

Scientific paper

Synthesis, Characterization, X-Ray Crystal Structures and Antibacterial Activity of Cobalt(III) Complexes Derived from 5-Bromo-2-((2-(phenylamino)ethylimino)methyl)phenol

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Abstract

Two new and similar cobalt(III) complexes, $[\text{CoL}_2] \cdot \text{NO}_3$ (**1**) and $[\text{CoL}_2] \cdot \text{Cl}$ (**2**), where L is 5-bromo-2-((2-(phenylamino)ethylimino)methyl)phenolate, have been synthesized and characterized by IR and UV-Vis spectra. Structures of the complexes were confirmed by single crystal X-ray determination. The Co atoms in the complexes are in octahedral coordination, with the donor atoms come from the two Schiff base ligands, viz. phenolate oxygen, and imino and amino nitrogen. The anions of the cobalt salts crystallized as counteranions in the complexes. The complexes were assayed for antibacterial activities by MTT method.

Keywords: Schiff base; cobalt complex; synthesis; X-ray diffraction; antibacterial activity

1. Introduction

Schiff bases due to their facile synthesis and interesting biological activities, have attracted considerable attention in inorganic and biological chemistry. In the past years, a number of Schiff base compounds have shown antifungal, antibacterial, antiproliferative, and antitumor activities.¹ Schiff base compounds possess N and O donor atoms, are important ligands in coordination chemistry. A huge number of Schiff base complexes have been reported for their structures and interesting properties like magnetism, catalytic application, and biological activities.² It is well known that some drug activities, when administered as metal complexes, are being increased.³ The field of medicine has witnessed an increase in the number of complexes with therapeutic value, for example Schiff base copper, nickel and zinc complexes are biological active agents,⁴ cobalt complexes with Schiff bases have shown effective antibacterial activities.⁵ The coordination chemistry of cobalt is of considerable interest since cobalt(II) and cobalt(III) complexes derived Schiff bases are reported to be biologically active.⁶ In addition, the Schiff bases synthesized from salicylaldehyde derivatives with halo atoms in the aromatic ring, showed effective antibacterial activities.⁷ Herein, we report the

synthesis, characterization, and single crystal structures of two new cobalt(III) complexes, $[\text{CoL}_2] \cdot \text{NO}_3$ (**1**) and $[\text{CoL}_2] \cdot \text{Cl}$ (**2**), where L is 5-bromo-2-((2-(phenylamino)ethylimino)methyl)phenolate. The antibacterial activity of the compounds against Gram-positive bacterial strains (*B. subtilis*, *S. aureus* and *St. faecalis*) and Gram-negative bacterial strains (*E. coli*, *P. aeruginosa* and *E. cloacae*) by MTT method was studied.

2. Experimental

2.1. Materials and Physical Methods

4-Bromosalicylaldehyde, *N*-phenylethane-1,2-diamine, cobalt nitrate and cobalt chloride were purchased from Aldrich. The solvents used in the synthesis and biological assay were commercial obtained and used as received. Elemental analyses for C, H and N were performed on a Perkin-Elmer 2400 II analyzer. FT-IR spectra were recorded as KBr pellets on Bruker Tensor-27. UV-Vis spectra were recorded on Lambda 35 spectrophotometer. Single crystal X-ray diffraction was carried out with a Bruker Apex II CCD diffractometer. Molar conductivity was measured in methanol with a DDS-11A molar conductivity meter.

2. 2. Synthesis of [CoL₂] · NO₃ (1)

4-Bromosalicylaldehyde (0.20 g, 1.0 mmol) and *N*-phenylethane-1,2-diamine (0.14 g, 1.0 mmol) were reacted in methanol (30 mL). The mixture was stirred at reflux for 30 min, and cobalt nitrate hexahydrate (0.29 g, 1.0 mmol) was added. After one hour stirring, the solution was cooled to room temperature, filtered, and with the filtrate kept still for slow evaporation. The diffraction quality block like brown single crystals that deposited over a period of 8 days were collected by filtration and washed with methanol. The yield was 0.16 g (42%). Anal. Calc. (%) for C₃₀H₂₈Br₂CoN₅O₅: C, 47.58; H, 3.73; N, 9.25. Found (%): C, 47.39; H, 3.82; N, 9.13. IR data (KBr, cm⁻¹): 3155w, 3054w, 2971w, 2933w, 2853w, 1650s, 1586s, 1520m, 1490w, 1427s, 1382s, 1325w, 1310w, 1291m, 1196w, 1130m, 1077w, 1055w, 1038w, 943w, 913m, 852w, 800w, 777w, 770w, 755w, 693w, 604w, 561w, 470w. UV–Vis data [methanol, λ/nm (ε/L·mol⁻¹·cm⁻¹): 245 (16,630), 263 (15,820), 302 (9,870), 385 (2,450), 495 (243). Λ_M (10⁻³ mol L⁻¹ in methanol): 128 Ω⁻¹ cm² mol⁻¹.

