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Dioxygen affinities of Schiff base cobalt(II) complexes with aza-crown or morpholino pendants

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Abstract Schiff base Co(II) complexes, CoL_2^1–CoL_2^6 with aza-crown or morpholino pendants were synthesized. The oxygenation constants (KO_2) of these complexes in $MeOCH_2CH_2OMe$ solution were measured over the range of −5 to 25 °C, and the thermodynamic parameters (ΔH^0, ΔS^0) for oxygenation were calculated based on these KO_2 values. The effects of different substituents on the Schiff base ligand with respect to the modulation of O_2-binding capability were explored. The results indicate that the dioxygen affinities of the Co(II) complexes are much more enhanced by aza-crown pendants than that by morpholino pendants, and the O_2-binding capabilities of the aza-crown pendants complexes can also be enhanced by adding Na^+ cations.

Introduction

Synthetic oxygen carriers are of great interest as models for metalloenzymes involved in oxygen storage and transport. Studies on dioxygen binding to transition metal complexes are of intrinsic importance and provide opportunities to address the fundamental chemistry of biological oxygen carriers and oxygenases. The Co(II) complexes of Schiff bases, such as Salen and its analogues, have been the first and most extensively investigated due to their structural similarity to the metal sites found in biological systems [1]. Schiff base transition metal complexes have been one of the most successful classes of synthetic oxygen carriers [2, 3]. In the past decades, a number of reports have appeared on Schiff base complexes as models for oxygen carrying metalloenzymes [4] and oxygenases [5], such as haemoglobin and cytochrome P-450 enzymes, which play important roles in the catalytic oxidation of various organic compounds [1]. However, these complexes can easily dimerize, and so lose activity upon absorption of dioxygen [6]. Avdeef demonstrated that it is possible to form stable oxygen adducts when these Co(II) complexes are modified by appropriate substituents [7]. Thus, various substituents have been employed to improve O_2-binding capabilities of the complexes [8, 9]. Among them, the crown ether-containing Schiff bases have attracted much attention, due to the interesting properties conferred by the hydrophobicity of the outer ethylene groups and orderly arrangement of inner oxygen atoms [10, 11]. Previous reports have indicated that oxa-crowned Schiff base transition metal complexes are good receptors for alkali cations, which bind in the crown ether cavity [12, 13]. These complexes showed much improved O_2-binding activity due to the special configuration and function of the crown ether ring [14, 15]. Co-complexation of a hard cation close to the transition metal centre is believed to play an important role in modulating its oxygen-binding properties [16]. However, to the best of our knowledge, very few studies on the dioxygen affinities of aza-crown-substituted Schiff base complexes have been reported to date. Recent studies have indicated that crown ether-containing Schiff base complexes can also show catalytic activity [17, 18]. These observations inspired us to study the influence of pendant aza-crown substituents on the O_2-binding capabilities of Schiff base Co(II)

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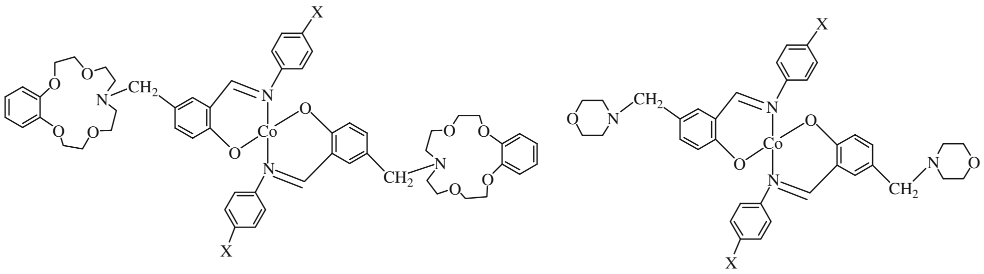

CoL^1_2, X = Me; CoL^2_2, X = Cl; CoL^3_2, X = NO_2

CoL^4_2, X = Me; CoL^5_2, X = Cl; CoL^6_2, X = NO_2

Fig. 1 The structures of the Schiff base cobalt(II) complexes

complexes. Herein, the synthesis of Schiff base Co(II) complexes with aza-crown pendants CoL^n_2 ($n = 1$–3) and their analogues CoL^n_2 ($n = 4$–6; Fig. 1), and the study on their dioxygen affinities are described. The influences of pendant aza-crown ring substituents and the addition of alkali cations on the dioxygen affinities of the Co(II) complexes are also reported.

