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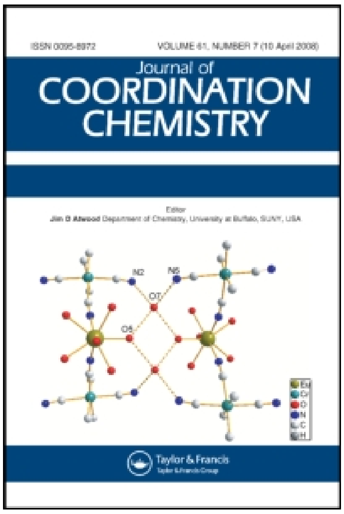

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Tetradentate asymmetric Schiff bases and their Ni(II) and Fe(III) complexes

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Tetradentate asymmetric Schiff bases and their Ni(II) and Fe(III) complexes

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Three asymmetric Schiff-base tetradentate diimines H_2L^1, H_2L^2, and H_2L^3 [$(2\text{-OH})C_6H_4N{=}CHC_6H_4 2\text{-}N{=}CHC_6H_3(2\text{-OH})(5\text{-X})$, X = H, CH_3, Cl respectively] have been synthesized by a two step process. The reaction of 2-hydroxy aniline with 2-*nitro*-benzaldehyde in EtOH gave the starting Schiff base, 2-hydroxy-*N*-(2-nitrobenzylidene)aniline (SB-NO_2), which was reduced into the amino derivative (SB-NH_2) in solution. Reacting SB-NH_2 with 2-hydroxybenzaldehyde, 2-hydroxy-5-methylbenzaldehyde and 2-hydroxy-5-chlorobenzaldehyde gave the three new ligands H_2L^1, H_2L^2, and H_2L^3 respectively. Their dimeric, binuclear metal complexes with Ni(II) and Fe(III) have also been synthesized. The ligands and their complexes were characterized by elemental analyses, LC–MS, IR, electronic, ^1H and ^{13}C-NMR spectra, TGA, conductivity and magnetic measurements. All of the spectroscopic, analytical and other data indicate octahedral geometry $M_2L_2(H_2O)X_2$ (M: Ni,Co;X: Cl or H_2O), except for NiL^2 which is monomeric. Antimicrobial activities of the ligands and the complexes were evaluated against five bacteria. While the ligands and the Ni complexes are inactive towards *Pseudomonas aeruginosa* and *Staphylococcus aureus*, Fe complexes are active; only Fe complexes are inactive against *Escherichia coli*. All of the compounds have antimicrobial activities against *Bacillus subtilis*, and *Yersinia enterecolitica*.

Keywords: Asymmetric Schiff bases; Dimeric Fe(II) complexes; Nickel(II) complexes; Reduction; Antimicrobial activities

1. Introduction

Schiff bases, formed by reaction of primary amines with aromatic aldehydes have interesting biological [1, 2], anticancer [3, 4] and antioxidant activities [5, 6], complex stabilities [7, 8] and so on.

Schiff bases with two imine groups in an aromatic ring can be synthesized by condensation of diamines with aldehydes [9, 11], or condensation of dialdehydes with amines [12, 13], thus the symmetric Schiff bases (–C=N–Ar–N=C–) and (–N=C–Ar–C=N–) are obtained. Since the condensation occurs between NH_2 and CHO groups, a stable compound including both of these groups in an aromatic ring could not be synthesized.

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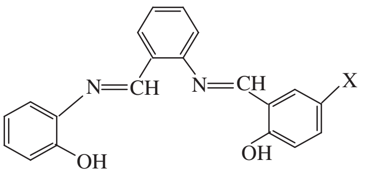


H_2L

X	H_2L
–H	H_2L^1
–CH_3	H_2L^2
–Cl	H_2L^3

Figure 1. Structures of synthesized Schiff bases.

In the present work we report the preparation of three new asymmetric Schiff bases (figure 1) with a new method and synthesis and characterization of their Ni(II) and Fe(III) complexes. All the synthesized compounds were investigated for their antibacterial activity against *Pseudomonas aeruginosa* ATCC 29212, *Staphylococcus aureus* ATCC 25973, *Escherichia coli* ATCC 11230, *Bacillus subtilis* ATCC6633 and *Yersinia enterecolitica* ATCC1501.

