Synthesis of *N*-(4-Salicylideneiminoaryl)monoaza Crown Ethers and Dioxygen Affinities of Their Cobalt(II) Complexes

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The Schiff base-containing pendant monoaza crown ether HL^1, HL^2, HL^3 and HL^4 have been synthesized by condensation of salicylaldehyde with *N*-(4-aminoaryl)monoaza crown ethers, which were prepared conveniently from 4-nitro-*N*, *N*-di(hydroxyethyl) aniline or 4-nitrobenzyl chloride via cyclization or condensation and reduction. The structures of HL^1—HL^4 were verified by ^1H NMR, IR spectra, MS and elemental analysis. Moreover, the oxygenation constants (K_{O_2}) and thermodynamic parameters (ΔH^0 and ΔS^0) of their cobalt(II) complexes were determined in the range of −5 ℃ to 25 ℃, and the effect of crown ring bonded to a Schiff base on the dioxygen affinities of cobalt(II) complexes was also observed as compared to the uncrowned analogue (CoL_2^5).

Keywords synthesis, monoaza crown ether, cobalt(II)-Schiff base complex, oxygenation

Introduction

Schiff bases and their metal complexes have been applied widely in the field of catalytic chemistry, liquid crystals and photochromism.[1-3] Many Schiff base ligands containing crown ether possess different recognition sites for both alkali and transition metal guest cations,[4] for example, the Na(I) or K(I)/Co(II) hetero-nuclear complexes of crowned Schiff base can bind oxygen molecule (O_2) to form stable solid dioxygen adducts.[5-7] Gebbink and co-workers have reported a K(I)/Cu(II) sandwich complex which can accelerate the formation of dioxygen adducts.[8] Recently we have reported that benzo crowned Schiff base complexes showed significantly improved dioxygen affinities and biomimetic catalytic activity compared to uncrowned analogues.[9]

Since the aza crown ether ring appended Schiff base complexes can be close to the coordination center, and control more efficiently the coordination environment, we designed and synthesized *N*-pivot lariat ethers with Schiff base as side arm, and expected their cobalt(II) complexes to have enhanced dioxygen affinities.

Results and discussion

The synthetic route of compounds HL^1—HL^4 is shown in Scheme 1. Condensation reaction of 4-nitro-*N*, *N*-di(hydroxyethyl) aniline (**1**) with 1.1 equiv. of diethyleneglycol ditosylate or triethyleneglycol ditosylate in the presence of NaH and THF yielded the desired monoaza crown ether **2a** (79%) or **2b** (76%) respectively. Reduction of **2a** and **2b** were accomplished with Pd/C (10%) in EtOH at 50 ℃ to provide **3a** (98%) and **3b** (95%), respectively. 4-Nitrobenzyl chloride (**4**) was treated with monoaza crown ether and 2 equiv. of Na_2CO_3 in CH_3CN under reflux to give **5a** (93%) and **5b** (94%). We tried to reduce the nitro group of **5a** and **5b** to form the desired **6a** and **6b** by catalytic hydrogenation using Pd/C (10%) as the catalyst. Unfortunately, no desired products were obtained because the monoaza crown ether was removed during hydrogenation. Reduction of **5a** and **5b** was carried out in the presence of 1.3 equiv. of $SnCl_2 \cdot 2H_2O$, EtOAc and conc. HCl to produce **6a** (80%) and **6b** (85%), respectively. Finally, the *N*-(4-aminoaryl) monoaza crown ethers **3a**, **3b**, **6a** and **6b** were treated with 1.0 equiv. of salicylaldehyde under N_2 atmosphere followed by recrystallization or silica-gel chromatography using CH_3OH as eluent to afford target compounds HL^1—HL^4.

In the IR spectra of **HL**, the characteristic frequency of CH = N at 1620—1618 cm^{-1} was shown, in the case of CoL_2 (see Scheme 2), the characteristic vibration of CH = N at 1614—1610 cm^{-1} was shown. Meanwhile, the characteristic vibration of OH at 3225—3220 cm^{-1} disappeared. Moreover, the elemental analysis of CoL_2 indicated the formation of 1:2 (Co/HL) Cobalt(II) Schiff base complexes. The crystal structures of HL^2 and CoL_2^2 are presented in Figs. 1 and 2, respectively, and their crystallographic data will be published in due course.