Experimental

Melting points were determined on a Yanaco MP-500 micro-melting point apparatus and are uncorrected. IR spectra were recorded on a Nicolet-1705X spectrometer. ^1H NMR spectra were recorded on a Bruker AC-200 MHz spectrometer using Me_4Si as internal standard and $CDCl_3$ as solvent. Mass spectra were obtained on a Finnigan LCQ spectrometer. Metal contents were measured using an IRIS-Advantage ICP emission spectrometer. The halogen content was measured using the mercury titration method [19, 20]. Other elemental analyses were performed on a Carlo Erba 1106 elemental analyser. Molar conductances were obtained on a DDS-11A conductivity meter in DMF solutions (1.0×10^{-3} mol L^{-1}). Molar Magnetic Susceptibilities were obtained on a T3-200 magnetic balance at 25 °C.

The Schiff bases HL^1–HL^3 were synthesized according to the literature [21], as was *N*-(3-formyl-4-hydro-xybenzyl) morpholine [22]. All other analytical grade reagents were purchased within China and used without further purification.

Synthesis of Schiff bases HL^4–HL^6

Schiff base HL^4

A solution of *N*-(3-formyl-4-hydroxybenzyl)morpholine (1.77 g, 8 mmol) and *p*-toluidine (0.87 g, 8 mmol) in EtOH (20 cm^3) was stirred for 4 h under N_2 atmosphere at 80 °C, then cooled to room temperature. The yellow precipitate was filtered off and washed with EtOH. After recrystallisation from EtOH, yellow crystals (2.54 g, yield 82%) were obtained. m.p. 95–96 °C. ^1H NMR δ (p.p.m.): 13.04 (s, 1H, OH, D_2O exchangeable), 8.36 (s, 1H, N=CH), 7.57–6.82 (m, 7H, ArH), 3.71–3.52 (m, 6H, OCH_2, NCH_2Ar), 2.81 (t, $J = 5.5$ Hz, 4H, NCH_2), 2.26 (s, 3H, CH_3); I.r. (KBr, cm^{-1}) ν_{max}: 3239, 2961, 2857, 1621, 1601, 1500, 1255, 1194,1112; ESI–MS m/z: 311 (M^++1); (Found C 73.4, H 7.3, N 9.2. $C_{19}H_{22}N_2O_2$ calcd.: C 73.6, H 7.1, N 9.%).

Schiff base HL^5

HL^5 was prepared as described for *HL^4* except for the use of 4-chloroaniline instead of *p*-toluidine to give yellow solid, yield 74%, m.p.144–145 °C. ^1H NMR δ (p.p.m.): 13.08 (s, 1H, OH, D_2O exchangeable), 8.43 (s, 1H, N=CH), 7.52–6.88 (m, 7H, ArH), 3.70–3.54 (m, 6H, OCH_2, NCH_2Ar), 2.82 (t, $J = 5.3$ Hz, 4H, NCH_2). I.r. (KBr, cm^{-1}) ν_{max}: 3245, 2932, 2857, 1623, 1600, 1502, 1254, 1195, 1116; ESI–MS m/z: 332 (M^++1); (Found C 65.5, H 5.6, N 8.3, Cl 10.6. $C_{18}H_{19}N_2O_2Cl$ calcd.: C 65.4, H 5.8, N 8.5, Cl 10.7%).

Schiff base HL^6

HL^6 was prepared as described for *HL^4* except for the use of *p*-nitroaniline instead of *p*-toluidine to give yellow solid, yield 74%, m.p.162–164 °C. ^1H NMR δ (p.p.m.): 13.01 (s, 1H, OH, D_2O exchangeable), 8.41 (s, 1H, N=CH), 7.57–6.84 (m, 7H, ArH), 3.73–3.58 (m, 6H, OCH_2, NCH_2Ar), 2.84 (t, $J = 5.2$ Hz, 4H, NCH_2). I.r. (KBr, cm^{-1}) ν_{max}: 3239, 2930, 2860, 1624, 1601, 1500, 1253, 1192, 1118; ESI–MS m/z: 342 (M^++1); (Found C 63.5, H 5.4, N 12.1. $C_{18}H_{19}N_3O_4$ calcd.: C 63.3, H 5.6, N 12.3%).