2. Experimental

2.1. *Reagent and instrumentation*

All reagents were commercially available and used without further purification. ^{1}H and ^{13}C-NMR spectra were recorded with a Bruker Avance DPX-400 using TMS as internal standard and DMSO-d_6 as solvent. IR spectra in the 4000–400 cm^{-1} ranges were recorded using KBr discs on a Mattson-1000 FTIR spectrophotometer. Carbon, hydrogen and nitrogen analyses were obtained using a LECO CHNS-932 analyzer. Metal ions were determined with a Perkin Elmer Analyst 800 model AAS. Uv–vis spectra were taken on a Shimadzu UV-160 A spectrophotometer and mass spectra were recorded at 70 eV on an Agilent 1100 MSD mass spectrometer. The TGA curves were obtained a Du Pont Instrument 951 between 35–800°C at a heating rate of 10°C min^{-1} in nitrogen. Molar conductivities in DMF (10^{-3} mol L^{-1}) at 20°C were measured using a Siemens WPA CM 35 apparatus. The room temperature magnetic susceptibilities of complexes were determined using a Sherwood Scientific MKI model Evans magnetic balance.

2.2. *Synthesis of the ligands*

Asymmetric Schiff bases were prepared as shown in figure 2. 2-Hydroxy-*N*-(2-nitrobenzylidene)aniline [figure 2(a)] was prepared by reacting equimolar amounts (50 mmol) of 2-hydroxy aniline with 2-nitro-benzaldehyde in EtOH (100 mL) [14, 15]. Yield: 94% (yellow; λ_M: 7.2 Ω^{-1}cm^{2}mol^{-1}; found/Calcd: C: 64.05/64.46, H: 4.13/4.27, N: 11.57/11.84; FTIR = ν_{OH}: 3372, $\nu_{CH=N}$: 1587, ν_{NO2}(asym/sym): 1522/1343, $\nu_{CH(arom)}$: 3037; ^{1}H/^{13}C-nmr = $\delta_{Ar–OH}$: 9.12 (1H, s)/154.4, $\delta_{CH=N}$: 8.90 (1H, s)/158.5, $\delta_{CH(arom)}$: 6.79–8.34 (8H, m)/117–155). The nitro group of (a) was reduced to amino with solid sodium dithionite, as reducing agent, thus (b) was obtained in solution [16, 17].

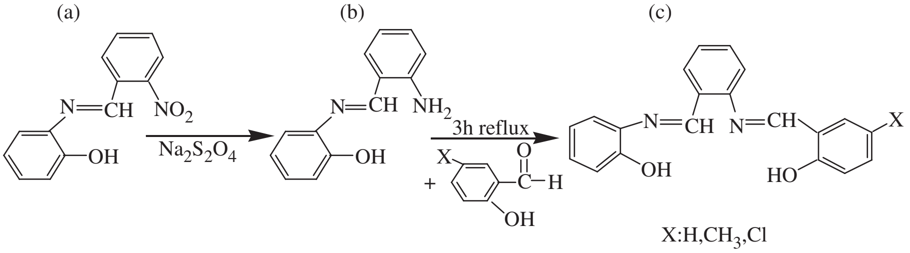


Figure 2. Reactions scheme for preparing the new asymmetric Schiff bases.

Finally appropriate aldehyde was added to the solution to form (c). In a typical procedure 5 mmol of solid sodium dithionite was slowly added to the solution of 1 mmol A in 50 mL ethanol: water (1 : 1) over an hour. The mixture was stirred for two hours at 45–55°C. To this solution 1mmol of appropriate aldehyde in 25 mL ethanol was added dropwise during the period of an hour with stirring. The mixture was stirred an additional three hours at 40–50°C. Slow evaporation of the mother liquor over three days at room temperature gave a deep yellow crystalline material (c). The crystals were filtered and recrystallized from ethanol.

