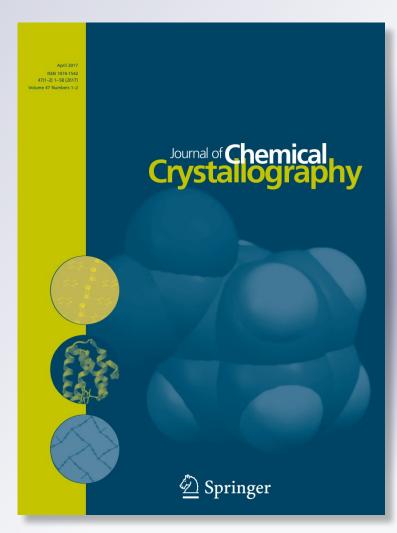
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Crystal Structure and Corrosion Inhibition Properties of Ferrocenyl- and Phenylendiamine-Iminomethylphenoxy Cobalt Complexes

Nadra Belhadj $^{1,2}\cdot$ Ali Ourari $^1\cdot$ Saida Keraghel $^1\cdot$ Bernd Schöllhorn $^3\cdot$ Dominique Matt 4

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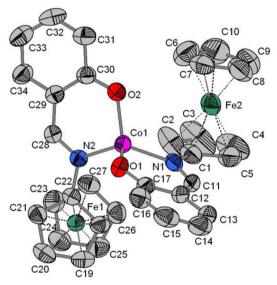
Abstract The crystal structures of bis[2-(ferrocenyl- $(Co^{II}(L))$ iminomethyl)phenoxy]cobalt(II) [monoclinic, a = 12.5466(3) Å, b = 10.6782(3) Å, c = 21.2695(6) \dot{A} , $\alpha = \gamma = 90^{\circ}$, $\beta = 92.944(2)^{\circ}$, V = 2845.82(13) \dot{A}^{3} , Z=4, space group $P2_1/c$] and bis(2-[(4-dimethylamino-phenylimino)-methyl]-phenoxy) cobalt (II) $(Co^{II}(M)_2)$ [triclinic, a=10.2916(4) Å, b=16.4867(10) Å, c = 17.6782(11) Å, $\alpha = 114.754(2)^{\circ}$, $\beta = 96.614(4)^{\circ}$, $\gamma = 97.736(4)$, Z=4, space group P-1] dominated by extensive hydrogen bonding such as O-H...N, N-H...O and N-H...N interactions. In both structures the central CoII is displaying a slightly distorted tetrahedral coordination sphere involving two iminoethyl-phenoxy ligands. The inhibition efficiency of the Co complexes concerning the corrosion of mild steel in acidic solution has been investigated by electrochemical impedance spectroscopy.

Graphical Abstract The crystal structures of two bis(iminomethylphenoxy) cobalt complexes containing

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respectively a ferrocenyl and a phenylendiamine group are described. First results on their inhibition properties concerning the corrosion of mild steel in hydrochloric acid are reported and discussed.



Bis[2-(ferrocenyl-iminomethyl)phenoxy]cobalt(II)

Keywords Crystal structure · Cobalt · Schiff base · Ferrocene · Phenylenediamine · Corrosion inhibition · Mild steel · Electrochemical impedance spectroscopy

Introduction

Schiff bases are important ligands for transition metals leading to coordination compounds and materials with interesting catalytic, redox, optical and magnetic properties [1]. Metal complexes based on iminomethylphenoxy ligands such as L and M depicted in Scheme 1 revealed properties for potential applications regarding corrosion inhibition [2], antitumor activity [3], uranium(VI) [4, 5] and fluoride [6] receptors, for electroluminescence in organic light-emitting diodes [7, 8] and catalysts [9, 10]. In this work we describe the crystal structure of the cobalt complexes $Co^{II}(L)_2$ and $Co^{II}(M)_2$. The synthesis of both compounds has been published previously but no X-ray diffraction data have been reported so far [11, 12]. First results on the respective inhibition efficiency concerning the corrosion of mild steel in 1 M hydrochloric acid are discussed.