General method for preparation of the complexes

A solution of the appropriate Schiff base (2.0 mmol) and $Co(AcO)_2{\cdot}4H_2O$ (1.1 mmol) in EtOH (15 cm^3) was stirred for 2 h under a N_2 atmosphere at 70 °C, and then cooled to room temperature. The precipitate was filtered off and washed with EtOH to give Schiff base Co(II) complex. The pure product was obtained after recrystallization from EtOH.

CoL^1_2

Purple, 84% yield, m.p. 197–199 °C; I.r. (KBr, cm^{-1}) ν_{max}: 2965, 2859, 1609, 1600, 1501, 1254, 1131, 1049; ESI–MS m/z: 1038 (M^{+}+1); (Found C 67.2, H 6.2, N 5.4, Co 5. 8. $C_{58}H_{66}N_4O_{10}Co$ calcd.: C 67.1, H 6.4, N 5.4, Co 5.7%). Λ_m (S cm^2 mol^{-1}): 9.82. Molar magnetic susceptibility $\chi_M = 7.19 \times 10^{-2}$ J mol^{-1} T^{-2}, magnetic moment $\mu_m = 3.84 \times 10^{-23}$ J T^{-1}.

CoL^2_2

Purple, 84% yield, m.p. 207–209 °C; I.r. (KBr, cm^{-1}) ν_{max}: 2930, 2860, 1608, 1601, 1500, 1253, 1126, 1050; ESI–MS m/z: 1079 (M^{+}+1); (Found C 62.2, H 5.6, N 5.4, Cl 6.5, Co 5.6. $C_{56}H_{60}N_4O_{10}Cl_2Co$ calcd.: C 62.3, H 5.8, N 5.2, Cl 6.6, Co 5.5%). Λ_m (S cm^2 mol^{-1}): 10.32, $\chi_M = 6.61 \times 10^{-2}$ J mol^{-1} T^{-2}, $\mu_m = 3.68 \times 10^{-23}$ J T^{-1}.

CoL^3_2

Purple, 82% yield, m.p. 189–191 °C; I.r. (KBr, cm^{-1}) ν_{max}: 2930, 2861, 1609, 1600, 1501, 1251, 1127, 1048; ESI–MS m/z: 1100 (M^{+}+1); (Found C 61.4, H 5.5, N 7.5, Co 5. 5. $C_{56}H_{60}N_6O_{14}Co$ calcd.: C 61.2, H 5.5, N 7.6, Co 5.4%). Λ_m (S cm^2 mol^{-1}): 10.75, $\chi_M = 7.08 \times 10^{-2}$ J mol^{-1} T^{-2}, $\mu_m = 3.81 \times 10^{-23}$ J T^{-1}.

CoL^4_2

Purple, 79% yield, m.p. 192–194 °C; I.r. (KBr, cm^{-1}) ν_{max}: 2962, 2855,1609,1600,1502, 1253, 1196, 1116; ESI–MS m/z: 678 (M^{+}+1); (Found C 67.5, H 6.4, N 8.4, Co 8.9. $C_{38}H_{42}N_4O_4Co$ calcd.: C 67.4, H 6.2, N 8.3, Co 8.7%). Λ_m (S cm^2 mol^{-1}): 8.95, $\chi_M = 6.68 \times 10^{-2}$ J mol^{-1} T^{-2}, $\mu_m = 3.70 \times 10^{-23}$ J T^{-1}.

CoL^5_2

Purple, 78% yield, m.p. > 270 °C; I.r. (KBr, cm^{-1}) ν_{max}: 2930, 2854, 1611, 1600, 1502, 1252, 1197, 1114; ESI–MS m/z: 719 (M^{+}+1); (Found C 60.0, H 5.2, N 8.0, Cl 9.7, Co 8.4. $C_{36}H_{36}N_4O_4Cl_2Co$ calcd.: C 60.2, H 5.0, N 7.8, Cl 9.9, Co 8.2.%. Λ_m (S cm^2 mol^{-1}): 11.45, $\chi_M = 6.79 \times 10^{-2}$ J mol^{-1} T^{-2}, $\mu_m = 3.73 \times 10^{-23}$ J T^{-1}.

