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Original Article

Metal Complexes of π-Expanded Ligands (3): Synthesis and Characterization of tris[2-[(octylimino)methyl]-1-pyrenolato-N,O] cobalt(III)

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Abstract: The reactions of $Co(OAc)_2$ with two equivalents of 1-hydroxy-2-[(octylimino)methylpyrene **L**, performed in air, lead to the formation of the cobalt(III) complex, tris[2-[(octylimino)methyl]-1-pyrenolato-N,O] cobalt(III) **CoL**₃, accommodating three chelating pyrenebased salicylaldiminato-type ligands. The complex **CoL**₃ and the referent tris(salicylaldiminato) cobalt(III) **1'(Co**^{III}) were obtained in excellent yields, and their characterisation by ¹H NMR, IR, mass spectroscopy, elemental analysis and X-ray diffraction revealed that they were of diamagnetic nature, octahedral geometry with the cobalt centre and meridional configuration. The redox behaviour of these complexes shows an irreversible reduction wave with a peak potential in the range -1.9 to -1.2 V. Upon reduction, the complexes decompose, giving rise to a redox pattern compatible with the formation of bis[2-[(octylimino)methyl]-1-pyrenolato-N,O] cobalt(II).

Keywords: Coordination chemistry, Cobalt, Pyrene, *π*-Expanded ligand, Salicylaldimine.

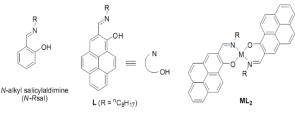
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1. Introduction

Design and synthesis of a new ligand aiming at a new metal complex is one of the obligations for synthetic chemists. Especially, the π electronic ligands and the corresponding metal complexes have been attracting much attention because of their versatile properties attributable to smaller HOMO-LUMO gap. We focus on metal complexes of N-alkylsalicylaldimine (N-Rsal), one of the most basic and important metal complexes in chemistry, and modify the π system of this ligand with pyrene. We have reported recently the synthesis and characterization of L and the corresponding metal complex, $M^{II}L_2$ (M = Pt, Pd, Ni) (Chart 1) [1-3].



(M = Pt, Pd, Ni)

Chart 1. Studied ligands and their corresponding metal complexes.

Cobalt N-alkylsalicylaldiminato (Co(N-Rsal)_n) and the related complexes show a high binding affinity with dioxygen and are proposed for the best model compounds as artificial blood and dioxygen transport materials (Chart 2) [4,5]. Furthermore, these Co complexes are used as catalysts for selective cyclopropanation of aromatic alkenes [6], catalysts for fuel cells [7], MOCVD precursors [8]. With a view to extending the π -system of our ligand **L** so as to enhance the properties of Co(N-Rsal)_n, a synthesis and characterization of cobalt complexes of **L** were carried out in this study.

All the previous metal(II) complexes have been obtained by the reaction of metal(II) acetate, or metal complexes, with two equivalents of the 1-hydroxy-2-[(octylimino) methyl-pyrene ligand. In this study, we report the

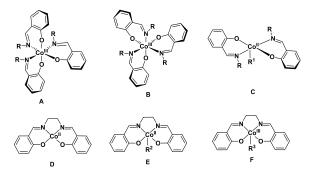


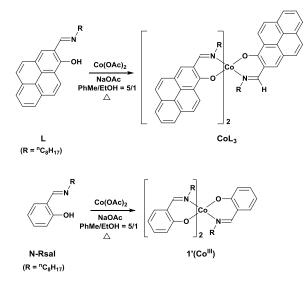
Chart 2. Cobalt N-alkylsalicylaldiminato and the related cobalt complexes.

synthesis and structural characterization of a cobalt(III) complex of the ligand. Herein, the resulting tris[2-[(octylimino)methyl]-1-pyrenolato-N,O] Co(III) **CoL**₃ and the referent cobalt(III) Co(N-Rsal)₃ are described and characterised by mass spectroscopy, elemental analysis, ¹H NMR spectroscopy, and X-ray diffraction. Moreover, absorption spectra and electrochemical properties of the complexese were investigated to confirm their improved photophysical properties due to small HOMO-LUMO gap.