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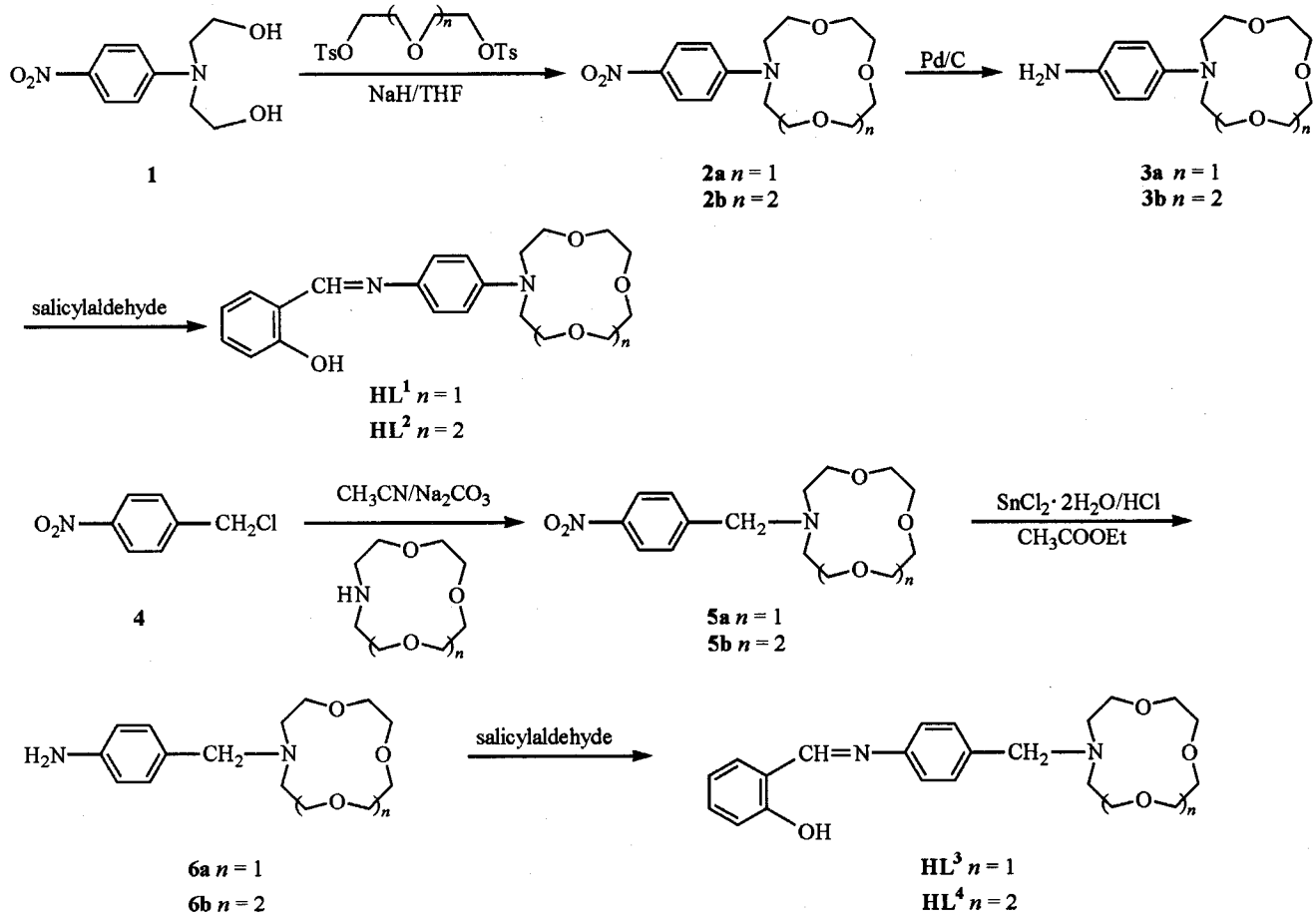
Scheme 1