2.3. *Synthesis of the complexes*

The metal complexes were synthesized by reaction of equimolar amounts (1 mmol) of asymmetric Schiff base ligands and Ni(II) or Fe(III) chlorides in ethanol. The mixture was refluxed on a water bath for ca 4–6 hours. Over a period of 7–10 days the solid complex obtained was filtered, washed with hot water, ethanol and ether, respectively, and dried in a vacuum dessicator over anhydrous $CaCl_2$.

2.4. *Bactericidal screening*

Antibacterial activities of 1000 and 500 µg of the synthesized compounds were studied using the well-diffusion method. The strains chosen were *Pseudomonas aeruginosa* ATCC 29212, *Staphylococcus aureus* ATCC 25973, *Escherichia coli* ATCC 11230, *Bacillus subtilis* ATCC6633 and *Yersinia enterecolitica* ATCC1501.

The bacteria cultures were incubated at 37°C for 18 h. Two stock solutions of 20 and $10\,\mathrm{mg\,mL^{-1}}$ in DMSO were prepared for all the ligands and the complexes. Molten nutrient agar (15 mL) kept at ca 45°C, was then poured in the petri-dishes and allowed to solidify. Holes of 6 mm diameter were punched carefully using a sterile cork borer and these were filled with 5 µL of the stock solutions. The plates were incubated for 24 h at 37°C. The mean value obtained for the two holes was used to calculate the zone of growth inhibition of each sample.

3. Results and discussion

Condensation of aromatic dialdehydes with primary amines or aromatic diamines with aldehydes give symmetrical diimines, N=C–Ar–C=N and C=N–Ar–N=C.

Herein, C=N–Ar–C=N asymmetric diimines H_2L^1, H_2L^2, and H_2L^3 are prepared and characterized by elemental analysis, electronic, IR, ^1H,^{13}C-NMR spectroscopy and mass spectra. The elemental analyses (table 1) agree with the chemical formulae of the compounds.

The IR spectra of the ligands show two strong bands in 1593 and 1619 cm^{-1} for H_2L^1, 1580 and 1593 cm^{-1} for H_2L^2 and 1589 and 1607 cm^{-1} for H_2L^3 assigned to the two asymmetric imine groups. The ν_{OH}, $\nu_{CH(ring)}$ and $\nu_{C=C(ring)}$ are observed at 3448, 3071, 1638; 3467, 3044, 1631 and 3416, 3063, 1631 cm^{-1}, respectively [18].

Electronic spectra of the ligands in ethanol showed bands in the ranges 222–230, 269–285 and 350–363 nm. The higher energy bands in the region 222–230 nm are assigned to π–π* transition of the aromatic rings. The medium energy bands (269–285 nm) are attributed to n–Π* transition of the imine. The lower energy bands in the region 350–363 nm are assigned to charge transfer transition [19].

^1H and ^{13}C-NMR spectral data of the ligands are assigned in table 2. ^1H-NMR spectra exhibit two different signals in the range 8.88–8.97 ppm and 9.66–10.22 ppm attributed to asymmetric imines due to different chemical environments. Furthermore, two singlets in the 9.65–10.53 ppm and 13.40–13.76 ppm ranges are assigned to the phenolic protons. OH signals are also defined with D_2O exchange. The signals within 6.60–6.70 ppm range are assigned to aromatic protons.

More detailed information about the structures of the new asymmetric Schiff bases is provided by the ^{13}C NMR spectral data. In the ^{13}C NMR spectra of the ligands two signals are observed for the asymmetric imine carbon atoms between 159.9–163.3 ppm for one and 159.5–162.0 ppm for the other.