2. 3. Synthesis of [CoL₂] · Cl (2)

This complex was prepared with the similar method as described for complex 1, with cobalt nitrate hexahydrate replaced by cobalt chloride hexahydrate (0.24 g, 1.0 mmol). The diffraction quality block like brown single crystals that

deposited over a period of 5 days were collected by filtration and washed with methanol. The yield was 0.21 g (58%). Anal. Calc. (%) for C₃₀H₂₈Br₂ClCoN₄O₂: C, 49.31; H, 3.86; N, 7.67. Found (%): C, 49.43; H, 3.81; N, 7.58. IR data (KBr, cm⁻¹): 3167w, 3047w, 2962w, 2891w, 2849w, 1648s, 1586s, 1519m, 1489w, 1427s, 1327w, 1291m, 1202w, 1189m, 1124m, 1079w, 1058w, 1040w, 943w, 913m, 853w, 787w, 770w, 757w, 701w, 693w, 600w, 561w, 466w. UV–Vis data [methanol, λ/nm (ε/L·mol⁻¹·cm⁻¹): 230 (16,120), 248 (16,530), 260 (16,400), 304 (9,720), 385 (1,810), 497 (225). Λ_M (10⁻³ mol L⁻¹ in methanol): 135 Ω⁻¹ cm² mol⁻¹.

2. 4. X-Ray Structure Determination

Intensity data of the complexes were collected at 298(2) K on a Bruker Apex II CCD diffractometer using graphite-monochromated MoK_α radiation (λ = 0.71073 Å). For data processing and absorption correction the packages SAINT and SADABS were used.⁸ Structures of the complexes were solved by direct and Fourier methods and refined by full-matrix least-squares based on *F*² using SHELXL.⁹ The non-hydrogen atoms were refined anisotropically. The amino hydrogen atom were located from electronic density maps, and refined with N–H distances restrained to 0.90(1) Å. The remaining hydrogen atoms have been placed at geometrical positions with fixed thermal parameters. Crystallographic data of the complexes are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