1

2a $n = 1$
2b $n = 2$

3a $n = 1$
3b $n = 2$

HL^1 $n = 1$
HL^2 $n = 2$

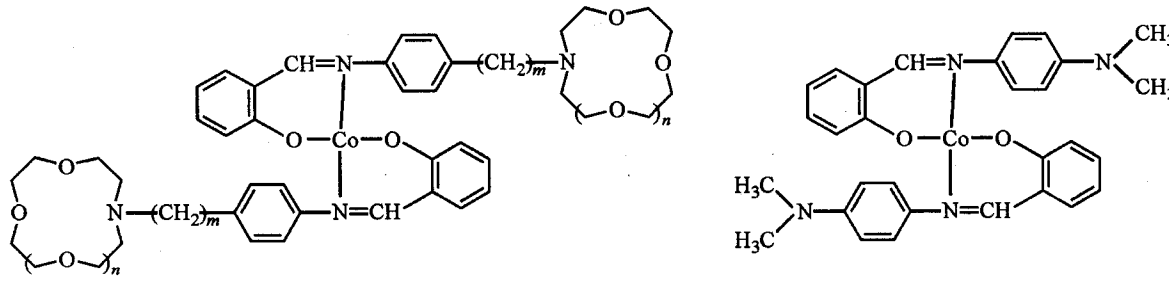
4

5a $n = 1$
5b $n = 2$

6a $n = 1$
6b $n = 2$

HL^3 $n = 1$
HL^4 $n = 2$

Scheme 2



CoL_2^1 $n = 1, m = 0$
CoL_2^2 $n = 2, m = 0$
CoL_2^3 $n = 1, m = 1$
CoL_2^4 $n = 2, m = 1$
CoL_2^5

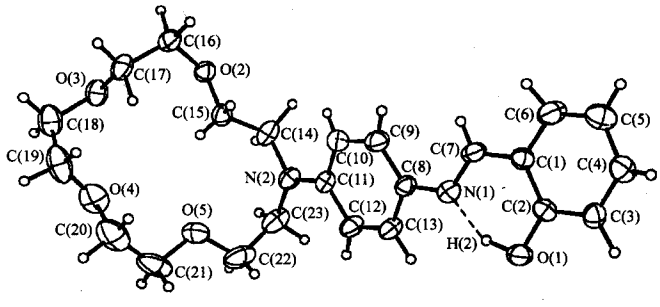


Fig. 1 Crystal structure of HL^2.

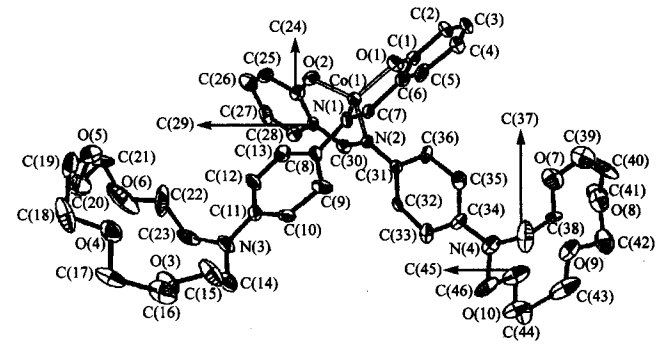


Fig. 2 Crystal structure of CoL_2^2.

The oxygenation constants (K_{O_2}) and thermodynamic parameters (ΔH^0 and ΔS^0) of cobalt(II) crowned Schiff base complexes CoL_2^1—CoL_2^4 and uncrowned analogue CoL_2^5 derived from *N*,*N*-dimethyl-[4-(2-hydroxybenzylideneimino)] aniline[10] are listed in Table 1. As illustrated in Table 1, the dioxygen affinities of cobalt(II) complexes were influenced greatly by substitutent located in aromatic ring bonded to nitrogen atom of CH = N. The oxygenation constants (K_{O_2}) of CoL_2^1—CoL_2^4 with aza crown ring are bigger than that of uncrowned analogue (CoL_2^5). The fact should be due to the macrocycle effect of crown ring instead of their electron-donating effect, because crown ring with special configuration will probably favor oxygen molecule to approach coordination center of cobalt(II) complexes and stabilize the bond of Co—O_2 for its hydrophobicity of outer ethylene group and orderly arrangement of inner oxa atom.[11] The K_{O_2} of CoL_2^1 is similar to that of CoL_2^2, and K_{O_2} of CoL_2^3 is similar to that of CoL_2^4. The fact indicates that the size of aza-12-crown-4 and aza-15-crown-5 has little effect on the oxygenation performance of CoL_2^1—CoL_2^4. But the oxygenation constants (K_{O_2}) of CoL_2^1 and CoL_2^2 are bigger than that of CoL_2^3 and CoL_2^4. It should be obviously due to the presence of the methylene (—CH_2—) between the aromatic ring and aza crown ether, which makes aza crown ring further away from the coordination center of CoL_2^3 and CoL_2^4 as compared to CoL_2^1 and CoL_2^2, and results in a poor ability for aza crown ether to control efficiently the coordination environment.