CoL^6_2

Purple, 76% yield, m.p. 164–166 °C; I.r. (KBr, cm^{-1}) ν_{max}: 2932, 2859, 1609, 1600, 1500, 1256, 1195, 1116; ESI–MS m/z: 740 (M^{+}+1); (Found C 58.3, H 4.7, N 11.2, Co 7.8. $C_{36}H_{36}N_6O_8Co$ calcd.: C 58.5, H 4.9, N 11.4, Co 8.0%). Λ_m (S cm^2 mol^{-1}): 12.44, $\chi_M = 6.87 \times 10^{-2}$ J mol^{-1} T^{-2}, $\mu_m = 3.75 \times 10^{-23}$ J T^{-1}.

Oxygen uptake studies

The values of KO_2 for complexes CoL^1_2–CoL^6_2 were determined from the volumes of dioxygen absorbed at equilibrium, as described in the literature [8]; these experiments used diglyme (saturated with O_2) as solvent, plus 1.0 mol dm^{-3} of pyridine as axial ligand (B), complex concentration 5×10^{-3} $mol.dm^{-3}$, 9.7×10^4 Pa of O_2 partial pressure. The equilibrium constant (KO_2) was calculated as follows:

$$\mathrm{CoL} + \mathrm{B} \rightleftharpoons \mathrm{CoLB}$$

$$\mathrm{CoLB} + \mathrm{O_2} \rightleftharpoons \mathrm{CoLBO_2}$$

$$KO_2 = \frac{[\mathrm{CoLBO_2}]}{[\mathrm{CoLB}]\,PO_2}$$

where the complexes are expressed in terms of molarities, and the dioxygen concentration is expressed as partial pressure (PO_2) in Torr. Thermodynamic parameters ΔH^0 and ΔS^0 for oxygenation were determined from variation of KO_2 over a range of temperatures. ΔH^0 and ΔS^0 were calculated from the equations $\Delta G^0 = -\mathrm{RTln}\ KO_2$ and $\mathrm{T}\Delta S^0 = \Delta H^0 - \Delta G^0$.

Results and discussion

Compared with the i.r. spectra of the free Schiff bases, those of Co(II) complexes, CoL^1_2–CoL^6_2, were almost at the same frequencies, except for the C=N stretching vibration which was shifted 12–16 cm^{-1} to lower frequency and also showed greater intensity compared to the free imine. The absence of an OH stretching vibration (~3440 cm^{-1}) in the complexes indicated deprotonation of OH in the Schiff base upon complex formation, suggesting the formation of a Co–O coordination bond. The C–O stretching vibrations in the crown ether ring for the complexes were at almost the same frequencies as for the free Schiff bases. These facts suggest that the cobalt atom only interacts with the Ar–OH and CH=N groups [23]. The observed molar conductances of the complexes in DMF solution

(1.0×10^{-3} mol L^{-1}) at 25 °C showed that they are non-electrolytes [24]. The molar magnetic susceptibility χ_M and magnetic moment μ_m values indicated that the cobalt has three non-paired electrons in all of these complexes and is therefore divalent. The ESI–MS mass spectra and elemental analysis of the complexes indicated that HL^1–HL^6 all form complexes of stoichiometry CoL_2. We therefore suggest a structural formula for these Schiff base cobalt complexes as shown in Fig. 1.

Dioxygen affinities of the complexes

The equilibrium constants (KO_2) and thermodynamic parameters ΔH^0 and ΔS^0 for oxygenation of complexes CoL_2^1–CoL_2^6

Table 1 Equilibrium constants and thermodynamic parameters for oxygenation of CoL_2

