Syntheses, X-Ray Single Crystal Structures and Biological Activities of Cobalt(III) Complexes with Reduced Schiff Base Ligands

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Abstract

A new mononuclear cobalt(III) complex, $[Co(HL^1)_2]Cl(1)$, derived from the reduced Schiff base 2,2'-((ethane-1,2-diyl-bis(azanediyl))bis(methylene))diphenol (H_2L^1), and a new dinuclear cobalt(III) complex, $[Co_2(L^2)_2] \cdot 2H_2O(2)$, derived from the reduced Schiff base 6,6'-(2-hydroxypropane-1,3-diyl)bis(azanediyl)bis(methylene)bis(2-bromo-4-chlorophenol) (H_2L^2), were synthesized and characterized by infrared and electronic spectroscopy, and single crystal X-ray diffraction techniques. The ligands were synthesized first, and then bound to the Co(III) centre. Compound 1 contains a mononuclear $[Co(HL^1)_2]^+$ cation and a chloride anion. The cationic moiety possesses crystallographic inversion center symmetry. Compound 2 contains a dinuclear $[Co_2(L^2)_2]$ molecule and two water hydrate molecules. The Co atoms in the complexes are in octahedral coordination. Both complexes showed potential antimicrobial activity.

Keywords: Cobalt complex; reduced Schiff base ligand; crystal structure; antimicrobial activity

1. Introduction

Schiff bases derived from the condensation reactions of carbonyl containing compounds with primary amines have received tremendous attention in coordination chemistry because of their facile coordination ability to a large number of metals. Schiff bases have various biological applications. A number of complexes with Schiff base ligands have presented interesting biological properties, such as antibacterial, antifungal, antitumor, and enzymatic catalytic property. The Schiff bases have good complexing ability and their biological activity increases on complex with metal ions. Multi-dentate Schiff base ligands containing both nitrogen and oxygen donor atoms,

derived from the condensation of salicylaldehyde and various diamines have intensely been studied, due to their ability to stabilize a great variety of coordination numbers and coordination geometries.⁵ Cobalt(III) Schiff base complexes are also important in bioinorganic chemistry for important biological processes like antibacterial, antitumor, and antifungal activities.⁶ However, Schiff bases are not very stable in acid condition due to the azomethine groups. Reduced Schiff bases are in general more stable than Schiff bases. In this paper, two new cobalt(III) complexes, $[Co(HL^1)_2]Cl(1)$ and $[Co_2(L^2)_2] \cdot 2H_2O(2)$, where HL^1 and L^2 are the monoanionic form of the reduced Schiff base 2,2'-((ethane-1,2-diylbis(azanediyl))bis(methylene))diphenol (H_2L^1) and the trianionic form of the re-

Scheme 1. H_2L^1 and H_3L^2 .

duced Schiff base 6,6'-(2-hydroxypropane-1,3-diyl)bis (azanediyl)bis(methylene)bis(2-bromo-4-chlorophenol) (H₃L²; Scheme 1), were synthesized and studied on their antibacterial potential.

2. Experimental

2. 1. General Methods and Materials

All reagents and solvents were purchased from the commercial sources and used as received. Elemental (C, H and N) analyses were performed on a Perkin–Elmer 2400 II analyzer. IR spectra were recorded in the region 4000–400 cm⁻¹ on a Perkin Elmer IR RXI spectrometer with samples as KBr disks. UV-Vis spectra were recorded on a Shimadzu UV-3600 spectrophotometer. Molar conductivity was measured at 25 °C with a DDS-11A conductivity meter. The NMR spectra were recorded on a Bruker spectrometer at 300 MHz. X-ray diffraction was carried out on a Bruker Apex II CCD diffractometer.