Analysis by LC-Mass spectroscopy gave the molecular ion of the Schiff bases at the desired position *m/z*: 317 $(M+H^+)$, 332 $(M+2H^+)$ and 342 $(M-8H^+)$; respectively for the H_2L^1, H_2L^2 and H_2L^3. In the spectra of H_2L^1 and H_2L^2, the highest intensity peaks at *m/z* 214 (100%) and *m/z* 228 (100%) may be ascribed to elimination of C_7H_5N. The loss of the same fragment also occurred in the spectra of H_2L_3 at the position *m/z* 248 (28%). The peaks at *m/z* 108 (5.3%), *m/z* 120 (7.8%) and *m/z* 141 (3.7%) in the spectra of H_2L^1, H_2L^2 and H_2L^3, respectively, may be attributed to elimination of OH-C_6H_4N.

Reaction of the tetradentate asymmetric ONNO type ligands with Ni(II) and Fe(III) chlorides, gave dimeric $M_2L_2(H_2O)X_2$ (X : Cl or H_2O) complexes except for NiL^2 which was monomeric. All of the complexes are stable at room temperature, insoluble in water, slightly soluble in polar organic solvents such as acetone, methanol and soluble in DMF and DMSO. All complexes are amorphous solids and their melting points are above 350°C. The molar conductance values of the complexes measured in 10^{-3} M DMSO indicate nonelectrolytic nature [20].

Important IR spectral bands of the complexes are given in table 1. In the spectra of the complexes, two νCH=N vibrations are shifted to lower frequencies ca 10–30 cm^{-1}. These shifts confirm that imine nitrogens are coordinated to the metal ions. Broad bands in the 3275–3435 cm^{-1} region may be attributed to ν_{OH} of coordinated water. In addition, L^1Fe and L^2Fe complexes exhibit characteristic sharp bands of lattice water ca 3400 cm^{-1}, 882 cm^{-1} and 768 cm^{-1} assigned as ν_{OH}, ρ_{rOH} and ρ_{wOH_2} [21]. The $\nu_{(M–O)}$ and $\nu_{(M–N)}$ frequencies are observed in the 491–500 and 455–473 cm^{-1} range, respectively.

Table 1. Analytical, physical electronic and selected spectral data of the asymmetric Schiff bases and their metal complexes.

Compound	Empirical formula Decomp. Temp. (°C)	Color $\mu_{(BM)}$	Found (Calcd)%				λ_{max}(nm) $(\varepsilon)10^2$	ν_{OH} ν_{H_2O}	$\nu_{CH(ring)}$ $\nu_{C=C(ring)}$	$\nu_{C=N}$	ν_{M-N} ν_{M-O}
			C	H	N	M					
H_2L^1	$C_{20}H_{16}N_2O_2$	Orange	75.65	4.64	8.68	–	230 (64), 269 (41), 350 (45)	3448	3071	1619	–
	(143–145)	–	(75.95)	(5.06)	(8.86)		434 (10)	–	1638	1593	–
H_2L^2	$C_{21}H_{18}N_2O_2$	Orange	76.47	6.09	6.96	–	222 (111), 257 (39), 353 (46)	3467	3044	1593	–
	(148–150)	–	(76.36)	(5.45)	(8.48)		416 (25)	–	1631	1580	–
H_2L^3	$C_{20}H_{15}N_2O_2Cl$	Orange	68.26	4.87	7.72	–	223 (183), 283 (36), 363 (39)	3416	3063	1607	–
	(166–168)	–	(68.57)	(4.28)	(8.00)		420 (16)	–	1631	1589	–
FeL^1	$[Fe_2(C_{20}H_{14}N_2O_2)_2Cl_2(H_2O)_2$	Brown	55.05	4.13	6.82	12.92	226 (486), 280 (330), 370 (225)	–	3063	1589	455
	(>360)	6.05	(55.55)	(3.93)	(6.48)	(12.96)	507 (35)	3275	1606	1571	500
NiL^1	$[Ni_2(C_{20}H_{14}N_2O_2)_2(H_2O)_4$	Green	59.30	4.22	6.35	14.43	221 (213), 289 (76), 325 (69),	–	3025	1561	464
	(>360)	3.14	(58.82)	(4.41)	(6.86)	(14.21)	437 (29), 613 (0.02)	3423	1626	1522	482
FeL^2	$[Fe_2(C_{21}H_{16}N_2O_2)_2Cl_2(H_2O)_3$	Brown	55.98	4.81	5.82	12.99	238 (190), 289 (119), 334 (94),	3384	3027	1587	473
	(>360)	6.22	(56.50)	(4.26)	(6.28)	(12.56)	503 (26)	–	1618	1542	491
NiL^2	$[Ni(C_{21}H_{16}N_2O_2)$	Green	65.62	4.01	7.21	14.85	224 (123), 254 (24), 349 (25),	–	3036	1571	452
	(>360)	–	(65.28)	(4.14)	(7.25)	(15.02)	441 (9)	–	1625	1553	482
FeL^3	$[Fe_2(C_{20}H_{13}N_2O_2Cl)_2Cl_2(H_2O)_2$	Brown	52.17	3.67	3.53	11.74	221 (539), 285 (126), 341 (137)	–	3075	1604	464
	(>360)	5.82	(52.52)	(3.28)	(3.06)	(12.25)	506 (70)	3435	1660	1580	491
NiL^3	$[Ni_2(C_{20}H_{13}N_2O_2Cl)_2(H_2O)_4$	Green	54.93	3.32	6.78	12.57	220 (571), 293 (148), 359 (144),	–	3012	1590	464
	(>360)	3.12	(54.30)	(3.85)	(6.33)	(13.12)	440 (26), 608 (0.07)	3371	1625	1548	480