Table 1. Crystallographic data and refinement details for the complexes

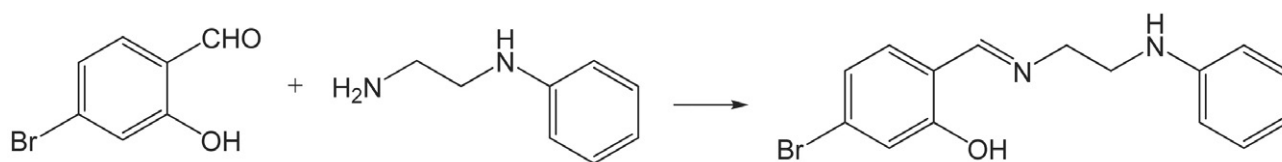
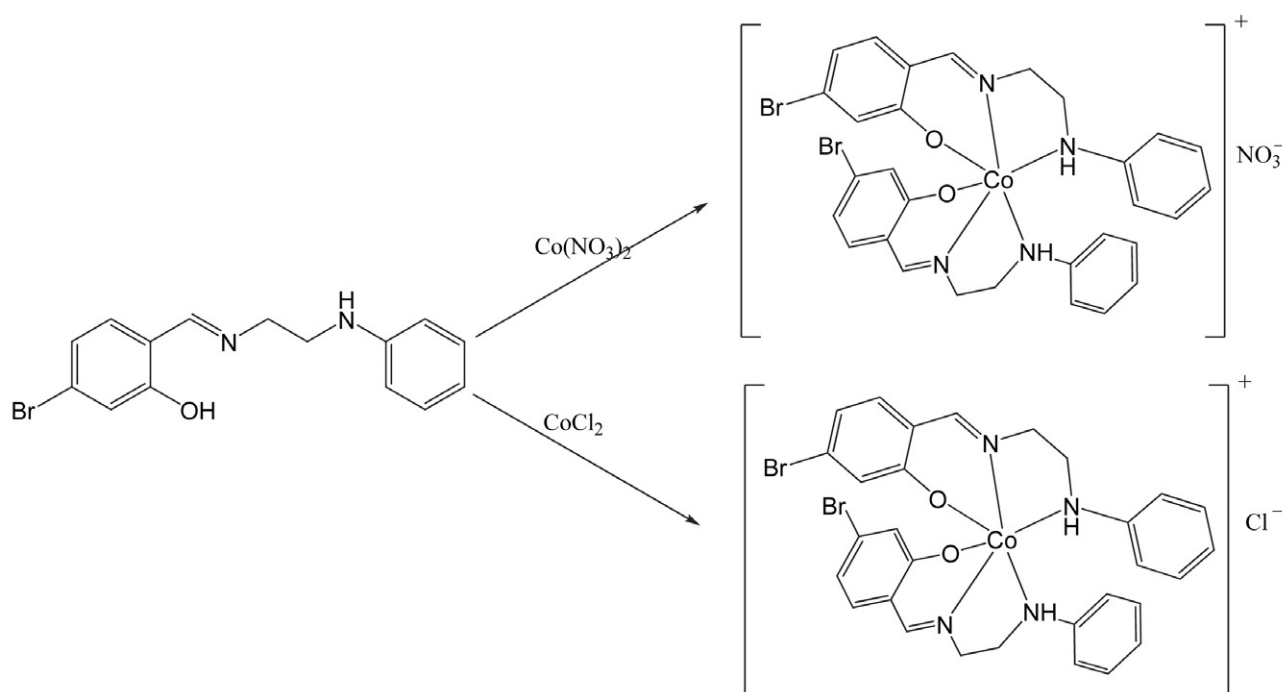
	1	2
Chemical Formula	C ₃₀ H ₂₈ Br ₂ CoN ₅ O ₅	C ₃₀ H ₂₈ Br ₂ ClCoN ₄ O ₂
Molecular weight	757.32	730.76
Crystal color, habit	Brown, block	Brown, block
Crystal size, mm	0.23 × 0.22 × 0.20	0.26 × 0.23 × 0.23
Crystal system	Monoclinic	Monoclinic
Space group	Cc	Cc
Unit cell dimensions:		
<i>a</i> , Å	10.667(2)	10.4703(11)
<i>b</i> , Å	19.0095(16)	19.0897(13)
<i>c</i> , Å	15.1924(11)	15.1449(12)
β, °	100.151(1)	103.973(1)
<i>V</i> , Å ³	3032.5(7)	2937.5(4)
<i>Z</i>	4	4
ρ _{calcd} , g cm ⁻³	1.659	1.652
μ, mm ⁻¹	3.249	3.431
θ Range collected, deg	2.143–25.493	2.134–25.496
<i>T</i> _{min} and <i>T</i> _{max}	0.522 and 0.563	0.469 and 0.506
Reflections collected/unique	7800/4645	7793/4102
Observed reflections (<i>I</i> ≥ 2σ(<i>I</i>))	3526	3496
Data/restraints/parameters	4645/4/394	4102/4/370
GOOF on <i>F</i> ²	0.983	0.970
<i>R</i> ₁ , <i>wR</i> ₂ (<i>I</i> ≥ 2σ(<i>I</i>))	0.0556, 0.1445	0.0328, 0.0603
<i>R</i> ₁ , <i>wR</i> ₂ (all data)	0.0740, 0.1558	0.0435, 0.0644