2. 2. Synthesis of H₂L¹

To salicylaldehyde (1.22 g, 10 mmol) diluted by MeOH (50 mL), 1,2-diaminoethane (0.30 g, 5 mmol) diluted by MeOH (50 mL) was added with stirring. The reaction mixture was refluxed for 1 h and cooled by ice-water bath. Then, NaBH₄ (1.0 g, 25 mmol) was added. The mixture was stirred for another 1 h and filtered. The solvent was removed by distillation. The residue was treated with the aqueous solution of 1 M NaOH (50 mL) and extracted by chloroform. The solution was treated with 3 M HCl and the acid phase was made basic by 1 M NaOH. The crude product was then extracted into chloroform. The chloroform extracts were combined and dried over anhydrous Na₂SO₄. The solvent was removed to give the colorless product. Yield 0.9 g (66%). IR data (v, cm⁻¹): 3337, 3212, 3061, 2983, 2930, 2855, 1600, 1481, 1075. UV-Vis data (MeOH; λ_{max}, nm): 260, 285. ¹H NMR (300 MHz, CDCl₃, ppm) δ 7.06 (q, 2H, ArH), 7.03 (d, 2H, ArH), 6.91 (q, 2H, ArH), 6.77 (d, 2H, ArH), 3.85 (s, 4H, CH₂), 2.61 (t, 4H, CH₂). Anal. Calcd. (%) for C₁₆H₂₀N₂O₂: C, 70.56; H, 7.40; N, 10.29. Found (%): C, 70.45; H, 7.47; N, 10.23.

2. 3. Synthesis of H_3L^2

 H_3L^2 was prepared by the same method as described for $\,H_2L^1,\,\,$ with salicylaldehyde replaced by 3-bromo-5-chlorosalicylaldehyde (2.34 g, 10 mmol), and with 1,2-diaminoethane replaced by 1,3-diaminopropan-2-ol (0.45 g, 5 mmol). Yield 1.6 g (61%). IR data (v, cm^-1): 3382, 3235, 3053, 2977, 2941, 2872, 1597, 1478, 1081. UV-Vis data (MeOH; $\lambda_{max},\,$ nm): 255, 273. 1H NMR (300 MHz, CDCl_3, ppm) δ 11.83 (s, 1H, OH), 7.41 (s, 2H, ArH), 7.23 (s, 2H, ArH), 3.71 (d, 4H, CH_2), 3.67 (m, 1H, CH), 2.68 (t, 4H, CH_2). Anal. Calcd. (%) for $C_{17}H_{18}Br_2ClN_2O_3$:

C, 38.59; H, 3.43; N, 5.30. Found (%): C, 38.44; H, 3.52; N, 5.27.

2. 4. Synthesis of Complex 1

A solution of H_2L^1 (54.4 mg, 0.20 mmol) in MeOH (10 mL) was added dropwise with stirring at room temperature to a solution of $CoCl_2 \cdot 6H_2O$ (23.8 mg, 0.10 mmol) in MeOH (10 mL). The solution immediately became deep brown and was stirred for 1 h. Single crystals suitable for X-ray diffraction were obtained after 11 days by slow evaporation of the reaction solution. Yield: 23 mg (36%). IR data (v, cm⁻¹): 3245, 1218, 1073. UV-Vis data (MeOH; λ_{max} , nm): 280, 372. Λ_{M} (10⁻³ mol L⁻¹ in MeOH): 151 Ω^{-1} cm² mol⁻¹. Anal. Calcd. (%) for $C_{32}H_{38}ClCoN_4O_4$: C, 60.33; H, 6.01; N, 8.79. Found (%): C, 60.46; H, 6.12; N, 8.76.

2. 5. Synthesis of Complex 2

Complex **2** was prepared by the same method as described for complex **1**, with H_2L^1 replaced by H_3L^2 (52.9 mg, 0.10 mmol), and with $CoCl_2 \cdot 6H_2O$ replaced by $Co(CH_3COO)_2 \cdot 4H_2O$ (24.9 mg, 0.10 mmol). Single crystals suitable for X-ray diffraction were obtained after 3 days by slow evaporation of the reaction solution. Yield: 23 mg (38%). IR data (ν , cm⁻¹): 3419, 3246, 1587, 1443, 1303, 1274, 1213, 1169, 1085, 861, 730, 605. UV–Vis data (MeOH; λ_{max} , nm): 210, 263, 317, 372. Λ_{M} (10⁻³ mol L⁻¹ in MeOH): 21 Ω^{-1} cm² mol⁻¹. Anal. Calcd. (%) for $C_{34}H_{34}Br_4Cl_4Co_2N_4O_9$: C, 33.42; H, 2.80; N, 4.59. Found (%): C, 33.56; H, 2.87; N, 4.47.

