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Synthesis, crystal structures and antibacterial activities of nickel(II) complexes derived from 2-Bromo-4-chloro-6[(2-diethylaminoethylimino)methyl]phenol

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ABSTRACT

Two new nickel(II) complexes, $[\text{Ni}(\text{HL})_2] \cdot 2\text{NO}_3$ (**1**) and $[\text{NiL}(\text{N}_3)]$ (**2**), where HL and L are 2-bromo-4-chloro-6[(2-diethylaminoethylimino)methyl]phenolate and 2-bromo-4-chloro-6[(2-diethylaminoethylimino)methyl]phenolate, respectively, have been synthesized and characterized. The complexes were characterized by elemental analyses, IR spectra, molar conductivity, and single crystal X-ray diffraction. The complexes and the free Schiff base were assayed 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.

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Schiff base; nickel(II) complex; X-ray diffraction; antibacterial activity

Introduction

Schiff bases represent one of the most widely utilized classes of ligands in metal coordination chemistry. They offer versatile and flexible ligands capable of binding various metal ions to give complexes with versatile structures and properties.^[1–5] Over the past few decades considerable study has been made on the chemistry of nickel(II) complexes derived from Schiff base ligands due to their important role in catalytic, magnetic and biological properties.^[6–10] The Schiff bases have good complexing ability and their biological activity increases on complex with metal ions.^[11–13] Nickel activates certain enzymes related to the breakdown or utilization of glucose. Nickel aid in prolactin production, iron absorption as well as adrenaline and glucose metabolism, hormones, lipids, cell membrane, improve bone strength and may also a role in production of red blood cells. Nickel present in RNA in our body where it functions in association with nucleic acids and probably has a role in stabilizing RNA structure.^[14] An important aspect of Ni-salen type complexes is their antibacterial application.^[15–18] Recent reports indicate that halido groups are positive factors for the antimicrobial activities.^[19] We report here the synthesis, characterization including single crystal X-ray structures of two new nickel(II) complexes, $[\text{Ni}(\text{HL})_2] \cdot 2\text{NO}_3$ (**1**) and $[\text{NiL}(\text{N}_3)]$ (**2**), where HL and L are 2-bromo-4-chloro-6[(2-diethylaminoethylimino)methyl]phenolate and 2-bromo-4-chloro-6[(2-diethylaminoethylimino)methyl]phenolate. The antibacterial activity 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 was studied.

Experimental

Materials and physical methods

3-Bromo-5-chlorosalicylaldehyde and *N,N*-diethylethane-1,2-diamine were purchased from Sigma-Aldrich. The Schiff base compound HL was prepared by 1:1 condensation of 3-bromo-5-chlorosalicylaldehyde with *N,N*-diethylethane-1,2-diamine in methanol, according to the literature method.^[20] All the other reagents and solvents were purchased from Beijing Chemical Co. Ltd. and used as received. FT-IR spectra were recorded as KBr pellets on Bruker Tensor-27. Elemental (C, H, and N) analyses were performed on a Perkin-Elmer 2400 II analyzer. Single crystal X-ray diffraction was carried out with a Bruker Apex II CCD diffractometer. Molar conductivity of the complexes in acetonitrile was measured with a DDS-11A molar conductivity meter.

Caution! Perchlorate and azide complexes of metal ions are potentially explosive. Only a small amount of material should be prepared, and they should be handled with caution.

Synthesis of Complex 1

To a stirred suspension of HL (0.332 g, 1.00 mmol) and sodium azide (0.065 g, 1.00 mmol) in methanol (20 mL) was added dropwise a methanol solution (10 mL) of nickel nitrate hexahydrate (0.291 g, 1.00 mmol). After a few minutes a green solution was formed. After one hour stirring, the solution was filtered and the filtrate was kept for slow evaporation. The diffraction quality green single crystals that deposited after 7 days were collected by filtration and washed with methanol. The yield was 177 mg (42%).

Anal. Calcd. for $C_{52}H_{72}Br_4Cl_4N_{12}Ni_2O_{16}$ (%): C, 36.74; H, 4.27; N, 9.89. Found (%): C, 36.57; H, 4.36; N, 9.81.

Synthesis of Complex 2

To a stirred suspension of HL (0.332 g, 1.00 mmol) and sodium azide (0.065 g, 1.00 mmol) in methanol (20 mL) was added dropwise a methanol solution (10 mL) of nickel nitrate perchlorate (0.366 g, 1.00 mmol). After a few minutes a red solution was formed. After one hour stirring, the solution was filtered and the filtrate was kept for slow evaporation. The diffraction quality red single crystals that deposited after 5 days were collected by filtration and washed with methanol. The yield was 177 mg (42%). Anal. Calcd. for $C_{13}H_{17}BrClN_5NiO$ (%): C, 36.03; H, 3.95; N, 16.16. Found (%): C, 36.21; H, 4.03; N, 16.08.