2. Results and discussion

Synthesis

The pyrene-based salicylaldiminato-type ligand L and the referent salicylaldimine N-Rsal used in this work (Scheme 1) were prepared and characterised according to the methods described in the publications [1]. Treatment of the ligands L and N-Rsal with NaOAc in a solvent mixture of toluene and ethanol, resulted in the deprotonation of the ligand L, leaded to the formation of the corresponding sodium salts. The addition of cobalt(II) acetate tetrahydrate to the resulting solution, using a molar ratio of 2:1 $(ligand:Co(OAc)_2),$ under rigorous air atmosphere condition, afforded the Co(III) complexes CoL₃ and 1'(Co^{III}), containing three chelating pyrene-based salicylaldimine-type and salicylaldimine ligands. These complexes were purified by column chromatography (CHCl₃ as eluent) yielding a black solid **CoL**₃ and a green solid **1'(Co^{III})**, in excellent yields (67-99%).



Scheme 1. Synthesis of Co(III) complexes salicylaldiminato-type ligands.

It is well-known that cobalt(III) salicylaldiminato complexes can be easily prepared by the oxidation of a cobalt(II) salicylaldiminato complexes in the presence of excess of salicylaldimine with hydrogen peroxide [9]. The complexes are highly crystalline and generally appear black in reflected light but when crushed, they are brown-yellow or yellow-green. Similarly, in the present work, it is shown that the pyrenebased salicylaldiminato-type cobalt(III) CoL₃ and the referent cobalt(III) complex 1'(Co^{III}) were obtained under air atmosphere condition. This is slightly surprising because a recent report has shown that cobalt(III) complexes were obtained under nitrogen atmosphere condition from CoCl₂ [10]. The obtained products are diamagnetic octahedral Co(III) complexes containing three salicylaldimine-type ligands. The ¹H NMR spectra of these complexes do not show the hydroxyl group of the free ligand, at ca. 14.92 ppm, which attests to the presence of the ligand coordinated to the metal centre in a bidentate chelating mode.

It should be mentioned that the new complex CoL_3 is stable under ambient condition and/or toward the usual manipulations such as silica-gel chromatography and recrystallization from hot solvents, *e.g.*, boiling chloroform, under the air and room light. The reference complex **1**'(Co^{III}) was prepared according to the similar method.

Isomerism

The presence of two different kinds of coordinating atom on each ligand, such as oxygen and nitrogen, allows the existence of structural isomers for the cobalt(III) complexes with the configurations as shown in Figure 1. Attempts to isolate the two forms of any of the complexes prepared have failed. Molecular models suggest that a molecule bearing the cis-configuration, facial isomer, would be under very great steric strain because of the nearness of the nitrogen So much congestion arises between atoms. neighbouring alkyl or aryl substituents that models having the cis-configuration could not be assembled. Models of the trans-form still show considerable, but much less, strain, and it is tentatively concluded that the complexes are isolated mainly in the trans-form. Most of the cobalt(III) complexes prepared show some tendency to be reduced to the 4-co-ordinate cobalt(II) state when solutions containing these complexes are refluxed or evaporated, and this would support the concept of steric strain in the 6-coordinate cobalt(III) form since reduction would lead to a great decrease of strain in the cobalt complex that would be formed [9]. These were confirmed by ¹H NMR spectra for both complexes CoL₃ and 1'(Co^{III}).

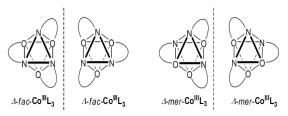


Figure 1. Possible configurations of salicylaldiminato-type cobalt(III) complexes.