2. 6. X-ray Crystallography

The crystallographic data for the complexes are summarized in Table 1. Diffraction data of the complexes were collected on a Bruker APEX II CCD diffractometer at 298(2) K using graphite-monochromated Mo Ka radiation $(\lambda = 0.71073 \text{ Å})$. For data processing and absorption correction the packages SAINT and SADABS were used.7 The structures were solved by direct and Fourier methods and refined by full-matrix least-squares based on F2 using SHELXTL and SHELXL-97 packages.8 The non-hydrogen atoms were refined anisotropically. The hydrogen atoms on water molecules and the amino groups of complex 2 were located from a Fourier difference map and refined isotropically, with O-H, N-H and H.-.H distances restrained to 0.85(1), 0.90(1) and 1.37(2) Å, respectively. The structure of complex 2 containing solvent accessible voids of 236 Å³ may accommodate disordered solvent molecules. The remaining hydrogen atoms were inserted on geometrical calculated positions with fixed thermal parameters and were refined isotropically.

CCDC 1906730 and 1946056 contain the supplementary crystallographic data for the complexes 1 and 2, respectively. The data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from

Table 1. Crystallographic and refinement data for	r the complexes
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	1	2
Formula	C ₃₂ H ₃₈ ClCoN ₄ O ₄	C ₃₄ H ₃₄ Br ₄ Cl ₄ Co ₂ N ₄ O ₉
FW	637.04	1221.95
Crystal system	Monoclinic	Monoclinic
Space group	C2/c	$P2_1/c$
a (Å)	23.905(1)	15.732(2)
b (Å)	7.376(1)	12.478(2)
c (Å)	17.745(1)	23.992(3)
β (°)	95.753(1)	103.727(2)
$V(Å^3)$	3113.2(6)	4575.2(9)
Z	4	4
$\mu (\text{Mo}K\alpha) (\text{cm}^{-1})$	0.679	4.504
Collected reflections	8606	26672
Unique reflections	2861	8507
Observed reflections $[I \ge 2\sigma(I)]$	1591	4823
Parameters	192	514
Restraints	0	6
Goodness of fit on F^2	0.947	1.039
R_1 , wR_2 [$I \ge 2\sigma(I)$]	0.0532, 0.1031	0.0718, 0.2049
R_1 , wR_2 (all data)	0.1275, 0.1307	0.1345, 0.2399

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.

2. 7. Antibacterial Activity

The antibacterial activities were tested against *B. sub-tilis*, *E. coli*, *P. fluorescence* and *S. aureus* using MH medium

(Mueller–Hinton medium). The MICs (minimum inhibitory concentrations) of the test compounds were determined by a colorimetric method using the dye MTT [3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide]. A stock solution of the synthesized compound (50 µg mL⁻¹) in DMSO was prepared and quantities of the test compounds were incorporated in specified quantity of sterilized liquid MH medium. A specified quantity of the

Scheme 2. The synthetic procedure of the complexes.

medium containing the compound was poured into micro-titration plates. A suspension of the microorganism was prepared to contain approximately 10^5 cfu mL⁻¹ and applied to micro-titration plates with serially diluted compounds in DMSO to be tested and incubated at 37 °C for 24 h. After the MICs were visually determined on each of the micro-titration plates, 50 μ L of PBS containing 2 mg of MTT per millilitre was added to each well. Incubation was continued at room temperature for 4–5 h. The content of each well was removed and $100~\mu$ L of isopropanol containing hydrochloric acid was added to extract the dye. After 12 h of incubation at room temperature, the optical density (OD) was measured with a micro-plate reader at 550 nm.

3. Results and Discussion

3. 1. Chemistry

The cobalt(III) complexes 1 and 2 were prepared by the reaction of $CoCl_2 \cdot 6H_2O$ with the reduced Schiff base H_2L^1 , and $Co(CH_3COO)_2 \cdot 4H_2O$ with the reduced Schiff base H_3L^2 , respectively in MeOH (Scheme 2). The aerial oxidation of cobalt(II) to cobalt(III) and metal assisted deprotonation of the phenolic moieties took place during the formation of the complexes. Molar conductivity values of complexes 1 and 2 measured in MeOH indicate the 1:1 electrolytic nature of complex 1 and non-electrolytic nature of complex 2.9

3. 2. Spectral Characterization

In the IR spectra of the complexes, the bands corresponding to the azomethine groups (-CH=N-) are not

observed, instead, new bands indicative of the C–N groups are observed at 1073–1085 cm $^{-1}$, indicating the reduction of the –CH=N– double bonds to the –CH $_2$ –NH– single bonds. The complexes show medium bands at 1213–1218 cm $^{-1}$ due to the presence of Ar–O stretching. Weak and sharp bands for the spectra of the complexes located at 3245 cm $^{-1}$ indicates the presence of amino groups. The weak and broad band centered at 3419 cm $^{-1}$ for complex 2 can be assigned to the stretching vibration of water molecules.