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 ($\lambda = 0.71073 \text{ \AA}$). For data processing and absorption correction the packages SAINT and SADABS were used.^[21] Structures of the complexes were solved by direct and Fourier methods and refined by full-matrix least-squares based on F^2 using SHELXL-97 package.^[22] The non-hydrogen atoms were refined anisotropically. The hydrogen atoms have been placed at geometrical positions with fixed thermal parameters. Crystallographic data of the complexes **1** and **2** are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

Antibacterial activity

Antibacterial activity of the complexes was tested against *B. subtilis*, *S. aureus*, *S. faecalis*, *P. aeruginosa*, *E. coli*, and *E. cloacae* using 3-(4,5-dimethyl-2-thiazyl)-2,5-diphenyl-2H-tetrazolium bromide (MTT) medium. The minimum inhibitory concentrations (MICs) of the compounds were determined by a colorimetric method using MTT dye.^[23] 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: $Na_2HPO_4 \cdot 12H_2O$ 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 incubation at room

Table 1. Crystallographic data and refinement details for complexes **1** and **2**.

| | 1 | 2 |
|--|--------------------------------|--------------------------------|
| Molecular weight | 1700.08 | 433.39 |
| Crystal color, habit | Green, block | Red, block |
| Crystal size, mm | $0.31 \times 0.28 \times 0.27$ | $0.19 \times 0.18 \times 0.18$ |
| Crystal system | Triclinic | Monoclinic |
| Space group | $P\bar{1}$ | $P2_1/c$ |
| Unit cell dimensions: | | |
| $a, \text{ \AA}$ | 13.4598(11) | 10.8120(14) |
| $b, \text{ \AA}$ | 13.7286(11) | 11.6596(15) |
| $c, \text{ \AA}$ | 20.7128(17) | 13.2663(17) |
| $\alpha, ^\circ$ | 87.660(2) | 90 |
| $\beta, ^\circ$ | 89.539(2) | 100.660(2) |
| $\gamma, ^\circ$ | 61.693(1) | 90 |
| $V, \text{ \AA}^3$ | 3366.6(5) | 1643.5(4) |
| Z | 2 | 4 |
| $\rho_{\text{calcd}}, \text{ g cm}^{-3}$ | 1.677 | 1.751 |
| $\mu, \text{ mm}^{-1}$ | 3.163 | 3.782 |
| θ Range collected, deg | 0.98–25.50 | 2.34–25.50 |
| T_{min} and T_{max} | 0.4405 and 0.4821 | 0.5335 and 0.5493 |
| Reflections collected/unique | 20305/12538 | 9455/3053 |
| Observed reflections ($I \geq 2\sigma(I)$) | 5826 | 2314 |
| Data/restraints/parameters | 12538/1/819 | 3053/0/201 |
| GOOF on F^2 | 0.992 | 1.033 |
| R_1, wR_2 ($I \geq 2\sigma(I)$) | 0.0689, 0.1748 | 0.0367, 0.0945 |
| R_1, wR_2 (all data) | 0.1662, 0.2459 | 0.0578, 0.1059 |
| Largest differences in peak/hole, $e \text{ \AA}^{-3}$ | 0.767/−1.095 | 0.534/−0.496 |

Table 2. Selected bond distances (\AA) and angles ($^\circ$) for complexes **1** and **2**.

| 1 | | | |
|-----------------|------------|-----------------|------------|
| Ni(1)–O(1) | 1.922(5) | Ni(1)–O(2) | 1.912(5) |
| Ni(1)–N(1) | 2.018(7) | Ni(1)–N(3) | 2.022(7) |
| Ni(2)–O(3) | 1.901(5) | Ni(2)–O(4) | 1.899(6) |
| Ni(2)–N(5) | 1.995(8) | Ni(2)–N(7) | 1.999(7) |
| O(2)–Ni(1)–O(1) | 171.9(3) | O(2)–Ni(1)–N(1) | 88.4(2) |
| O(1)–Ni(1)–N(1) | 91.3(2) | O(2)–Ni(1)–N(3) | 91.8(3) |
| O(1)–Ni(1)–N(3) | 88.8(3) | N(1)–Ni(1)–N(3) | 178.5(3) |
| O(4)–Ni(2)–O(3) | 171.3(3) | O(4)–Ni(2)–N(5) | 88.8(3) |
| O(3)–Ni(2)–N(5) | 90.9(3) | O(4)–Ni(2)–N(7) | 90.8(3) |
| O(3)–Ni(2)–N(7) | 89.6(3) | N(5)–Ni(2)–N(7) | 179.4(3) |
| 2 | | | |
| Ni(1)–O(1) | 1.893(3) | Ni(1)–N(1) | 1.930(3) |
| Ni(1)–N(2) | 2.070(3) | Ni(1)–N(3) | 1.945(4) |
| O(1)–Ni(1)–N(1) | 93.57(13) | O(1)–Ni(1)–N(3) | 92.70(14) |
| N(1)–Ni(1)–N(3) | 165.40(16) | O(1)–Ni(1)–N(2) | 171.45(13) |
| N(1)–Ni(1)–N(2) | 84.32(14) | N(3)–Ni(1)–N(2) | 91.30(15) |