Experimental

General

Ferrocenyl amine or p-N,N'-dimethylamino-aniline were purchased respectively from ICE (Japan) and ACROS (France). ¹H NMR spectra were recorded on a Brucker AC 250 spectrometer in deuterated chloroform (CDCl₃). Chemical shifts were reported using TMS signals as internal reference. IR (ATR) spectra were measured on a IRAffinity-1CE.SHIMADZU spectrophotometer and UV–Visible spectra were measured in 1 cm quartz cells using a Spectroscan 50.

Synthesis

The Schiff bases L [13] and M [14] were prepared via condensation of salycilic aldehyde with the corresponding amine (ferrocenyl amine or p-N,N'-dimethylaminoaniline) according to a previously published method [15]. 2-(((ferrocenyl)imino)methyl)-phenol (L): Red crystals; yield 97%; IR (KBr, v/cm⁻¹) : 3400 (O–H), 1658 (C=N), 1199 (C–N). UV–Vis: (ethanol, λ/nm , $\epsilon/cm^{-1}.M^{-1}$) : 500 (1403), 385 (3673), 350 (2807), 310 (28,173), 275 (4538). ¹H NMR (CDCl₃, 250 MHz) : δ/ppm 8.56 (s, 1 H, N=CH), 7.20–7.32 (m, 3 H), 6.92 (d, 1 H, J=7 Hz), 6.84 (t, 1 H, J=7 Hz), 4.53 (t, 2 H, J=1.8 Hz, Cp-BB'), 4.22 (t, 2 H, J=1.8 Hz, Cp-AA'), 4.12 (s, 5 H, Cp). 2-(((4-(dimethylamino)phenyl)imino)methyl)phenol (M): Bright yellow crystals; yield 80%; IR (KBr, v/cm⁻¹): 3445 (O-H), 1623 (C=N), 1355 (C–N). UV-Vis: (ethanol, λ/nm , $\epsilon/cm^{-1} M^{-1}$): 385 (11,151), 295 (1863), 245 (1851), 230 (6893). ¹H NMR (CDCl₃, 250 MHz) : δ/ppm8.55 (s, 1 H, N=CH), 7.18-7.32 (m, 4 H), 6.92 (d, 1 H, 7.5 Hz), 6.84 (t, 1 H, 7.5 Hz), 6.64–6.72 (m, 2 H), 2.93 (s, 6 H, CH₃).

The cobalt(II) complexes $Co^{II}(L)_2$ and $Co^{II}(M)_2$ were prepared by a previously published template condensation method [16]. A solution containng cobalt(II)acetate (0.177 g, 1.0 mmol) and the respective ligand L or M (2 mmol) in anhydrous ethanol was refluxed for one hour under nitrogen atmosphere. The resultinging precipitate was filtered, washed with ethanol and recrystallized from chloroform/petrolether. Bis[(E)-2-(((ferrocenyl)imino) methyl)phenoxy]cobalt(II) ($Co^{II}(L)_2$): Brown crystals, yield 83%; IR (KBr, v/cm⁻¹) : 1600 (C=N), 1143 (C–N), 862 (Co–O), 736 (Co–N). UV–Vis: (ethanol, λ /nm, ϵ / cm⁻¹ M⁻¹) : 500 (2217), 325 (57,173), 280 (4478), 260 (5913). Bis[(E)-2-(((4-(dimethylamino)phenyl)imino) methyl)phenoxy] cobalt (II) ($Co^{II}(M)_2$): Red crystals, yield 75%; IR (KBr, v/cm⁻¹): 1605 (C=N), 1334 (C–N), 863 (Co–O), 671 (Co–N). UV–Vis: (ethanol, λ /nm, ϵ / cm⁻¹ M⁻¹): 393 (21,344), 330 (12,931), 294 (8586).

Single Crystal X-ray Structure Determination

The cobalt(II) complexes $Co^{II}(L)_2$ and $Co^{II}(M)_2$ were recrystallized from chloroform/petrolether yielding crystals suitable for single X-ray diffraction. Single crystals were mounted on a Nonius four-circle diffractometer equipped with a CCD camera and a graphite-monochromated MoKa radiation source ($\lambda = 0.71073$ Å), from the Centre de Diffractomtrie (CDFIX), Université de Rennes 1, France. Data were collected at 293 K. Structures were solved with direct methods using the SIR-97 program and refined with a fullmatrix least-squares method on F² using the SHELXL-97 program. (G. M. Sheldrick, Acta Crystallogr. Sect. A, 2008, 64, 112-122). All non-H atoms were refined with anisotropic displacement parameters and H atoms were simply introduced at calculated positions (riding model with isotropic temperature factors fixed at 1.2-times that of the parent atom). All crystallographic details are listed in Table 1. The molecular structures of $Co^{II}(L)_2$ and $Co^{II}(M)_2$ are shown in Fig. 1. Selected bond distances and angles for both structures are listed in Tables 2 and 3.