The UV-Vis spectra of the complexes were measured in MeOH. There are two bands centered at 280 and 372 nm for $\bf 1$ and four bands centered at 210, 263, 317 and 372 nm for $\bf 2$. The bands arise due to internal ligand transition or ligand to metal charge transfer. The complexes exhibit low intensity bands at 615–630 nm which are due to the d-d transition of Co^{III} center.

3. 3. Structure Description of the Complexes

Selected bond lengths and bond angles in the coordination environment of the metal center are listed in Table 2. Complex 1 contains a mononuclear [Co(HL¹)₂]⁺ cation and a chloride anion (Fig. 1). The cationic moiety possesses a crystallographic inversion symmetry. The Co atom, lying on the inversion center, is coordinated by two phenolate O and four amino N atoms from two HL¹ ligands, forming octahedral coordination. The Co–O and Co–N bond lengths in the complex are in the range 1.906(3)–1.991(3) Å, which are very close to those reported in literature. The cisoid (85.8(1)–94.1(1)°) and transoid angles (180°) in the complex are almost near to the ideal values.

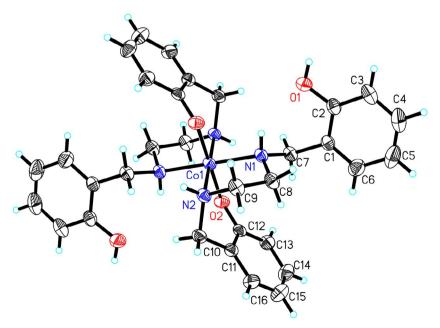


Fig. 1. The mononuclear complex cationic moiety of 1. The chloride anion is omitted for clarity. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

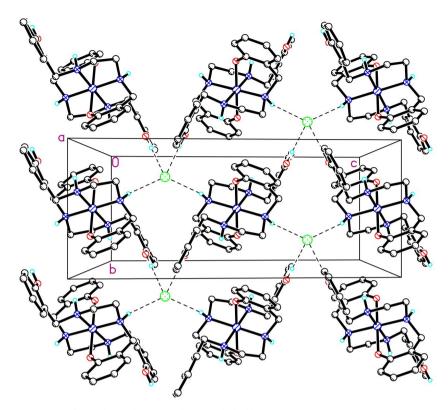


Fig. 2. Molecular packing structure of 1. Hydrogen bonds are shown as dashed lines.

In the crystal structure, the complex cations are linked by chloride anions through intermolecular hydrogen bonds of types N–H····Cl, N–H····O and O–H····Cl (Table 3), to form two-dimensional network along the *bc* plane (Fig. 2).

Complex 2 contains a dinuclear $[Co_2(L^2)_2]$ molecule and two water hydrate molecules (Fig. 3). The Co1 atom is coordinated by two phenolate O, two amino N, and two hydroxy O atoms from two L^2 ligands, forming octahedral coordination. The Co2 atom is coordinated by two pheno-

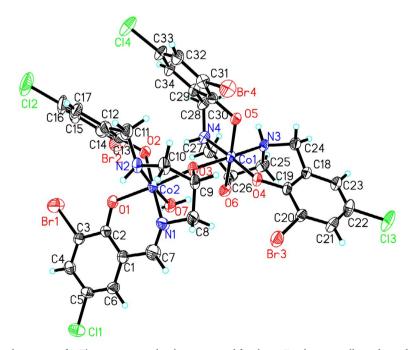


Fig. 3. The dinuclear complex moiety of 2. The two water molecules are omitted for clarity. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

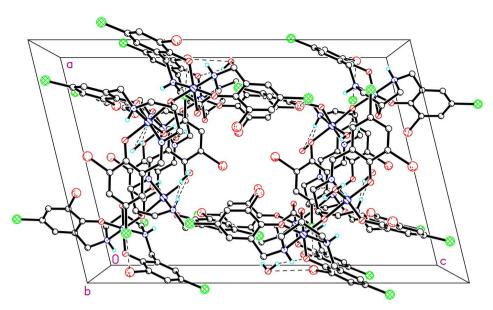


Fig. 4. Molecular packing structure of 2. Hydrogen bonds are shown as dashed lines.