Table 1 Oxygenation constants and thermodynamic parameters (ΔH^0 and ΔS^0) of CoL_2

Complex	B	T (℃)	ln K_{O_2} (mm^{-1})	ΔH^0 ($kJ \cdot mol^{-1}$)	ΔS^0 ($J \cdot K^{-1} \cdot mol^{-1}$)
		-5	-4.20		
CoL_2^1	Py	0	-4.54	-43.37	-233.66
		15	-5.53		
		25	-6.16		
		-5	-4.22		
CoL_2^2	Py	0	-4.56	-40.85	-228.19
		15	-5.49		
		25	-6.06		
		-5	-4.89		
CoL_2^3	Py	0	-5.15	-31.82	-200.07
		15	-5.88		
		25	-6.33		
		-5	-4.95		
CoL_2^4	Py	0	-5.18	-28.57	-188.44
		15	-5.84		
		25	-6.24		
		-5	-5.62		
CoL_2^5	Py	0	-5.78	-19.25	-159.21
		15	-6.22		
		25	-6.48		

Experimental

Melting points were determined on a Yanaco MP-500 micro-melting point apparatus and uncorrected. IR spectra were recorded on a Nicolet-1705X IR spectrometer. ^{1}H NMR spectra were recorded on a Bruker AC-200 MHz spectrometer using tetramethylsilane as internal standard. Mass spectra were obtained on a Finnigan MAT 4510 spectrometer. Elemental analysis was performed on a Carlo Erbo-1160 elemental analyzer. Silica gel (60H for TLC, Qingdao, China) was used for flash column chromatography. Compound 4-nitro-*N*,*N*-di(hydroxyethyl)aniline (**1**),[12] diethyleneglycol ditosylate and triethyleneglycol ditosylate,[13] aza-12-crown-4 and aza-15-crown-5[14] were synthesized according to reported procedures. All other reagents were of analytical grade and were used without further purification.

Synthesis of N-(4-salicylideneiminoaryl) monoaza crown ethers (**HL**)

N-(4-Nitrophenyl) monoaza-12-crown-4 (**2a**)

A 1-L three-necked flask was purged with N_2, NaH (1.86 g, 77.5 mmol) was added to the reaction vessel and washed with hexane (4 × 50 mL). THF (300 mL) was then added to the flask. This suspension was heated to reflux with vigorous stirring. A solution of compound **1** (16.95 g, 75 mmol) and diethyleneglycol ditosylate (31.05 g, 75 mmol) in THF (300 mL) was added dropwise. Reflux was continued for 20 h. The reaction mixture was cooled and quenched with H_2O, and the solvent was evaporated *in vacuo*. The residue was dissolved in H_2O (400 mL), which was extracted with CH_2Cl_2 (3 × 200 mL). The combined organic layers were reduced to a minimum volume. The pure product was obtained as yellow crystal (17.75 g, 79%) after chromatography (silica gel, CH_3COOEt). M.p. 114—116 ℃; ^{1}H NMR ($CDCl_3$) δ: 6.75—6.70 (m, 4H, ArH), 3.85—3.65 (m, 12H, 3 × CH_2OCH_2), 3.58 (m, 4H, 2 × NCH_2); IR (KBr, film) ν_{max}: 1600, 1340, 1126 cm^{-1}; MS *m/z*: 296 (M^+). Anal. calcd for $C_{14}H_{20}N_2O_5$: C 56.76, H 6.76, N 9.46; found C 56.51, H 6.89, N 9.28.