Electrochemical Impedance Spectroscopy

The impedance measurements were carried out using a VoltaLab GPZ 301 electrochemical analyzer with open circuit with frequencies ranging from 5×10^4 to 1×10^{-1} Hz and 10 mV amplitude in aerated 1 M hydrochloric acid at 30 °C, at various inhibitors concentrations after immersion of 30 min.

Results and Discussion

In the crystal structure of $Co^{II}(L)_2$, the cobalt atom is coordinated by two iminomethyl-phenoxy ligands (Fig. 2). The attached ferrocenyl moieties are almost perpendicular to each other (dihedral angle N1…O1…O2…N2, 96.69(10)°). The tetrahedral geometry of the cobalt coordination sphere is slightly distorted as indicated by the Co…O (ca. 1.90 Å)

Table 1 Crystal data and details of the structure determination of the cobalt complexes

	Co ^{II} (L) ₂	Co ^{II} (M) ₂	
Formula	$C_{34} H_{28} Co Fe_2 N_2 O_2$	C ₃₀ H ₃₀ Co N ₄ O ₂	
Formula weight	667.22	537.51	
CCDC reference	1,446,313	1,446,312	
Crystal system	Monoclinic	Triclinic	
Space group	P2 ₁ /c	P-1	
a (Å)	12.5466 (3)	10.2916 (4)	
b (Å)	10.6782 (3)	16.4867 (10)	
c (Å)	21.2695 (6)	17.6782 (11)	
α(°)	90.00	114.754 (2)	
β (°)	92.944 (2)	96.614 (4)	
γ (°)	90.00	97.736 (4)	
V (Å ³)	2845.82 (13)	2649.6 (3)	
Z	4	4	
$D_{calc} (g \text{ cm}^{-3})$	1.557	1.347	
$\mu(MoK_{\alpha}) (mm^{-1})$	1.617	0.682	
F(000)	1364	1124	
Crystal size (mm ³)	/	$0.1 \times 0.05 \times 0.05$	
Temperature (K)	293 (2)	293 (2)	
Radiation (Å)	ΜοΚα 0.71073	ΜοΚα 0.71073	
Theta min-max (°)	1.63–26.37	2.28-25.35	
Index ranges	-15/+15, -13/+13, -26/+26	-11/+11, -19/+19, -20/+21	
Total, unique data	11,232, 5813	14,904, 9091	
N _{ref} , N _{par}	5813, 371	9091, 667	
R, wR2, S	0.0398, 0.0972, 1.043	0.0401, 0.0925, 1.006	
	-0.453, 0.497	-0.354, 0.249	

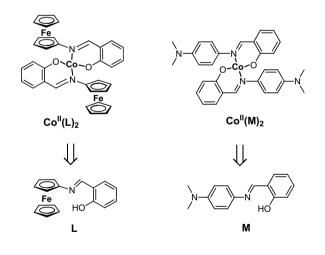


Fig. 1 Iminomethylphenoxyligands L, M and the corresponding cobalt complexes Co^{II}(L)₂ and Co^{II}(M)₂