Complex	B	T (°C)	Ions added	ln KO_2 (mm^{-1})	ΔH^0 (kJ mol^{-1})	ΔS^0 (J K^{-1}mol^{-1})
CoL_2^1	Py	−5	/	−3.53	−31.63	−147.36
		0	/	−3.79		
		10	/	−4.28		
		25	/	−4.96		
		10	$LiNO_3$	−4.14		
		10	$NaNO_3$	−3.21		
		10	KNO_3	−4.08		
CoL_2^2	Py	−5	/	−3.68	−29.03	−138.92
		0	/	−3.92		
		10	/	−4.37		
		25	/	−4.99		
		10	$LiNO_3$	−4.31		
		10	$NaNO_3$	−3.51		
		10	KNO_3	−4.25		
CoL_2^3	Py	−5	/	−3.76	−28.25	−136.65
		0	/	−3.99		
		10	/	−4.43		
		25	/	−5.04		
		10	$LiNO_3$	−4.35		
		10	$NaNO_3$	−3.61		
		10	KNO_3	−4.75		
CoL_2^4	Py	−5	/	−5.31	−23.98	−133.64
		0	/	−5.51		
		10	/	−5.88		
		25	/	−6.39		
		10	$LiNO_3$	−5.78		
		10	$NaNO_3$	−5.70		
		10	KNO_3	−5.76		
CoL_2^5	Py	−5	/	−5.34	−23.20	−130.96
		0	/	−5.53		
		10	/	−5.89		
		25	/	−6.39		
		10	$LiNO_3$	−5.83		
		10	$NaNO_3$	−5.76		
		10	KNO_3	−5.80		
CoL_2^6	Py	−5	/	−5.40	−22.68	−129.55
		0	/	−5.59		
		10	/	−5.94		
		25	/	−6.42		
		10	$LiNO_3$	−5.86		
		10	$NaNO_3$	−5.82		
		10	KNO_3	−5.85		

are listed in Table 1. The data indicate that the dioxygen affinities of these complexes are influenced greatly by both the temperature and the structure of the Schiff base ligand. Higher temperature results in a smaller oxygenation constant. This may be due to the reduced solubility of O_2 at higher temperatures. Figure 2 shows the effect of temperature on the oxygen uptake of CoL^1_2, which is a representative example of oxygen uptake by these complexes at different temperatures. As shown in Fig. 2, the maximum oxygen uptakes of CoL^1_2 are 0.83, 0.79, 0.75 and 0.69 at −5, 0, 10 and 25 °C, respectively. This suggests that the complex forms a limiting 1:1 adduct with O_2 [25], similar to other reported examples [9]. A plot of $\ln KO_2$ versus $1/T$ is shown in Fig. 3. There is a good linear relationship ($R^2 > 0.99$), which suggests that the 1:1 adducts were almost completely formed [26]. The dioxygen affinities of the complexes decrease in the order $CoL^1_2 > CoL^2_2 > CoL^3_2$ or $CoL^4_2 > CoL^5_2 > CoL^6_2$ under the same conditions. The results can be attributed to the effects of substituent, which could include both steric and electronic effects. Although the steric effect seems likely to be the more important, the electronic effect of electron-donating groups can also enhance the electron density of the cobalt atom and so facilitate the formation of the Co–O_2 complex [8, 27]. Therefore, the dioxygen affinities of the complexes which are substituted by an electron-donating group (–CH_3) on the aromatic ring are higher than those containing electron-withdrawing groups (–Cl or –NO_2). The three complexes CoL^1_2, CoL^2_2 and CoL^3_2 containing an aza-crown pendant group show much more enhanced dioxygen affinities than CoL^4_2, CoL^5_2 and CoL^6_2 which possess a morpholino pendant. This observation can be attributed to the macrocycle effect of the crown rather than the electron-donating effect of the nitrogen methylene (NCH_2) on the aromatic ring of the ligand, because the crown ring offers a hydrophobic microenvironment and probably favours the oxygen molecule's approach to the central cobalt atom [28]. In addition, the aza-crown pendants offer larger steric hindrance around these Schiff base complexes than the morpholino pendants. This may help to prevent the aza-crown pendant complexes from dimerizing and so losing activity. On comparing with other, Schiff base Co(II) complexes reported in the literature [29], we note that the dioxygen affinities of these complexes are also influenced by the position of aromatic substituent. The oxygenation constants of Schiff base complexes of 3-substituted groups (benzo-10-aza-crown ether or morpholino) are bigger than those of 5-substituted groups. Although the steric hindrance of 3-substituents is larger than that of 5-substituents, the hydrophobic microenvironment of the complexes of 3-substituted groups is likely to be superior to that of 5-substituted groups, since the 3-substituted benzo-10-aza-crown ring is closer to the metal coordination centre compared to the 5-substituted analogue. These results indirectly illustrate that the dioxygen affinities of these complexes are not controlled by a single factor but by the integrated effect of several factors.