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

	1	2
Co1-O1	1.866(8)	1.884(4)
Co1-O2	1.881(8)	1.875(4)
Co1-N1	1.888(8)	1.909(5)
Co1-N2	2.021(9)	2.049(5)
Co1-N3	1.908(9)	1.899(5)
Co1-N4	2.021(10)	2.027(5)
O1-Co1-O2	90.7(4)	89.79(18)
O1-Co1-N1	94.1(4)	93.88(19)
O2-Co1-N1	86.6(3)	86.12(18)
O1-Co1-N3	85.7(4)	85.63(18)
O2-Co1-N3	95.3(4)	95.34(19)
N1-Co1-N3	178.0(4)	178.5(2)
O1-Co1-N2	177.7(4)	176.9(2)
O2-Co1-N2	87.2(4)	87.2(2)
N1-Co1-N2	84.9(4)	85.3(2)
N3-Co1-N2	95.3(4)	95.24(19)
O1-Co1-N4	88.4(4)	89.39(19)
O2-Co1-N4	179.0(4)	179.1(2)
N1-Co1-N4	93.9(4)	93.6(2)
N3-Co1-N4	84.2(4)	84.9(2)
N2-Co1-N4	93.7(4)	93.6(2)

2. 5. Antibacterial Activity

Antibacterial activity of the complexes was tested against *B. subtilis*, *S. aureus*, *S. faecalis*, *P. aeruginosa*, *E. coli*, and *E. cloacae* using MTT medium. The minimum inhibitory concentrations (MICs) of the compounds were determined by a colorimetric method using MTT dye.¹⁰ A stock solution of the compounds ($50 \mu\text{g mL}^{-1}$) in DMSO was prepared and quantities of the compounds were incorporated in specified quantity of sterilized liquid medium. A specified quantity of the medium containing the compounds was poured into micro-titration plates. 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 for bacteria. After the MICs were visually determined on each micro-titration plate, $50 \mu\text{L}$ of phosphate buffered saline (PBS 0.01 mol L^{-1} , pH 7.4: $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ 2.9 g, KH_2PO_4 0.2 g, NaCl 8.0 g, KCl 0.2 g, distilled water 1000 mL) containing 2 mg mL^{-1} of MTT was added to each well. Incubation was continued at room temperature for 4–5 h. The content of each well was removed, and $100 \mu\text{L}$ of isopropanol containing 5% 1 mol L^{-1} HCl was added to extract the dye. After 12 h of in-

**Scheme 1.** The synthetic procedure for HL.**Scheme 2.** The synthetic procedure for the complexes.

cupation at room temperature, the optical density (OD) was measured with a microplate reader at 570 nm.

3. Results and Discussion

3.1. Chemistry

The Schiff base HL was readily prepared by the reaction of 4-bromosalicylaldehyde and *N*-phenylethane-1,2-diamine in methanol (Scheme 1). The complexes were prepared in a similar method, by the reaction of the Schiff base ligand with cobalt nitrate and cobalt chloride, respectively (Scheme 2). The conductivity values of the complexes (128–135 $\Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$) indicated that they are 1:1 electrolytes in methanol solution.¹¹

3.2. IR and UV-Vis Spectra Study

The weak absorptions at 3155 and 3167 cm^{-1} for the infrared spectra of complexes **1** and **2** are assigned to $\nu_{\text{N-H}}$. The characteristic imine stretching for the complexes are observed at 1650 cm^{-1} for **1** and 1648 cm^{-1} for **2**.¹² The Schiff base ligand coordination through the phenolate oxygen is indicated by the absorption bands of the Ar–O bonds at about 1190 cm^{-1} in the complexes.¹³ In general, the infrared spectra of complexes **1** and **2** are similar to each other, except the absorption at 1382 cm^{-1} for the nitrate anion of complex **1**.¹⁴

The absorption spectral data of the complexes were measured in methanol solution. In both complexes, peaks between 240–250 nm, 260–310 nm and 380–390 nm are assigned to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and ligand to metal charge transfer transitions, respectively.¹² In the visible spectra of both complexes, weak bands are observed at 495–497 nm.