and Co-N (ca. 1.99 Å) interatomic distances and the corresponding bond angles of X...Co...Y (X,Y=O,N) in the range of ca. 95.86 to 122.12° (see Table 2). The intramolecular intermetallic distances Co--Fe of 4.1791(6) and 4.5725(6) Å are too long for magnetic interactions [17, 18]. Interestingly there is apparently only weak delocalization of the imine double bonds with the conjugated π systems of the phenyl group and the cyclopendadienyl ring. The interatomic distance of for example N1-C11 (1.298(4) Å) is indicating an isolated double bond character contrarily to the vicinal single bonds N1-C1 (1.411(4) Å) and C11-C12 (1.429(5) Å). The crystal structure shows evidence for weak intermolecular CH---O hydrogen bonding, the H---O interatomic distance being significantly shorter than the sum of the van der Waals radii of H and O (2.72 Å) [19]: C14-H14···O1, $d_{H...O} = 2.569(2)$ Å, $\delta = 147.25(27)^{\circ}$; C21-H21...O1, $d_{H...O} = 2.656(2)$ Å, $\delta = 155.97(20);$ C28-H28--O1, $d_{H\dots O} = 2.643(2)$ Å, $\delta = 144.92(20)^{\circ}$ (Table 2). Furthermore $C-H\cdots\pi$ interactions might also support the three dimensional arrangement of the molecules: C32–H···C5 ($d_{C...C}$ ca. 3.706 Å, angle C–H to plane: probably 90°).

In the crystal structure of $Co^{II}(M)_2$, the cobalt atom is also coordinated by two iminomethyl-phenoxy ligands (Fig. 3). There are two crystallographically different complexes with an intermolecular Co1...Co2 distance of 8.9761(7) Å. In the following only the structure of the Co1 complex is discussed. As for $Co^{II}(L)_2$, the tetrahedral geometry of the cobalt coordination sphere in $Co^{II}(M)_2$ is slightly distorted with the interatomic distances Co--O

Table 2 Selected inter- atomic distances (Å) and bond angles (°), $C_{34}H_{28}CoFe_2N_2O_2$ $(Co^{II}(L)_2)$							
	Cobalt environment						
	Co1…Fe1	4.5725 (6)					
	Co1…Fe2	4.1791 (6)					
	Co1–O1	1.904 (2)			O1-Co1-N1	95.89 (11)	
	Co1–N1	2.003 (3)			O1-Co1-O2	119.80 (10)	
	Co1–O2	1.895 (2)			O2-Co1-N2	96.69 (10)	
	Co1-N2	1.981 (3)			N1-Co1-N2	122.14 (11)	
	Imine double bo	ond					
	N1-C11	1.298 (4)	N2-C28	1.302 (4)			
	N1-C1	1.411 (4)	N2-C22	1.427 (4)			
	C11-C12	1.429 (5)	C28-C29	1.437 (4)			
	C-H-O interac	tions					
	H14…O1	2.569 (2)	C14…O1	3.389 (5)	C14-H14…O1	147.25 (27)	
	H21…O1	2.656 (2)	C21O1	3.526 (4)	C21-H21O1	155.97 (20)	
	H28…O1	2.643 (2)	C28…O1	3.445 (4)	C28-H28-01	144.92 (20)	

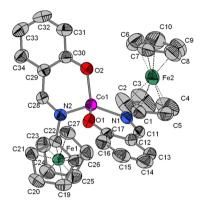


Fig. 2 Molecular structure of Co^{II}(L)₂. 50% percent probability for the thermal displacement ellipsoids

and Co---N being respectively ca. 1.90 Å and ca. 1.99 Å with bond angles in the range of ca. 95.98 to 114.82° (see Table 3). The interatomic distance of for example N1-C7 (1.300(3) Å) indicates an isolated double bond character in opposite to the vicinal single bonds N1–C8 (1.437(3))Å) and C6–C7 (1.436(4) Å) suggesting a weak delocalization of the imine double bonds with the conjugated π systems of the two phenyl groups. The crystal structure shows evidence for weak intermolecular CH---O hydrogen bonding: C25–H25···O1, $d_{H \cdots O} = 2.466(2)$ Å, $\delta = 161.76(16)^{\circ}$; C40–H40···O2, $d_{H...O} = 2.399(2)$ Å, $\delta = 154.99(2)^{\circ}$.