N-(4-Nitrophenyl) monoaza-15-crown-5 (**2b**)

The compound **2b** was prepared using a method similar to that for **2a**. Yield 75.7%, m.p. 127—130 ℃ (lit.[15] 127—130 ℃).

N-(4-Aminophenyl) monoaza-12-crown-4 (**3a**)

To a suspension of 0.10 g Pd/C (10%) in 50 mL of ethanol was added **2a** (0.5 g, 1.69 mmol), and the mixture was hydrogenated under H_2 at 50—55 ℃ for 20 h, then the reaction mixture was cooled and filtered. The filtrate was e-

vaporated to dryness and chromatographed (silica gel, CH_3OH) to give a slightly reddish oil (0.44 g, 98%). ^1H NMR ($CDCl_3$) δ: 6.64—6.55 (m, 4H, ArH), 5.30 (s, 2H, NH_2), 3.84—3.65 (m, 12H, 3 × CH_2OCH_2), 3.62—3.58 (m, 4H, 2 × NCH_2); IR (neat) ν_{max}: 3300, 1601, 1158 cm^{-1}; MS *m/z*: 266 (M^+). Anal. calcd for $C_{14}H_{22}N_2O_3$: C 63.16, H 8.27, N 10.53; found C 62.95, H 8.41, N 10.78.

N-(4-*Aminophenyl*) *monoaza*-15-*crown*-5 (**3b**)

The compound **3b** was prepared using a method similar to that for **3a**. Yield 95%, m.p. 44—45 ℃ (lit.[15] 46 ℃); ^1H NMR ($CDCl_3$) δ: 6.63—6.55 (m, 4H, ArH), 5.30 (s, 2H, NH_2), 3.74—3.63 (m, 16H, 4 × CH_2OCH_2), 3.61—3.57 (m, 4H, 2 × NCH_2); MS *m/z*: 310 (M^+).

N-[4-(*Salicylideneiminophenyl*)] *monoaza*-12-*crown*-4 (**HL**[1])

The compound **3a** (0.267 g, 1.0 mmol) was dissolved in 6 mL of MeOH. This solution was then mixed with 0.1 mmol of salicylaldehyde (0.122 g, 1.0 mmol) and stirred under N_2 for 2 h. The yellow separated precipitate was collected off and washed with water and ethanol, and after recrystallization from ethanol, yellow crystal (0.29 g, 78%) was obtained. M.p. 92—94 ℃; ^1H NMR ($CDCl_3$) δ: 13.76 (s, 1H, OH), 8.62 (s, 1H, CH = N), 7.36—7.23 (m, 4H, ArH), 7.00—6.76 (m, 4H, ArH), 3.89—3.60 (m, 16H, OCH_2 and NCH_2); IR (KBr, film) ν_{max}: 3225, 1619, 1120 cm^{-1}; MS *m/z*: 370 (M^+). Anal. calcd for $C_{21}H_{26}N_2O_4$: C 68.11, H 7.03, N 7.56; found C 68.23, H 6.95, N 7.39.

N-[4-(*Salicylideneiminophenyl*)] *monoaza*-15-*crown*-5 (**HL**[2])

This compound **HL**[2] was prepared using a method similar to that for **HL**[1]. Yield 79.5%, m.p. 97—100 ℃. ^1H NMR ($CDCl_3$) δ: 13.80 (s, 1H, OH), 8.60 (s, 1H, CH = N), 7.36—7.24 (m, 4H, ArH), 7.00—6.68 (m, 4H, ArH), 3.80—3.60 (m, 20H, OCH_2 and NCH_2); IR (KBr, film) ν_{max}: 3220, 1619, 1120 cm^{-1}; MS *m/z*: 414 (M^+). Anal. calcd for $C_{23}H_{30}N_2O_5$: C 66.67, H 7.25, N 6.76; found C 66.95, H 7.48, N 6.49.