3.3. Structure Description of the Complexes

Molecular structures of complexes **1** and **2** are shown in Figs. 1 and 2, respectively. Complex **1** contains a $[\text{CoL}_2]^+$ cation and a nitrate anion. Complex **2** contains a $[\text{CoL}_2]^+$ cation and a chloride anion. In the cation, the Co atom is coordinated by two phenolate oxygen (O1, O2), two imino nitrogen (N1, N3) and two amino nitrogen (N2, N4) from two Schiff base ligands, forming octahedral geometry. In general, the coordination geometry around the Co atom in each complex displays only slight distortion. In both complexes, the bond lengths of Co–O and Co–N are similar and range from 1.866(8) to 2.021(10) Å for complex **1**, and range from 1.875(4) to 2.049(5) Å for complex **2**. The greatest deviations of the bond angles from those expected for an ideal octahedral geometry are 84.2(4)° for N3–Co1–N4 and 95.3(4)° for O2–Co1–N3 for complex **1**, and 84.9(2)° for N3–Co1–N4 and 95.3(2)° for O2–Co1–N3 for complex **2**. The remaining bond angles around the Co atoms are nearly close to the ideal values for octahedral geometry. Moreover, the coordinate bond values are comparable to those ob-

served in similar Schiff base cobalt(III) complexes with octahedral geometry.¹⁵ The average deviation (0.033(4) Å for complex **1**, 0.037(3) Å for complex **2**) of the four donor atoms (O1, N1, N2, N3) and the displacement (0.001(2) Å for Co1 and 0.013(3) Å for Co2) of the Co atoms from the least-squares planes defined by the four donor atoms indicate that the ON₃ cavities afford almost perfect planes to the Co centers. The Schiff base ligands are twisted with dihedral angles between the benzene rings of 85.8(5) and 84.9(5)° for complex **1**, and 89.1(5) and 85.8(5)° for complex **2**.

In the crystal structure of complex **1** (Fig. 3), the nitrate anions are linked to the complex cations through N–H...O and C–H...O hydrogen bonds. In the crystal structure of complex **2** (Fig. 4), the chloride anions are linked to the complex cations through N–H...Cl and C–H...Cl hydrogen bonds. In addition, the molecules in both complexes are stack along the *a* axis via $\pi \cdots \pi$ interactions among the rings Co1–N1–C8–C9–N2, C1–C2–C3–C4–C5–C6, C10–C11–C12–C13–C14–C15 and C16–C17–C18–C19–C20–C21.

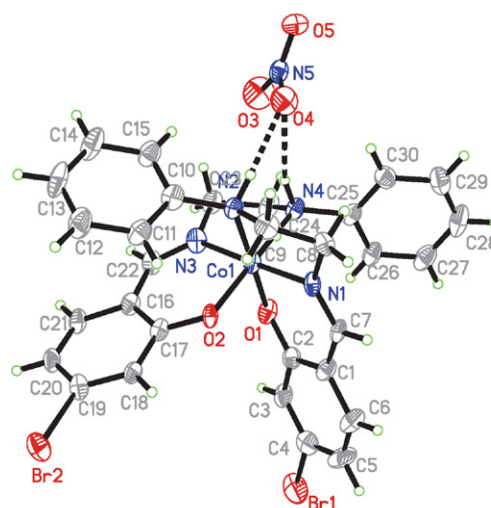


Fig. 1. Molecular structure of complex **1**.

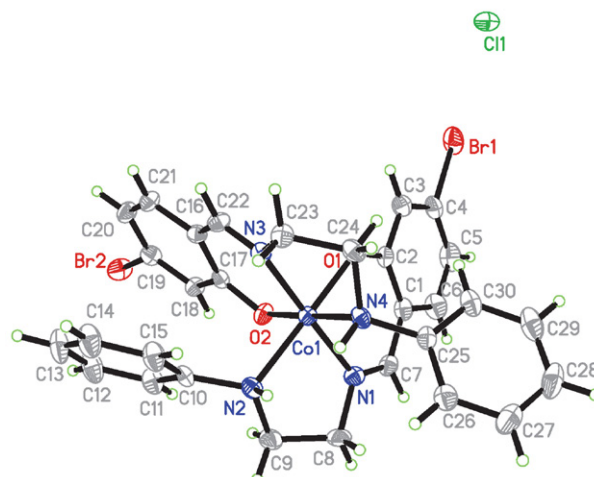


Fig. 2. Molecular structure of complex **2**.