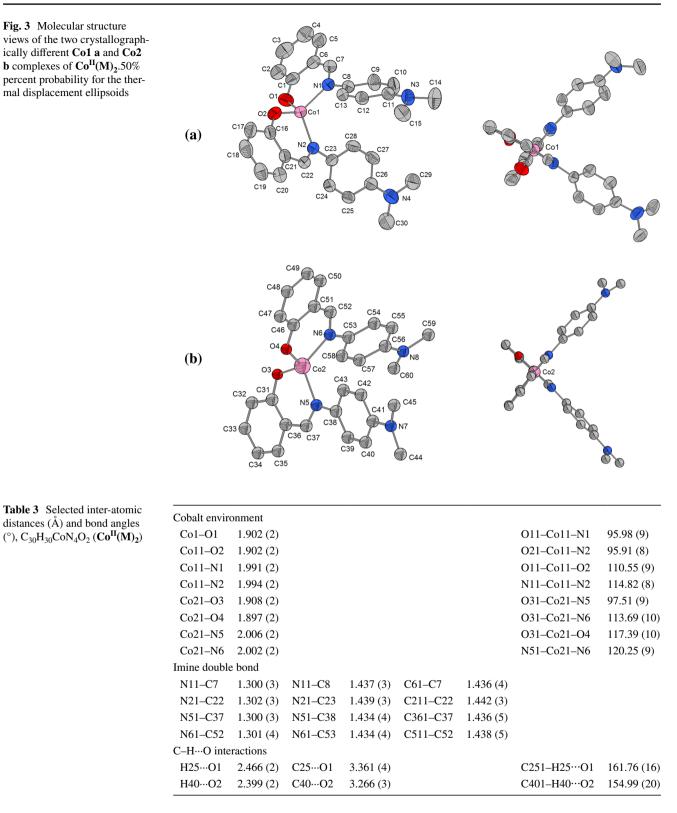
The main difference between the cristallographically different Co1 and Co2 complexes is of conformational nature. In both structures the imine double bond is almost in the same plane as the directly linked phenol group with torsion angles of ca. 2.9 and 4.0° (Co1-complex) and 0.3 and 3.0° (Co2-complex) whereas the dimethylaminophenyl group is found to be strongly twisted regarding the planar iminophenyl moiety in the Co1-complex with torsion angles of ca.

38.1° and 42.8° (Fig. 3). In contrast the π -conjugated ligand L is significantly closer to planarity with torsion angles of only ca. 8.1° and 24.6° .

The inhibition efficiency of $Co^{II}(L)_2$ and $Co^{II}(M)_2$ concerning the corrosion of mild steel in acidic solution has been investigated by electrochemical impedance spectroscopy. The impedance measurements were carried out in aerated 1 M hydrochloric acid. The corresponding Nyquist diagrams are shown in Fig. 4. The inhibition efficiency (IE%) of each Co complex was calculated from the charge transfer resistance values using the following equation:

$$IE\% = \frac{R_{ict} - R_{Oct}}{R_{ict}} \times 100$$
(1)

with Rct being the charge transfer resistance respectively in the presence (R_i ct) and in the absence (R_0 ct) of the inhibitor. Since corrosion reactions are strictly charge transfer controlled, the observed impedance behavior can be explained with the help of a simple and commonly used equivalent circuit composed of a double layer capacitance (Cd) being in series to the electrolyte resistance Rs and parallel to the double layer resistance Rct. This type of circuit has been used previously to model the iron/acid interface [20]. The obtained Nyquist plots are not perfect semi-circles which can be attributed to the frequency dispersion of the interfacial impedance [21, 22] due to the heterogeneity of the electrode surface (Fig. 4). This heterogeneity is generally resulting from roughness, impurities, dislocations and the formation of porous layers through irregular inhibitor adsorption [23, 24]. The impedance parameters Rct, Rs and the inhibition efficiency (IE%) are listed up in Table 4. The Rct values increase with increasing Co complex concentration $(10^{-5} \text{ to } 10^{-2} \text{ M})$ resulting in significant inhibition efficiencies of 25–61% for $Co^{II}(L)_2$ and 42–64% for Co^{II}(M)₂, respectively (Fig. 5; Table 4). These results



clearly indicate that both Co complexes act as effective corrosion inhibitors for mild steel in hydrochloric acid after only 30 min of immersion, suggesting partial passivation of the steel surface upon adsorption of the compounds as observed for analogues copper complexes cited below. The inhibition properties of $Co^{II}(L)_2$ proved to be comparable (IE%=62.9% for inhibitor concentration of c=10⁻⁴ M after 30 min immersion time at 30 °C) to the results