Table 2. ^{1}H and ^{13}C-NMR chemical shifts (ppm) of the asymmetric Schiff bases and diamagnetic L^2Ni.

		Compound			
		H_2L^1	H_2L^2	H_2L^3	L^2Ni
^{1}H-NMR	OH	10.34 (s), 1 H 13.66 (s), 1 H	9.65 (s), 1H 13.40 (s), 1 H	10.53 (s), 1H 13.76 (s), 1 H	– –
	CH=N	8.88 (s), 1 H 9.66 (s), 1 H	8.89 (s), 1 H 10.22 (s), 1 H	8.97 (s), 1 H 9.76 (s), 1 H	8.96 (s), 1 H 10.15 (s), 1 H
	Aromatic	6.72–7.90 (m)	6.83–7.41 (m)	6.60–7.70 (m)	6.78–7.40 (m)
	CH_3	–	2.33 (s), 3 H	–	2.26 (s), 3H
^{13}C-NMR	CH=N	161.2 162.2	158.9 162.1	159.5 159.9	159.2 162.1
	CH_3	–	20.4	–	20.30
	Ar C–OH	151.6	151.5	151.3	151.3
	Other Ar C	117.0–135.6	116.9–135.8	116.6–134.8	117.0–135.3

s: singlet, m: multiplet,

Complex	*m/z*	%	Peak
L^1Fe	870.8	2.2	$[M+7H]^+$
L^2Fe	892.9	1.1	$[M+H]^+$
L^3Fe	912.7	1.2	$[M+H]^+$
L^1Ni	816.8	1.8	$[M+H]^+$
L^2Ni	392.0	1.4	$[M+6H]^+$
L^3Ni	884.6	1.6	$[M+H]^+$

Scheme 1. Molecular ion peaks of the complexes.

The LC–mass spectra of all the complexes gave the molecular ion peaks at the desired positions as shown in scheme 1, indicating monomeric nature of L^2Ni and dimeric nature of the other complexes.

Magnetic moments of the Fe(III) complexes lie between 5.80–6.05 BM confirming high spin. In the electronic spectra, the new band, between 503–507 nm, may be due to L → M charge transfer, obscuring the low intensity, spin forbidden d–d bands [23]. Monomeric L^2Ni is diamagnetic, suggesting a square planar geometry. A weak absorption (shoulder) at 441 nm assigned to $^1A_1g \rightarrow {}^1A_2g$ transition supports planar geometry. The magnetic moments of the other Ni(II) complexes indicate two unpaired electrons. The electronic spectra of paramagnetic NiL^1 and NiL^3 showed a shoulder around 437–440 nm and a weak band around 613–608 nm, indicating $^3A_2g(F) \rightarrow {}^3T_1g(F)$ and $^3A_2g(F) \rightarrow {}^3T_1g(P)$ transitions for octahedral d^8 ions [22].

