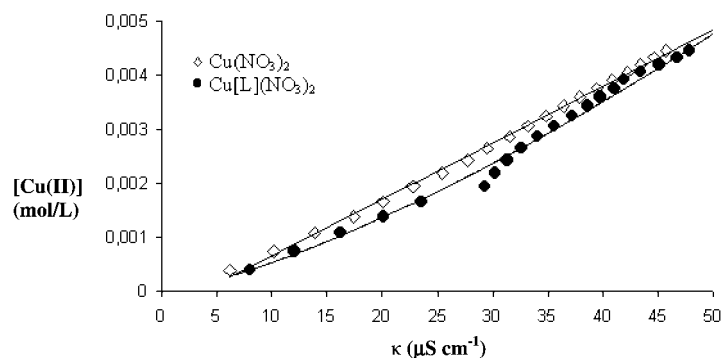


Figure 6. The plots of $[\text{Cu(II)}]$ (mol L^{-1}) vs. observed conductivity, κ ($\mu\text{S cm}^{-1}$) of $\text{Cu}(\text{NO}_3)_2$ with L in methanol at 25°C .

mixture has a dielectric constant lower than that of methanol (see Table 4). Thus, the energetic consequences of both primary and secondary solvation should be qualitatively dissimilar in the two solvents.^[22]

Additionally, it is expected that the metal ions in 80% dioxane/water are less shielded than the metal ions in methanol. That is, 80% dioxane/water is a poor solvator for cations, and the reason for this could be that the metal ions are assumed to be more strongly solvated by methanol than in 80% dioxane/water. As a result, there could be a poor ion–dipole interaction possible between the metal ion and a ligand molecule in methanol compared with that in 80% dioxane/water, and the results are found to be highly dependent on the nature of both ion–solvent and ion–ligand interactions.

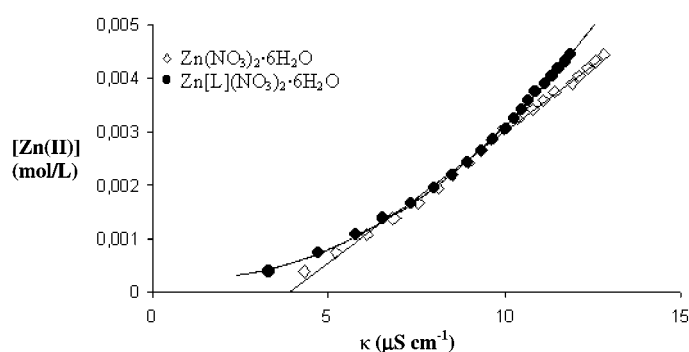


Figure 7. The plots of $[\text{Zn(II)}]$ (mol L^{-1}) vs. observed conductivity, κ ($\mu\text{S cm}^{-1}$) of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ with L in methanol at 25°C .



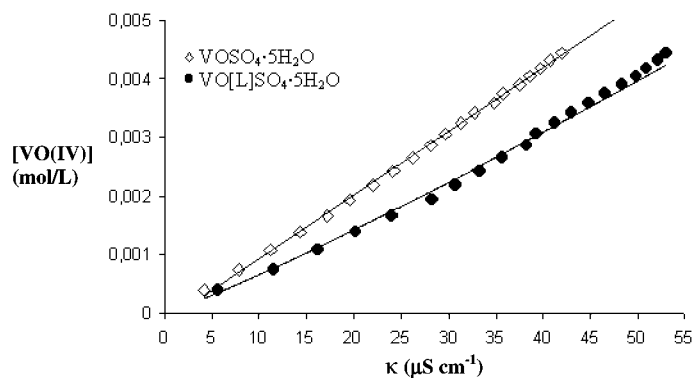


Figure 8. The plots of [VO(IV)] (mol L⁻¹) vs. observed conductivity, κ ($\mu\text{S cm}^{-1}$) of $\text{VOSO}_4 \cdot 5\text{H}_2\text{O}$ with L in methanol at 25 °C.