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

Results and discussion

Chemistry

Reaction of the Schiff base 2-bromo-4-chloro-6[(2-diethylaminoethylimino)methyl]phenol with nickel nitrate hexahydrate and nickel perchlorate hexahydrate in the presence of sodium azide in methanol produced the mononuclear nickel(II) complexes. The mere difference for the starting material is the nickel salts, *viz.* nickel nitrate hexahydrate for **1** and nickel perchlorate hexahydrate for **2**. The different anions of the nickel salts influence the coordination of the azide group. In complex **1**, the azide anion did not participate in coordination, with the Schiff base ligand in zwitterionic form. In complex **2**, the azide anion adopt terminal coordination mode, with the Schiff base ligand in deprotonated form. The conductivities of the complexes **1** and **2** in

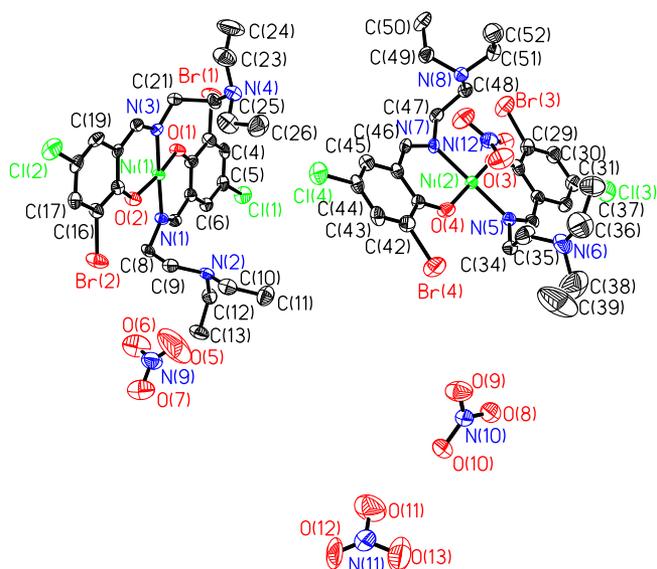


Figure 1. Molecular structure of complex 1.

methanol at concentration of $10^{-3} \text{ mol}\cdot\text{L}^{-1}$ are 245 and $27 \Omega^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$, indicating that the 1:2 electrolyte and non-electrolyte nature of them.^[24]

IR spectra

The characteristic imino stretching of the complexes is observed at 1605 cm^{-1} for **1** and 1607 cm^{-1} for **2** as strong signal.^[25] In the spectrum of complex **1**, the stretching band of NH group is observed at 3271 cm^{-1} . The band at 1382 cm^{-1} is due to nitrate anion.^[26] In the spectrum of complex **2**, appearance of intense band at 2015 cm^{-1} indicates the presence of azide ligand.^[27] The Schiff base ligands coordination is substantiated by the lowering of the phenolic C–O stretching band, which appears at $1180\text{--}1185 \text{ cm}^{-1}$ in the complexes, while observed at 1203 cm^{-1} in the free Schiff base.^[28] Coordination of the Schiff bases is further confirmed by the appearance of weak bands in the low wave numbers $400\text{--}600 \text{ cm}^{-1}$, corresponding to $\nu(\text{Ni}\text{--}\text{N})$ and $\nu(\text{Ni}\text{--}\text{O})$.