Sharp ¹H NMR signal of the product is the clear evidence that the product is Co^{III} complexes, Co^{III}L₃ (Figure 2). This complex Co^{III}L₃ should have two diastereomers, facial and *meridional*, and the corresponding enantiomers, Δ and Λ , therefore, four stereoisomers Δ -fac-, Λ -fac-, Δ -mer-, and Λ mer-Co^{III}L₃, are possible [11]. The fac- and merisomers are easy to be distinguished based on the ¹H NMR signal equivalency. The complicated signals attributable to the three different magnetically unequivalent ligands were observed via the number of all proton signals. Therefore, we concluded that the obtained Co(III) complex is mer-isomer and the *i.e.*, racemic corresponding enantiomers, mixture of Δ -mer- and Λ -mer-Co^{III}L₃. This conclusion was supported by single crystal diffraction study (see below).

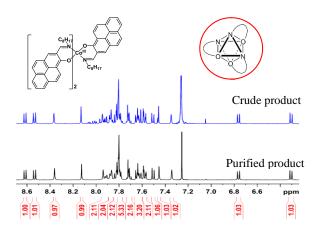


Figure 2. ${}^{1}H$ NMR spectra of crude product and purified product of CoL₃ complex.

X-ray diffraction study

Crystals suitable for X-ray diffraction were obtained for the synthesized complex CoL₃ and

the referent 1'(Co^{III}) [12]. The molecular structures of the complexes are shown in Figure 3 and the corresponding selected bond distance (Å) and angle (°) are listed in Table 1. All of them show the cobalt atom coordinated to three pyrene-based salicylald-imine-type chelating ligands, in which the Co¹-Oⁱ bond distances are always shorter than the metal-imine Co¹-Nⁱ ones (see Table 1), the corresponding values are in agreement with those found for the structure of the single cobalt(III) salicylaldiminato complex 1'(Co^{III}) and for other six-coordinated Co(III) complexes containing N,O-bidentate ligands [10]. Both complexes CoL₃ and 1'(Co^{III}) were assigned as meridional isomers that were described in isomerism part, perfectly reflecting the above discusses. The dihedral angles between the chelation planes of each ligand, which are defined by the cobalt, nitrogen and oxygen atoms, are close to 90°.

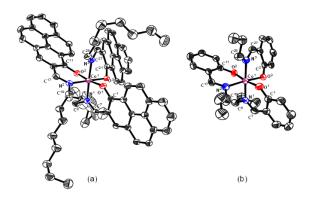


Figure 3. ORTEP view of the two complexes **CoL**₃ and **1'(Co^{III})** as obtained by single crystal X-ray diffraction: (a) **CoL**₃ perspective view, (b) **1'(Co^{III})** perspective view. Atomic displacement ellipsoids are draw at the 60%, 30% probability level for **CoL**₃ and **1'(Co^{III})**, respectively. Element (color): cobalt (violet), carbon (black), nitrogen (blue), oxygen (red). Hydrogen atoms are omitted for clarity.