late O, two amino N, and one hydroxy O atoms from one L^2 ligand, and by one water O atom, forming octahedral coordination. The Co–O and Co–N bond lengths in the

Table 2. Selected bond lengths (Å) and angles (°) for the complexes

1			
Co1-O2	1.906(3)	Co1-N2	1.951(3)
Co1-N1	1.991(3)		
O2-Co1-O2 ^{#1}	180	O2-Co1-N2#1	85.9(1)
O2-Co1-N2	94.1(1)	N2-Co1-N2 ^{#1}	180
O2-Co1-N1	93.8(1)	O2-Co1-N1#1	86.2(1)
N2-Co1-N1#1	93.7(1)	N2-Co1-N1	86.3(1)
N1-Co1-N1#1	180		
2			
Co1-O4	1.893(6)	Co1-O6	1.905(7)
Co1-N4	1.922(7)	Co1-O5	1.931(6)
Co1-N3	1.942(7)	Co1-O3	1.957(6)
Co2-O1	1.869(7)	Co2-N1	1.895(9)
Co2-O2	1.892(7)	Co2-O3	1.929(6)
Co2-O7	1.932(9)	Co2-N2	1.932(9)
O4-Co1-O6	95.3(3)	O4-Co1-N4	176.9(3)
O6-Co1-N4	81.9(3)	O4-Co1-O5	88.6(3)
O6-Co1-O5	175.8(3)	N4-Co1-O5	94.2(3)
O4-Co1-N3	94.3(3)	O6-Co1-N3	85.5(3)
N4-Co1-N3	87.0(3)	O5-Co1-N3	92.7(3)
O4-Co1-O3	90.1(3)	O6-Co1-O3	91.4(3)
N4-Co1-O3	88.5(3)	O5-Co1-O3	90.1(3)
N3-Co1-O3	174.8(3)	O1-Co2-N1	91.4(3)
O1-Co2-O2	89.4(3)	N1-Co2-O2	175.8(4)
O1-Co2-O3	173.3(4)	N1-Co2-O3	87.5(3)
O2-Co2-O3	92.2(3)	O1-Co2-O7	92.9(3)
N1-Co2-O7	90.3(4)	O2-Co2-O7	85.5(3)
O3-Co2-O7	93.8(3)	O1-Co2-N2	91.2(4)
N1-Co2-N2	89.1(4)	O2-Co2-N2	95.0(3)
O3-Co2-N2	82.1(3)	O7-Co2-N2	175.9(3)

Symmetry code for #1: -x, -y, 1 - z.

complex are in the range 1.871(6)-1.957(6) Å, which are very close to those reported in literature. The cisoid $(81.9(3)-95.3(3)^{\circ}$ for Co1 and $82.1(3)-95.0(3)^{\circ}$ for Co2) and transoid angles $(174.8(3)-176.9(3)^{\circ}$ for Co1 and $173.3(4)-175.9(3)^{\circ}$ for Co2) in the complex are almost near to the ideal values.

In the crystal structure, the complex molecules and the water molecules are linked through intermolecular hydrogen bonds of types O–H···O, O–H···N and N–H···O (Table 3), to form two-dimensional network along the *ab* plane (Fig. 4).

3. 4. Antibacterial Activity

The free reduced Schiff base ligands and their cobalt(III) complexes were screened for antibacterial activity against two Gram-positive bacterial strains (B. subtilis and S. aureus) and two Gram-negative bacterial strains (E. coli and P. fluorescence) by the MTT method. The MIC values of the compounds against these bacteria are presented in Table 4. Penicillin G and kanamycin were assayed as references. H₂L¹ is inactive against two Gram-positive bacterial strains B. subtilis and S. aureus, and has weak activity against the Gram-negative bacterial strains E. coli and P. fluorescence with MIC values of 25 μ g mL⁻¹. H₃L² is inactive against the Gram-negative bacterial strain P. fluorescence, and has weak activity against the Gram-negative bacterial strain E. coli and the Gram-positive bacterial strain S. aureus, with MIC values of 25 μ g mL⁻¹. H₃L² is active against the Gram-positive bacterial strain *B. subtilis*, with MIC value of 12.5 μ g mL⁻¹. The cobalt(III) complexes, in general, showed a wide range of bactericidal activities against all the Gram-positive and Gram-negative bacteria. Complex 1 has good activity against the Gram-positive bacterial strain B. subtilis and medium activity against