Structure description of the complexes

Molecular structure of complex **1** is shown in Figure 1. The asymmetric unit of the compound contains two $[\text{Ni}(\text{HL})_2]^{2+}$ cations and four nitrate anions. The Ni centre is four coordinated by two phenolate oxygen and two imino nitrogen from two zwitterionic Schiff base ligands HL. The coordination environment of the Ni atom is slightly distorted, as evidenced from the bond lengths and angles. The *trans* angles range from $171.3(3)^\circ$ to $178.5(3)^\circ$, and the *cis* angles from $88.8(3)$ to $91.8(3)^\circ$, deviate slightly from the ideal values. The coordinate bond values are in agreement with those observed in nickel(II) complexes with Schiff bases.^[29–31] In the crystal structure of the complex (Figure 2), the nitrate anions are linked to the complex cations through hydrogen bonds (N(8)–H(8C)···O(16): N(8)–H(8C) 0.91 Å, H(8C)···O(16) 2.44 Å, N(8)···O(16) 3.144(11) Å,

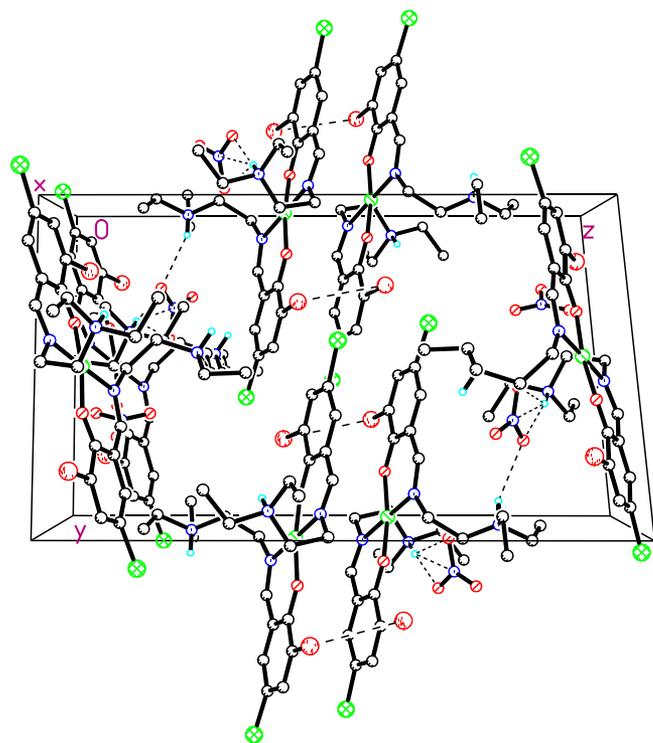


Figure 2. Molecular packing structure of complex 1. Hydrogen bonds are depicted by dashed lines.

N(8)–H(8C)···O(16) $134.5(3)^\circ$; N(8)–H(8C)···O(15): N(8)–H(8C) 0.91 Å, H(8C)···O(15) 1.88 Å, N(8)···O(15) 2.750(11) Å, N(8)–H(8C)···O(15) $158.9(3)^\circ$; N(6)–H(6A)···O(9)ⁱ: N(6)–H(6A) 0.91 Å, H(6A)···O(9)ⁱ 2.50 Å, N(6)···O(9)ⁱ 3.368(15) Å, N(6)–H(6A)···O(9)ⁱ $158.8(3)^\circ$; N(4)–H(4A)···O(13)ⁱⁱ: N(4)–H(4A) 0.91 Å, H(4A)···O(13)ⁱⁱ 2.11 Å, N(4)···O(13)ⁱⁱ 2.953(17) Å, N(4)–H(4A)···O(13)ⁱⁱ $153.7(3)^\circ$; N(2)–H(2A)···O(8)ⁱⁱ: N(2)–H(2A) 0.91 Å, H(2A)···O(8)ⁱⁱ 2.39 Å, N(2)···O(8)ⁱⁱ 3.094(10) Å, N(2)–H(2A)···O(8)ⁱⁱ $134.0(3)^\circ$; N(2)–H(2A)···O(9)ⁱⁱ: N(2)–H(2A) 0.91 Å, H(2A)···O(9)ⁱⁱ 2.01 Å, N(2)···O(9)ⁱⁱ 2.881(11) Å, N(2)–H(2A)···O(9)ⁱⁱ $159.4(3)^\circ$; symmetry codes: ⁱ $x, 1 + y, z$; ⁱⁱ $1 - x, 1 - y, 1 - z$. The molecules are further linked through the above hydrogen bonds through *y* axis, to form chains. The chains are further linked through weak Br···Br interactions along the *z* axis.