	CoL ₃	1' (Co ^{III})		CoL ₃	1' (Co ^{III})
Distances (Å)			Angle (°)		
Co ¹ -O ¹	1.8777(17)	1.898(2)	$O^{1}-Co^{1}-O^{2}$	172.28(7)	171.62(8)
Co^1-O^2	1.8799(16)	1.8681(19)	O^1 - Co^1 - O^3	89.06(8)	89.08(9)
Co^1-O^3	1.9085(17)	1.8999(19)	O^2 - Co^1 - O^3	85.92(7)	85.97(8)
Co ¹ -N ¹	1.945(2)	1.941 (2)	O^1 - Co^1 - N^1	93.67(8)	94.74(10)
Co ¹ -N ²	1.950(2)	1.955 (2)	O^2 - Co^1 - N^1	91.94(8)	91.64(10)
Co ¹ -N ³	1.946(2)	1.952 (2)	$O^3-Co^1-N^1$	87.26(8)	85.83(9)
$N^{1}-C^{7}$	1.287(3)	1.283(4)	O^1 - Co^1 - N^3	84.17(8)	85.39(9)
N^2-C^{17}	1.290(3)	1.289(3)	O^2 - Co^1 - N^3	90.06(8)	88.00(9)
$N^{3}-C^{27}$	1.285(3)	1.286(3)	O^3 - Co^1 - N^3	91.03(8)	91.74(9)
O^1 - C^1	1.303(3)	1.305(3)	$N^{1}-Co^{1}-N^{3}$	177.27(9)	177.56(10)
O^2-C^{11}	1.311(3)	1.305(3)	O^1 - Co^1 - N^2	93.79(8)	92.85(10)
$O^{3}-C^{21}$	1.308(3)	1.301(3)	O^2 - Co^1 - N^2	91.45(8)	92.80(10)
N^1-C^8	1.484(3)	1.482(4)	$O^3-Co^1-N^2$	176.48(9)	173.60(9)
N^2-C^{18}	1.482(3)	1.492(4)	N^1 - Co^1 - N^2	90.50(8)	87.93(10)
$N^{3}-C^{28}$	1.478(3)	1.482(3)	N^3 - Co^1 - N^2	91.31(8)	94.50(10)

Table 1. Selected bond distances (Å) and angle (°) for complexes CoL₃ and 1'(Co^{III})

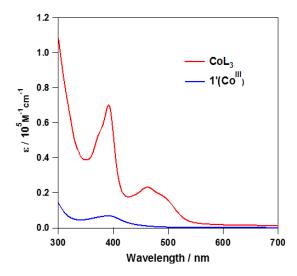


Figure 4. Absorption spectra of **CoL**₃ (solid line) and **1'(Co^{III})** (dotted line) in CH₂Cl₂ at 25°C.

Absorption Spectra

Absorption spectrum of CoL_3 measured in dichloromethane is shown in Figure 4 along with that of **1'(Co^{III})**. Similar to our reported pyrenebased metal complexes, the absorption of the complex **CoL**₃ shows bathochromic shift compared to that of the complex **1'(Co^{III})**. The lowest excitation energy of the Co(III) complex was observed at 462 nm.

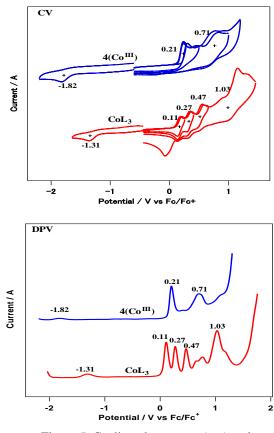


Figure 5. Cyclic voltammetry (top) and differential pulse voltammetry (bottom) of CoL_3 (solid line) and $1'(Co^{III})$ (dotted line) in CH_2Cl_2 .

Electrochemical studies

The redox behaviour of the Co(III) complexes, CoL₃ and 1'(Co^{III}), was investigated using cyclic voltammetry and differential pulse voltammetry (Figure 5). The study was performed at several scan rates (from 50 to 200 mV/s) at a Pt wire electrode in a [NBu₄][PF₆]/PhCN solution. It was established that these Co(III) complexes could be reduced at very cathodic potentials, in an irreversible process (even at the higher scan rates), attesting the chemical instability of the complexes formed after the reduction step. The difference in anodic and cathodic peaks (|Epc - Epa|) indicates that all observed oxidations are one electron and are diffusion-controlled under the conditions employed. The first reduction wave of CoL₃ (-1.31 V) is shifted positively by 510 mV, compared to that of 1'(Co^{III}) (-1.82 V), indicating a lower-lying LUMO level, which is certainly related to the donating influence of expansion of π system. In the case of CoL₃, it is possible to observe reversible first oxidation waves, which is irreversible in the cyclic voltammograms of the referent 1'(CoIII) complex. Furthermore, the first oxidation wave of CoL_3 (+0.11 V) is shifted negatively by 100 mV, compared to that of 1'(Co^{III}) (+0.21 V), indicating a higher-lying HOMO level, which is also certainly related to the donating influence of expansion of π -system. Therefore, the replacement of the benzene moiety by pyrene moiety induces smaller HOMO-LUMO gap that was also confirmed by absorption spectra in above items.