N-(4-*Nitrobenzyl*) *monoaza*-12-*crown*-4 (**5a**)

Monoaza-12-crown-4 (1.59 g, 9 mmol), Na_2CO_3 (1.91 g, 18 mmol), CH_3CN (50 mL), and **4** (1.54 g, 9 mmol) were stirred under reflux for 24 h, cooled, and filtered, and the solvent was evaporated *in vacuo*. The residue was dissolved in $CHCl_3$ (20 mL) and filtered, and the solvent was evaporated. The residual oil was chromatographed (silica gel, CH_3COOEt) to provide pure **5a** as a yellow oil (2.60 g, 93%). ^1H NMR ($CDCl_3$) δ: 6.78—6.70 (m, 4H, ArH), 3.75—3.60 (m, 16H, OCH_2 and NCH_2), 2.85 (t, J = 5 Hz, 2H, CH_2); IR (neat) ν_{max}: 1610, 1345, 1120 cm^{-1}; MS *m/z*: 310 (M^+). Anal. calcd for $C_{15}H_{22}N_2O_5$: C 58.06, H 7.10, N 9.03; found C 58.32, H 6.92, N 8.97.

N-(4-*Nitrobenzyl*) *monoaza*-15-*crown*-5 (**5b**)

Compound **5b**[16] was prepared as described for **5a**. The crude product was chromatographed (silica gel, CH_3COOEt) to give yellow oil, 94% yield. ^1H NMR ($CDCl_3$) δ: 6.78—6.71 (m, 4H, ArH), 3.71—3.64 (m, 20H, NCH_2 and OCH_2), 2.84 (t, J = 6 Hz, 2H, CH_2); MS *m/z*: 354 (M^+).

N-(4-*Aminobenzyl*) *monoaza*-12-*crown*-4 (**6a**)

A solution of compound **5a** (2.79 g, 9 mmol), $SnCl_2 \cdot 2H_2O$ (2.5 g, 11.06 mmol), CH_3COOEt (10 mL), and 2.95 mL of HCl (37%) was stirred at 40 ℃ for 40 min, followed by the addition of 20 mL of H_2O. After being stirred at the same temperature for 1 h, the mixture was adjusted to pH = 8—9 with 40% NaOH, filtered and extracted with CH_2Cl_2 (3 × 30 mL), dried with $MgSO_4$, evaporated *in vacuo* to dryness and chromatographed (silica gel, CH_3OH) to give oil (2 g, 80%). ^1H NMR ($CDCl_3$) δ: 6.65—6.58 (m, 4H, ArH), 5.30 (s, 2H, NH_2), 3.74—3.62 (m, 16H, OCH_2 and NCH_2), 2.85 (t, J = 5 Hz, 2H, CH_2); IR (neat) ν_{max}: 3300, 1128, 1029 cm^{-1}; MS *m/z*: 280 (M^+). Anal. calcd for $C_{15}H_{24}N_2O_3$: C 64.29, H 8.57, N 10.00; found C 64.43, H 8.27, N 10.23.

N-(4-*Aminobenzyl*) *monoaza*-15-*crown*-5 (**6b**)

Compound **6b** as oil was prepared as described for **6a**, 85% yield. ^1H NMR ($CDCl_3$) δ: 6.65—6.57 (m, 4H, ArH), 5.30 (s, 2H, NH_2), 3.70—3.63 (m, 20H, NCH_2 and OCH_2), 2.85 (t, J = 6 Hz, 2H, CH_2); IR (neat) ν_{max}: 3300, 1125, 1022 cm^{-1}; MS *m/z*: 324 (M^+). Anal. calcd for $C_{17}H_{28}N_2O_4$: C 62.96, H 8.64, N 8.64; found C 62.72, H 8.74, N 8.39.