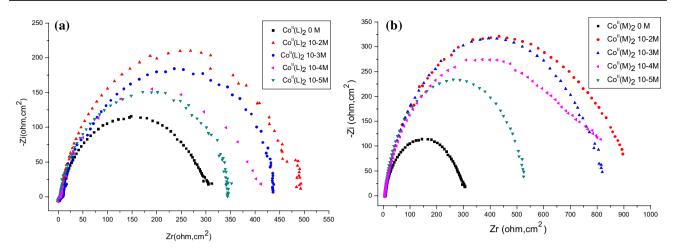


Fig. 4 Nyquist plots for mild steel exposed to (0, 10⁻⁵, 10⁻⁴, 10⁻³, 10⁻² M) solutions of Co^{II}(L)₂ (a) and Co^{II}(M)₂ (b) in 1 M hydrochloric acid

Table 4 Impedance parameters and inhibition efficiency for the corrosion of mild steel in 1 M aqueous HCl, in the absence and the presence of compounds $Co^{II}(L)_2$ and $Co^{II}(M)_2$ measured after 30 min of immersion time at 30 ± 1 °C

Inhibitor	Concentration (M)	Rs (Ω cm ²)	Rct	IE (%)
_	_	5.15	305.8	_
Co ^{II} (K) ₂	10^{-2}	13.64	796.5	61.6
	10^{-3}	3.48	488.8	37.4
	10^{-4}	9.5	444.8	31.4
	10^{-5}	4.22	409.6	25.3
Co ^{II} (L) ₂	10^{-2}	2.99	861.1	64.5
	10^{-3}	1.88	837.8	64.0
	10^{-4}	6.67	822.1	62.9
	10^{-5}	5.99	534	42.9

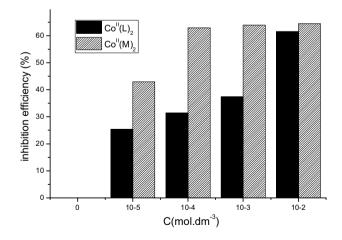


Fig. 5 Inhibition efficiency of $Co^{II}(L)_2$ and $Co^{II}(M)_2$ for the corrosion of mild steel in 1 M hydrochloric acid after 30 min of immersion time

reported by Keles et al. (IE% =77.0%, c=2.5 10^{-4} M, 60 min, 35 °C) [25]. Only Singh et al. measured particularly a high inhibition efficiency for a Schiff base cobalt complex (IE% =99.4%, c=0.6 10^{-4} M, 30 min, 30 °C) [26]. **Co^{II}(M)**₂ showed rather weak passivation properties under the same conditions (IE% = 31.4%, c= 10^{-4} M, 30 min, 30 °C) which is probably due to a lower surface affinity under similar acidic conditions. This could possibly arise from the presence of the exposed dimethyl amino group, protonated in the acidic solution and interacting with surface adsorbed chloride anions as proposed in the literature [25, 27].

Conclusion

The crystal structures of the two ferrocenyl- and phenylendiamine-substituted iminomethylphenoxy cobalt complexes $Co^{II}(L)_2$ and $Co^{II}(M)_2$ have been determined by X-ray diffraction. No particularly short intermetallic distances have been found. In spite of a complete Co coordination sphere both complexes were shown to be effective corrosion inhibitors for mild steel in acidic aqueous solution, suggesting partial surface passivation via adsorption of the compounds on the surface due to electrostatic and π interactions as proposed in literature [25, 27]. The significantly stronger inhibition properties of Co^{II}(L)₂ compared to Co^{II}(M)₂ could possibly arise from the presence of the exposed dimethyl amino group of the phenylendiamine ligand. No other particular structural elements explaining the observed behavior have been identified. So far only few data concerning analogues Co complexes as corrosion inhibitors have been reported in literature. Our results are confirming that not only free Schiff Bases [25, 26] but also the corresponding metal complexes can be interesting inhibitors. In order to

better understand the adsorption behavior and to improve the inhibition properties of this class of compounds a systematic investigation of analogue complexes and their parent free ligands (including compounds L and M) is in progress.

Supplementary Materials

CCDC-1446313 and CCDC-1446312 contain the supplementary crystallographic data for the paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/ data_request/cif.

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