Molecular structure of the complex **2** is shown in Figure 3. The Ni atom is four coordinated, with one phenolate oxygen, imino nitrogen and amino nitrogen of the deprotonated Schiff base ligand L, and one azido nitrogen, forming a slightly distorted square planar geometry. The coordination environment of the Ni atom is slightly distorted, as evidenced from the bond lengths and angles. The *trans* angles range from $165.4(2)^\circ$ to $171.4(1)^\circ$, and the *cis* angles from $84.3(1)$ to $93.6(1)^\circ$, deviate slightly from the ideal values. The coordinate bond values are in agreement with those observed in nickel(II) complexes with Schiff bases.^[32–34] In the crystal structure of the complex (Figure 4), molecules are linked through hydrogen bonds (O(7)–H(7)···N(5)ⁱⁱⁱ: O(7)–H(7) 0.93 Å, H(7)···N(5)ⁱⁱⁱ 2.57 Å, O(7)···N(5)ⁱⁱⁱ 3.4823(5) Å, O(7)–H(7)···N(5)ⁱⁱⁱ $168(6)^\circ$; symmetry code: ⁱⁱⁱ $x, 1/2 - y, -1/2 + z$) to form 1D chains running along the *z*

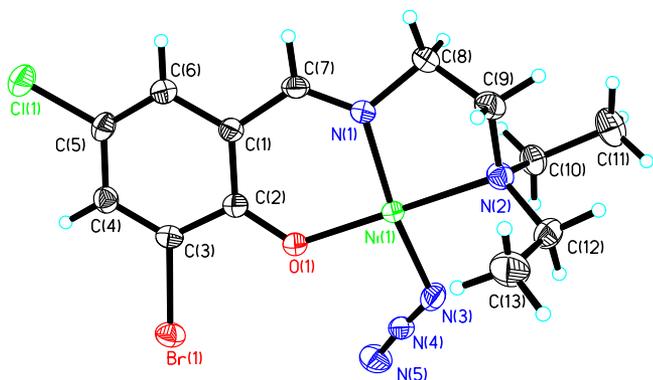


Figure 3. Molecular structure of complex 2.

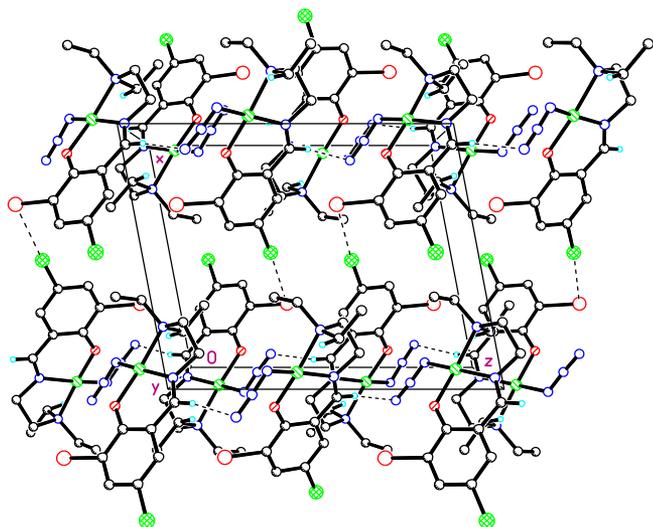


Figure 4. Molecular packing structure of complex 2. Hydrogen bonds are depicted by dashed lines.

axis. The chains are further linked through weak Br...Cl interactions along the x axis.

Antibacterial activity

The complexes and the free Schiff base 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. Complexes 1 and 2 show strong activities against *B. subtilis*, *S. aureus*, and *E. coli*, medium activity against *St. faecalis* and *P. aeruginosa*, and no activity against *E. cloacae*. Complex 1 has stronger activity against *S. aureus* and *E. coli* than Penicillin and Kanamycin. Complex 2 has equal activity against *B. subtilis* and stronger activity against *E. coli* than Penicillin. The Schiff base HL show medium activities against *B. subtilis*, *S. aureus*, and *E. coli*, and no activity against *St. faecalis*, *P. aeruginosa* and *E. cloacae*. In general, the antibacterial activities of the two complexes are higher than the free Schiff base. Complex 1 has the most activity against *S. aureus* and

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> |
| 1 | 3.12 | 0.78 | 25 | 12.5 | 1.56 | >50 |
| 2 | 1.56 | 3.12 | 25 | 25 | 3.12 | >50 |
| HL | 25 | 12.5 | >50 | >50 | 25 | >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 |

E. coli, with MIC values of 0.78 and $1.56 \mu\text{g mL}^{-1}$, respectively.

Conclusion

Thus, two new nickel(II) complexes with Schiff base ligands have been synthesized and characterized. Crystal structures of the complexes are described. The antibacterial assay of the free Schiff base and the two complexes show that the complexes, especially complex 1 are efficient drugs for bacteria strains *S. aureus* and *E. coli*.

Supplementary material for structures has been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1852355 (1) and 1852356 (2); deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

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