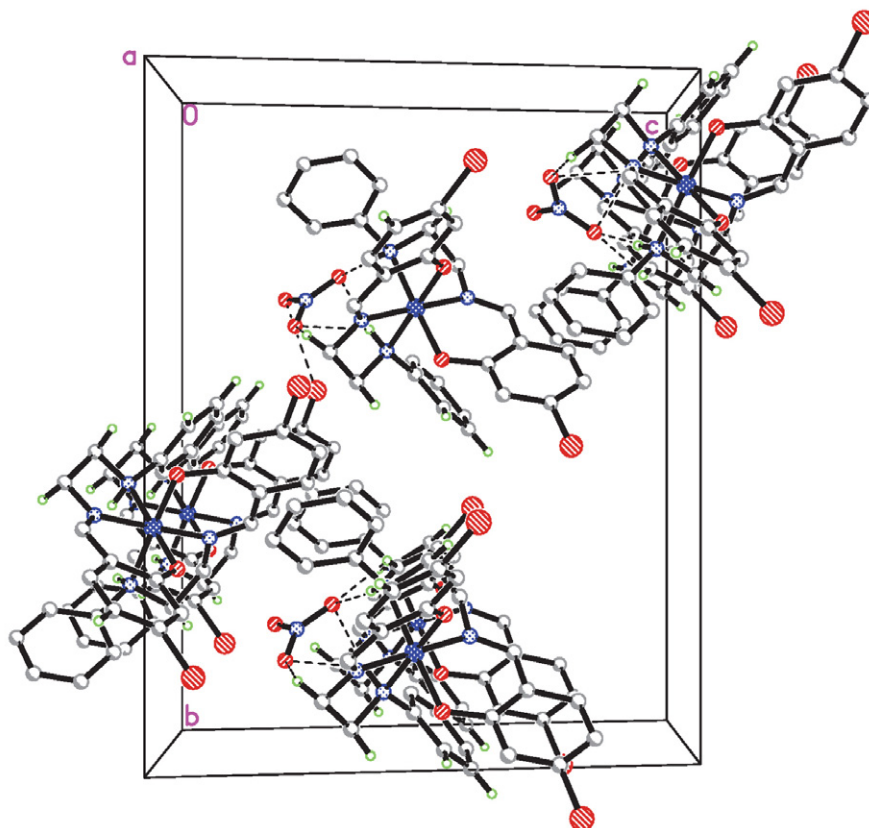


Fig. 3. Molecular packing diagram of complex 1, viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines.

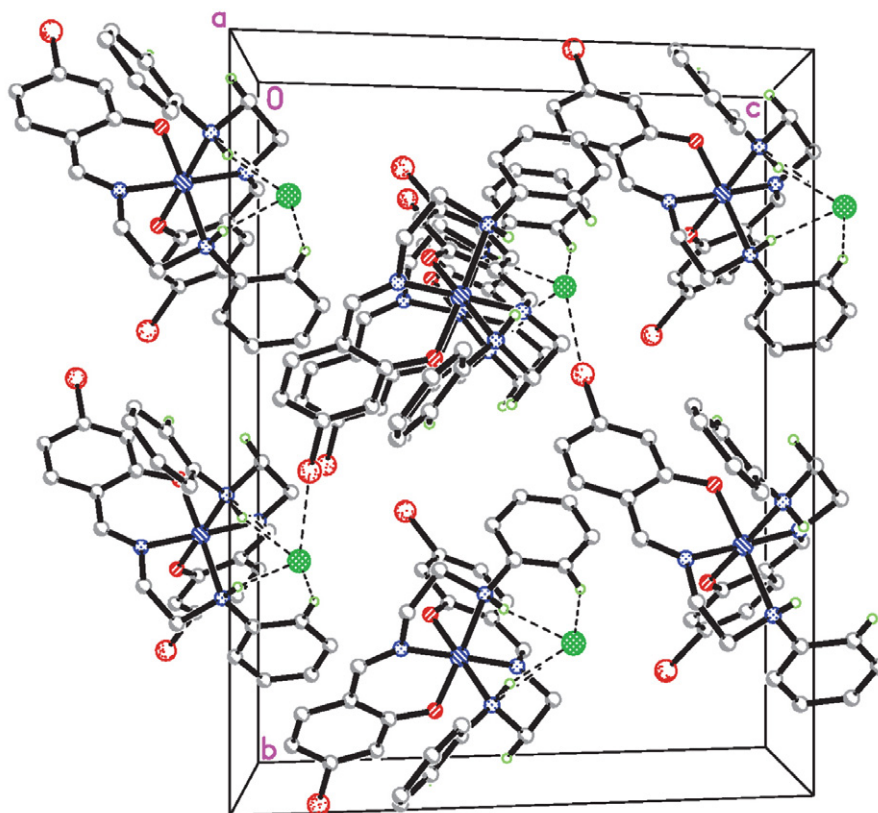


Fig. 4. Molecular packing diagram of complex 2, viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines.