EXPERIMENTAL

Reagents and Measurements

N,N'-bis(salicylidene)-1,2-bis-(*p*-aminophenoxy)ethane and its Cu(II) and Zn(II) complexes were synthesized by the method described in the literature (suggested structures are tetrahedral for the Zn(II) and square-planar for the Cu(II) complexes of the ligand).^[9] The electronic spectra of the complex in the UV-VIS region were recorded in DMF solutions using a Shimadzu Model 160 UV-VIS spectrophotometer. The IR spectra of the complex in KBr pellets were recorded with a Midac 1700 instrument. Magnetic susceptibilities were determined on a Sherwood Scientific magnetic susceptibility balance (Model MK1) at room temperatures (23 °C) using $\text{Hg}[\text{Co}(\text{SCN})_2]$ as calibrant.^[23] The elemental analyses were determined on a Carlo Erba instrument.

Table 3. $\log K_c$ and $-\Delta G^\circ$ values for the interaction of ligand with $\text{Cu}(\text{NO}_3)_2$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, and $\text{VOSO}_4 \cdot 5\text{H}_2\text{O}$ in 80% dioxane/water and methanol solvents at 25 °C by a conductometric study.

Value	Solvent	Cu(II)	Zn(II)	VO(IV)
$\log K_c$	80% Dioxane/water	4.17	3.00	—
	Methanol	2.32	2.75	—
$-\Delta G^\circ$	80% Dioxane/water	5684.53	4093.30	—
	Methanol	3159.23	3796.01	—



Table 4. Dielectric constant values of 80% dioxane/water and methanol.^[21]

Water	Dioxane	Methanol	80% Dioxane/water
78.54	2.21	33.62	18.23

Synthesis of the VO(IV)L Complex

The salt $\text{VOSO}_4 \cdot 5\text{H}_2\text{O}$ (20.00 mmol, 5.06 g) was dissolved in hot methanol (50 mL) and a mixture of NEt_3 (40 mmol, 2.26 g) and the ligand (20.00 mmol, 9.04 g) in DMF (50 mL) was added with stirring over about 10 min. The mixture was kept at 60–64 °C and stirred for about 2–3 hr. The refluxed solution was then poured into ice-cold water, when a colored solid separated which was isolated by filtration and washed with diethyl ether, hot water, and ethyl alcohol. The resulting solid was recrystallized in a mixture of 25 mL dimethyl sulfoxide/25 mL DMF and dried over anhydrous CaCl_2 *in vacuo* at room temperature. The yield was 10.34 g (65%) in the complex with respect to the ligand. The complex decomposes at 285 °C and is almost insoluble in water but partially soluble in polar solvents (dimethyl sulfoxide and DMF).

The Conductometric Study of the Ligand with Cu(II), Zn(II), and VO(IV) Salts

High purity $\text{Cu}(\text{NO}_3)_2$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Fluka; 99%) and $\text{VOSO}_4 \cdot 5\text{H}_2\text{O}$ (Fluka; 96%) were used without further purification. The water used in the conductometric studies was redistilled from alkaline potassium permanganate; the conductivity was less than $6 \times 10^{-7} \text{ S cm}^{-1}$.

The conductances were measured at 25 ± 0.05 °C. The measuring equipment consisted of a glass vessel (type Ingold) with an external jacket. At the same time, the system was connected to a thermostatted water bath (25 ± 0.05 °C) and a conductivity cell (Cole-Parmer 19050-66) with a cell constant of 0.3162 cm^{-1} . The conductivity was measured with a model Sc-170 Suntex conductometer.

The solutions were prepared at constant 1 : 1 ratio of metal salt to ligand in an 80% dioxane/water mixture and in methanol. All solutions were prepared in a dry box and transferred to the dry conductivity cell. The atmosphere was replaced by nitrogen gas. After the cell was thermally equilibrated in a water bath, the resistance of the solution was measured. $\log K_e$ and $-\Delta G^\circ$ values for the reaction of the ligand with the cations were determined by a conductometric procedure outlined previously.^[19,20] The results are reported



as the average and standard deviation from the average of four–six independent experimental determinations.

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