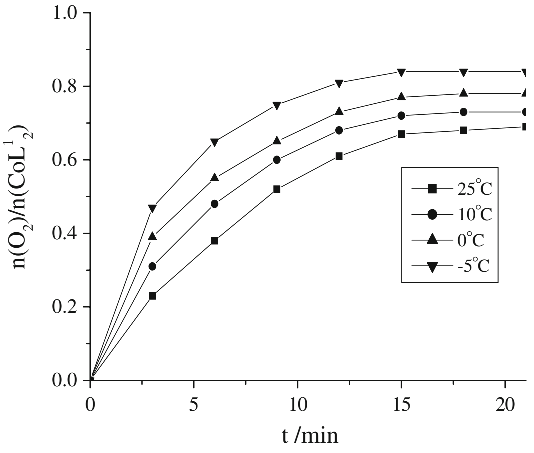

Fig. 2 The effect of temperature on oxygenation performance of CoL^1_2 $n(O_2)/n(CoL^1_2)$ means mol O_2 absorbed per mol of complex CoL^1_2, maximum oxygen uptake means mol O_2 absorbed per mol of complex CoL^1_2 at oxygenation equilibrium

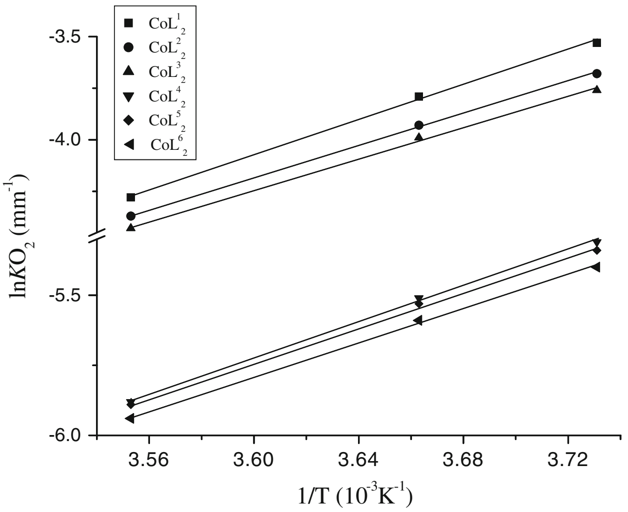

Fig. 3 The plot of $\ln KO_2$ versus $1/T$ at the range of −5 to +10 °C

It is interesting that the addition of alkali metal salts (metal nitrate/CoL_2 = 2:1) to the reaction system can enhance the O_2-binding capability of CoL^1_2, CoL^2_2 and CoL^3_2 but not of CoL^4_2, CoL^5_2 and CoL^6_2. This suggests that the alkali metal cations improve the O_2-binding capability only when they are coordinated within the crown ring of the complexes. Table 1 indicates that the KO_2 of complexes containing aza-crown pendants is significantly enhanced by adding excess $NaNO_3$. However, the enhancement obtained by adding an equal quantity of $LiNO_3$ or KNO_3 is very limited. This may be because the Li^+ ion (d = 1.36 Å) is too small to match the cavity size of aza-15-crown-5

($d = 1.7$–2.2 Å), whereas the K^+ ion ($d = 2.66$ Å) is too large. The Na^+ ion ($d = 1.90$ Å) matches well with the cavity size of aza-15-crown-5 [30] and so facilitates the formation and stabilization of the Co-O_2 complex.

Conclusions

Six Schiff base Co(II) complexes with aza-crown or morpholino pendants were synthesized. The presence of a pendant crown ether group in the Schiff base ligand significantly improves the O_2-binding capabilities of the complexes. The addition of Na^+ cations enhances the O_2-binding capability of CoL^1_2, CoL^2_2 and CoL^3_2 but not of CoL^4_2, CoL^5_2 and CoL^6_2. These results are explained in terms of the macrocyclic effect of the benzoaza-crown ether ring, which enlarges the steric hindrance around the Co(II) centre, and so prevents the complexes form being oxidized to the μ-oxodimer and losing activity. The dioxygen affinity of these complexes is influenced by several factors, including the temperature, the electronic proprieties of the substituent on the aromatic ring of the Schiff base ligand and the macrocyclic effect of the crown ring.

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