Table 3. Hydrogen bond distances (Å) and bond angles (°) for the complexes

D-H···A	d(D-H)	$d(\mathbf{H} \cdot \cdot \cdot A)$	$d(D\cdots A)$	$\angle (D\text{-H}\cdots A)$
1				
N2-H2···Cl1#1	0.89	2.50	3.246(3)	142
N1-H1···O1	0.90	2.45	3.011(4)	120
O1-H1A···Cl1	0.84	2.24	3.068(3)	171
2				
O7-H7A···O6	0.85(1)	1.68(3)	2.507(10)	162(9)
O9-H9AN2	0.85(1)	2.28(6)	3.048(17)	151(11)
N4-H4···O2	0.90(1)	1.88(4)	2.741(10)	159(11)
N2-H2···O9	0.90(1)	2.15(2)	3.048(17)	177(11)
O8-H8B···O5 ^{#2}	0.85(1)	2.18(5)	2.966(11)	154(10)
N3-H3···O8 ^{#3}	0.90(1)	2.23(7)	2.999(12)	143(10)

Symmetry codes: #1: -x, -y, 1 -z; #2: x, y, 1 +z; #3: -x, 3/2 +y, 3/2 -z.

S. aureus, with MIC values of 3.12 and 12.5 μ g mL⁻¹, respectively. Complex **2** has good activity against both Gram-positive bacterial strains *B. subtilis* and *S. aureus*, with MIC values of 0.78 and 6.25 μ g mL⁻¹, respectively. As for the two Gram-negative bacterial strains *E. coli* and *P. fluorescence*, both complexes have excellent activities with MIC values of 1.56 and 0.78 μ g mL⁻¹ for **1**, and 3.12 and 6.25 μ g mL⁻¹ for **2**, respectively, which are stronger than the reference drug kanamycin.

Table 4. MIC values (μg mL⁻¹) of the compounds

	B. subtilis	S. aureus	E. coli	P. fluorescence
1	3.12	12.5	1.56	0.78
2	0.78	6.25	3.12	6.25
H_2L^1	>100	>100	25	25
H_3L^2	12.5	25	25	>100
Penicillin G	0.78	3.13	>100	>100
Kanamycin	0.39	1.56	6.25	6.25

4. Conclusion

Two new cobalt(III) complexes with reduced Schiff base ligands 2,2'-((ethane-1,2-diylbis(azanediyl)) bis(methylene))diphenol and 6,6'-(2-hydroxypropane-1,3-diyl)bis(azanediyl)bis(methylene)bis(2-bromo-4-chlorophenol) have been synthesized and structurally characterized. One complex is in mononuclear and the other one is in dinuclear. The Co atoms are in octahedral coordination. The complexes showed potential antimicrobial activities against two Gram-positive bacterial strains (*B. subtilis* and *S. aureus*) and two Gram-negative bacterial strains (*E. coli* and *P. fluorescence*).

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Povzetek

Sintetizirali smo nov enojedrni kobaltov(III) kompleks, $[Co(HL^1)_2]Cl(1)$, z uporabo reducirane Schiffove baze 2,2'-((etan-1,2-diilbis(azandiil))bis(metilen))difenol (H_2L^1) , in nov dvojedrni kobaltov(III) kompleks, $[Co_2(L^2)_2] \cdot 2H_2O(2)$, z uporabo reducirane Schiffove baze 6,6'-(2-hidroksipropan-1,3-diil)bis(azandiil)bis(metilen)bis(2-bromo-4-chlorofenol) (H_2L^2) . Kompleksa smo okarakterizirali z infrardečo in elektronsko spektroskopijo ter rentgensko monokristalno analizo. Predhodno smo sintetizirali ligande ter jih nato vezali na Co(III) centre. Spojina 1 vsebuje enojedrni $[Co(HL^1)_2]^+$ kation in kloridni anion. Kation leži na kristalografskem centru inverzije. Spojina 2 vsebuje dvojedrne $[Co_2(L^2)_2]$ molekule in dve molekuli hidratne vode. V kompleksih je Co atom oktaedrično koordiniran. Oba kompleksa izkazujeta potencialne protimikrobne lastnosti.



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