N-[4-(*Salicylideneiminobenzyl*)] *monoaza*-12-*crown*-4 (**HL**[3])

The compound **6a** (0.28 g, 1.0 mmol), ethanol (10 mL), and salicylaldehyde (0.12 g, 1.0 mmol) were stirred under N_2 for 2 h, then the mixture was reduced to a minimum volume, and was chromatographed (silica gel, CH_3COOEt) to give yellow oil (0.35 g, 91.1%). ^1H NMR ($CDCl_3$) δ: 13.35 (s, 1H, OH), 8.64 (s, 1H, CH = N), 7.50—7.20 (m, 4H, ArH), 7.00—6.90 (m, 4H, ArH), 3.75—3.63 (m, 16H, OCH_2 and NCH_2), 2.81 (t, J = 5 Hz, 2H, CH_2); IR (neat) ν_{max}: 3221, 1620, 1132 cm^{-1}; MS *m/z*: 384 (M^+). Anal. calcd for $C_{22}H_{28}N_2O_4$: C 68.75, H 7.29, N 7.29; found C 69.02, H 7.10, N

7.52.

N-[4-(*Salicylideneiminobenzyl*)]*monoaza*-15-*crown*-5 (**HL^4**)

Compound **HL^4** was prepared as described for **HL^3**. The crude mixture was chromatographed (silica gel, CH_3COOEt) to give yellow oil, 90.5% yield. ^1H NMR ($CDCl_3$) δ: 13.34 (s, 1H, OH), 8.62 (s, 1H, CH=N), 7.42—7.21 (m, 4H, ArH), 7.00—6.90 (m, 4H, ArH), 3.70—3.63 (m, 20H, OCH_2 and NCH_2), 2.81 (t, J=6 Hz, 2H, CH_2); IR (neat) ν_{max}: 3220, 1618, 1128 cm^{-1}; MS m/z: 429 (M^+ + 1). Anal. calcd for $C_{24}H_{32}N_2O_5$: C 67.29, H 7.48, N 6.54; found C 67.53, H 7.19, N 6.75.

General methods for Synthesis of cobalt(II) Schiff base complexes (CoL_2)

A solution of **HL** (2.0 mmol) and $Co(OAc)_2 \cdot 4H_2O$ (1.1 mmol) in EtOH (15 mL) was stirred in EtoH for 2 h under N_2 at 70 ℃, then the mixture was cooled and filtrated, washed with methanol to give transition-metal complexes, and the pure product was obtained after recrystallization from ethanol (95%).

CoL_2^1: dark brown, 51% yield, m.p. 254—256 ℃; IR (KBr, film) ν_{max}: 1612, 1122 cm^{-1}. Anal. calcd for $CoC_{42}H_{50}N_4O_8$: C 63.24, H 6.27, N 7.03; found C 63.31, H 6.19, N 6.94.

CoL_2^2: dark brown, 53% yield, m.p. 203—206 ℃; IR (KBr, film) ν_{max}: 1610, 1123 cm^{-1}. Anal. calcd for $CoC_{46}H_{58}N_4O_{10}$: C 62.37, H 6.55, N 6.32; found C 62.45, H 6.49, N 6.28.

CoL_2^3: dark brown, 50% yield, m.p. 268—270 ℃; IR (KBr, film) ν_{max}: 1614, 1124 cm^{-1}. Anal. calcd for $CoC_{44}H_{54}N_4O_8$: C 64.00, H 6.55, N 6.79; found C 63.89, H 6.61, N 6.84.

CoL_2^4: dark brown, 43% yield, m.p. 228—231 ℃; IR (KBr, film) ν_{max}: 1610, 1126 cm^{-1}. Anal. calcd for $CoC_{48}H_{62}N_4O_{10}$: C 63.09, H 6.79, N 6.13; found C 63.14, H 6.88, N 6.07.

Oxygen uptake measurements

The oxygenation equilibrium constants were determined by the method of Chen[17] in the presence of diethyleneglycol dimethyl ether as solvent and pyridine as axial ligand (B). The concentration of the complex was 5×10^{-3} mol·dm^{-3} and the partial pressure of dioxygen was 97 kPa. The equilibrium constants (K_{O_2}) were calculated as follows.

$$CoL_2 + B \rightleftharpoons CoL_2B \qquad CoL_2B + O_2 \rightleftharpoons CoL_2BO_2$$

$$K_{O_2} = \frac{[CoL_2BO_2]}{[CoL_2B] \cdot p_{O_2}}$$

B, p_{O_2} represent axial base and partial pressure of oxygen respectively. Thermodynamic parameters ΔH^0, ΔS^0 were determined from variation of K_{O_2} and K'_{O_2} over a range of temperature.

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