3. Conclusions

In summary, we successfully synthesized a new cobalt(III) complex, tris[2-[(octylimino)methyl]-1-pyrenolato-N,O]

cobalt(III) in excellent yield. It was observed that the above-mentioned compound exhibits meridional configuration. Reflecting the expansion of the π -system, the title compound shows deep color, higher reduction potential and lower oxidation potential by decreasing the HOMO-LUMO energy gap of the complex CoL₃. The authors are modifying pyrene-based ligands to obtain cobalt(II) complexes and other metal complexes for advanced materials.

Experimental

General experiment. General experimental details are already reported in elsewhere.

Synthesis

Preparation of *tris*[2-[(*n*-octylimino)methyl] -1-pyrenolanato-N,O] Cobalt(III), CoL₃.

A mixture of L^1 (30.1 mg, 84 µmol, 2 eq.), sodium acetate anhydrous(26.7 mg, 326 mmol µmol, 7.8 eq., Junsei Chemical Co., Ltd.), cobalt(II) acetate tetrahydrate (10.4 mg, 42 µmol, 1 eq.), and 6 mL of a solvent mixture of toluene (5 mL) and ethanol (1 mL) was stirred at 70 °C. After being stirred for 2h, evaporation of the solvent gave a crude product, which was purified by column chromatography (CHCl₃ as eluent) to yield *CoL*₃ as a black powder, 21.1 mg (67%). To get purer product, the above powder was recrystallized with a mixture of ethanol and CH₂Cl₂.

 $R_f = 0.6$ (hexane/chloroform = 1:3 as eluent); Melting point: 162 °C; ¹H NMR (500 MHz, CDCl₃, TMS): $\delta = 8.62$ (d, J = 9.0 Hz, 1H), 8.53 (d, J = 9.1 Hz, 1H), 8.36 (s, 1H), 8.13 (s, 1H), $8.00 \sim 7.55$ (m, 18H), 7.50 (d, J = 9.0 Hz, 1H), 7.45 (s, 1H), 7.34 (s, 1H), 6.76 (d, J = 9.1 Hz, 1H), 6.30(d, J = 9.0 Hz, 1H), 4.25 (m, 1H), 3.91 (m, 1H), 3.81 (m, 1H), 3.56 (m, 1H), 3.41 (m, 1H), 3.29 (m, 1H), 2.20-0.50 (m, 42H), and 0.45 (t, J = 7.0 Hz, 3H) ppm; IR (KBr): v = 2922(s),2853(s), $1615(s, v_{C=N})$, 1599(s), 1539(s), 1473(s), 1418(s), 1386(s), 1371(s), 1283(s), 1222(m), 1185(s), 1142(m), 1024(m), 950(w), 876(s), 840(m), 828(w), 816(w), 775(m), 754(m), 745(m), 686(m), 594(m), 572(m), 548(w), and 484(w) cm⁻¹; λ_{max} (CH₂Cl₂, 9.93 × 10⁻⁶ M)/nm 462 (ε/dm³mol⁻¹cm⁻¹23,260), 392 (70,260) and 375 (sh., 54,260) (the measurement in different concentration, 9.93×10^{-5} M, in CH₂Cl₂ showed the similar spectrum), MS (APCI): *m*/*z* (%): 1129.55 (100) ([M+H]⁺); elemental analysis calcd (%) for C₇₅H₇₈N₃O₃Co: C, 79.83; H, 6.97; N, 3.72; found: C, 79.62; H, 6.99; N, 3.67. Absorption spectra measured at 9.93×10^{-5} M in CH₂Cl₂ afforded the mostly identical result; therefore, no concentration dependencies were observed in this concentration region. A single crystal suitable for diffraction study was obtained by slow diffusion of a system of chloroform and hexane at room temperature to give needle-shaped crystals.