3. 4. Antibacterial Activity of the Compounds

The cobalt complexes were screened for antibacterial activities against three Gram-positive bacterial strains (*B. subtilis*, *S. aureus*, and *St. faecalis*) and three Gram-negative bacterial strains (*E. coli*, *P. aeruginosa*, and *E. cloacae*) by MTT method. The MICs of the compounds against the bacteria are presented in Table 3. Penicillin and Kanamycin were tested as reference drugs. The cobalt complexes have equal activities against all the bacterial strains. Both complexes showed strong activity against *B. subtilis*, *S. aureus* and *E. coli* (MICs = 0.39–3.12 $\mu\text{g mL}^{-1}$), weak activity against *St. faecalis* (MIC = 25 $\mu\text{g mL}^{-1}$), and no activity against *P. aeruginosa* and *E. cloacae*. Notably, both complexes have similar or better activity against *B. subtilis*, *S. aureus* and *E. coli* than the reference drugs.

Both complexes have stronger activities against *S. aureus* and *P. aeruginosa* than the cobalt(II) complexes with the Schiff bases, (4-X-2-[[2-(2-pyridine-2-yl-ethylsulfanyl)ethylimino]methyl]phenol (X = methoxy (OMe), phenylazo (N_2Ph), bromo (Br), nitro (NO_2)),¹⁶ and against *B. subtilis*, *S. aureus* and *E. coli* than the oxidovanadium(V) complexes with hydrazone ligands.¹⁷ The complexes have similar activities against *B. subtilis*, *S. aureus* and *E. coli*, but weak activities against *St. faecalis*, *P. aeruginosa* and *E. cloacae* when compared with the manganese complex with the Schiff base N,N' -bis(3,5-dichlorosalicylidene)-1,4-butanediamine.¹⁸

4. Conclusion

Two new cobalt(III) complexes with the Schiff base 5-bromo-2-((2-(phenylamino)ethylimino)methyl)phenol have been synthesized and characterized. Crystal structures of the complexes are determined and described. Both complexes are mononuclear cobalt species with octahedral coordination. The anions of the cobalt salts used in the preparation of the complexes act as the counteranions of the final structures. Both complexes have effective antibacterial activities against *B. subtilis*, *S. aureus* and *E. coli*.

Supplementary Materials

The crystal data for the complexes have been deposited with the Cambridge Crystallographic Data Centre

(CCDC nos. 2086791 (1) and 2086793 (2); deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

Acknowledgment

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Table 3. MICs ($\mu\text{g mL}^{-1}$) of the compounds and related materials

Tested material	Gram positive			Gram negative		
	<i>B. subtilis</i>	<i>S. aureus</i>	<i>St. faecalis</i>	<i>P. aeruginosa</i>	<i>E. coli</i>	<i>E. cloacae</i>
HL	12.5	6.25	> 50	> 50	12.5	> 50
1	1.56	0.39	25	> 50	3.12	> 50
2	1.56	0.39	25	> 50	3.12	> 50
Penicillin	1.56	1.56	1.56	6.25	6.25	3.12
Kanamycin	0.39	1.56	3.12	3.12	3.12	1.56

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Povzetek

Sintetizirali smo dva strukturno podobna kompleksa Co(III), $[\text{CoL}_2] \cdot \text{NO}_3$ (**1**) in $[\text{CoL}_2] \cdot \text{Cl}$ (**2**), kjer je L = 5-bromo-2-((2-(fenilamino)etilimino)metil)fenolat, ter produkta karakterizirali z IR in UV-Vis spektroskopijo. Strukture kompleksov smo določili z monokristalno rentgensko difrakcijo. Kobaltovi atomi v kompleksih so oktaedrično koordinirani, pri čemer so donorski atomi iz dveh ligandov: kisik iz fenolne skupine ter dušikova atoma iz imino in amino skupine. Anioni kobaltovih soli kristalizirajo kot protiioni. Antibakterijsko učinkovitost kompleksov smo preverili z metodo MTT.



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