The reference cobalt(III) salicylaldiminato **1'(Co^{III})** has been prepared similar to the above synthesis method. The compounds 1-hydroxy-2-[(octylimino)methyl]-benzene (51 mg, 0.22 mmol, 2.2 eq.), anhydrous CH₃COONa (75 mg, 0.91 mmol, 7.6 eq) and cobalt(II) acetate tetrahydrate (30 mg, 0.12 mmol, 1 eq.) in 3 mL of ethanol was stirred at room temperature for 5h. The reaction solution was kept at room temperature about 2 days. Then, evaporation of the solvent gave a crude product, which was

purified by column chromatography on silica gel (CHCl₃ as eluent) to obtain 55 mg (99%) of the product $1'(Co^{III})$ as a green powder.

Mp: 82 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.52 (s, 1H), 7.49 (s, 1H), 7.15-7.00 (m, 4H), 7.00-6.85 (m, 3H), 6.76 (d, J = 8.3 Hz, 1H), 6.66 (d, J = 8.4 Hz, 1H), 6.62 (d, J = 8.4 Hz, 1H), 6.50-6.40 (m, 3H), 3.55 (m, 1H), 3.41 (m, 1H), 3.35-3.25 (m, 3H), 3.15 (m, 1H), 2.00-1.00 (m, 36H), 1.00-0.80 (m, 9H); IR (KBr): v = 1633 cm⁻¹ (s, $v_{C=N}$), λ_{max} (CH₂Cl₂, 2.01 × 10⁻⁴ M)/nm 391 (ϵ /dm³mol⁻¹cm⁻¹ 6,960), elemental analysis calcd (%) for C₄₅H₆₆N₃O₃Co: C, 71.50; H, 8.80; N, 5.56. Found: C, 71.24; H, 7.78; N, 5.42.

X-ray experimental data.

Suitable single crystals were grown as follows: CoL_3 as brown black needles was developed by slow diffusion of ethylacetate vapor into a saturated chloroform solution of CoL_3 at room temperature.

Samples	$CoL_3(R = {}^{n}C_8H_{17})$	1'(Co ^{III})	
Mol. Formula	C ₇₅ H ₇₈ CoN ₃ O ₃	$C_{30}H_{36}CoN_{3}O_{3}$	
Mol. Weight	1128.33	545.55	
Crystal habit	Black, block	Red, block	
Crystal dimens./mm	0.424 x 0.198 x 0.040	0.43 x 0.28 x 0.22	
Crystal system	Monoclinic	Tetragonal	
Space group	P 2 ₁ /n	$I4_1/a$	
a (Å)	15.9991(13)	19.588 (3)	
b (Å)	20.2213(13)	19.588 (3)	
<i>c</i> (Å)	19.1872(17)	29.877 (6)	
α (deg)	90	90	
β (deg)	105.457(4)	90	
γ (deg)	90	90	
V (Å ³)	5983.97	11464 (3)	
Ζ	4	16	
$D_{\rm calcd}$ (g/cm ³)	1.253	1.264	
F(000)	2400	4608	
μ (Mo K α) (cm ⁻¹)	3.38	6.30	
T/K	123 (2)	293 (2)	
$2\theta_{max}$ (deg)	55.0	50.6	
RadiationMoKa (1	$=$ MoK α	ΜοΚα	
0.71075 Å)			
R1, wR2 ($I > 2\sigma I$)	0.0622/0.1441	0.0474/0.126	
R _{int}	0.0622	0.0474	

Table 1 Crystal data and structure refinement details for CoL₃ and 1'(Co^{III})

Appendix A. Supplementary material

CCDC 1915760 contains the supplementary crystallographic data for 2019/05/13. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.htm l, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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