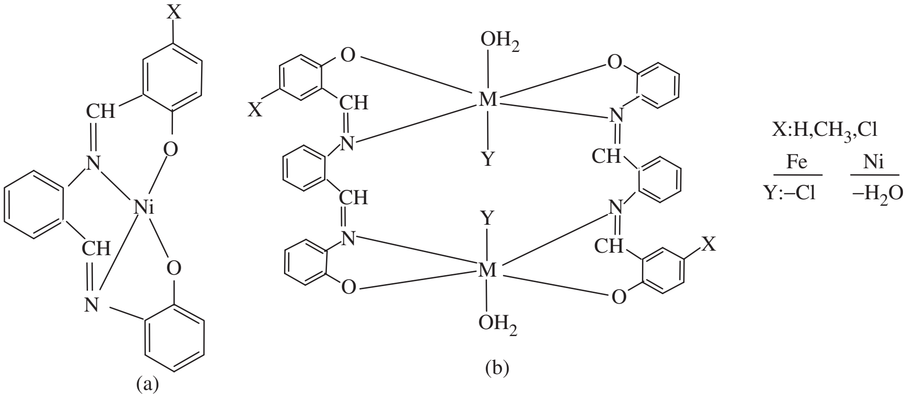
^{1}H and ^{13}C-NMR data of the diamagnetic L^2Ni complex is given in table 2. In the comparison to free H_2L^2: (i) phenolic protons in the ^{1}H-NMR spectrum disappear on complexations; (ii) the signal of the first imine proton (lower δ value) shows a downfield shift, whereas the second imine proton shifts to lower δ value; (iii) all of the C atoms show small shifts after complexation.

Thermal analyses are presented in table 3. TGA of Fe(III) complexes consist of three decomposition steps at ca 50, 250 and 400°C and octahedral Ni(II) complexes have two

Table 3. Thermal data of the complexes.

Compound	Step	*T_i	*T_f	Lost fragment (LF) Residue (R)	% Weight loss Found (Calcd)
FeL^1	1st	61.70	201.84	2 H_2O(coord)+H_2O(hyd) (Lf)	6.23 (6.25)
	2nd	227.86	361.26		
	3rd	381.20	800	Fe_2O_3 (r)	17.20 (18.52)
FeL^2	1st	46.69	194.63	2 H_2O(coord)+H_2O(hyd) (Lf)	6.13 (6.05)
	2nd	240.10	364.14		
	3rd	364.14	800	Fe_2O_3 (r)	17.94 (18.54)
FeL^3	1st	66.75	195.10	2 H_2O(coord) (Lf)	3.91 (3.93)
	2nd	249.11	400.07		
	3rd	435.07	800	Fe_2O_3 (r)	16.54 (17.51)
NiL^1	1st	83.96	252.61	4 H_2O(coord) (Lf)	8.75 (8.82)
	2nd	427.17	800	½ NiO (r)	4.83 (4.59)
NiL^2	1st	253.78	800	½ NiO (r)	9.61 (9.12)
NiL^3	1st	73.92	201.32	4 H_2O(coord) (Lf)	8.35 (8.14)
	2nd	201.32	800	½ NiO (r)	8.22 (8.45)

T_i Initial temperature.
T_f Final temperature.



(a) (b)

Figure 3. Suggested structure of L_2Ni (a) and the other complexes (b).

decomposition steps at ca 75 and 300°C. The first step seems to be consistent with loss of hydration and coordination water. Other steps represent degradation of the complexes. The TG of L^2Ni shows one step. The residues at the end of the decomposition for all of the complexes are found to be Fe_2O_3 or NiO.

Based on the spectroscopic, analytic and the other data, the suggested structures of the complexes are given in figure 3.

The results of antibacterial screening of the compounds are summarized in table 4. Ligands have activity at 500 and 1000 µg concentrations against *E. coli*, *B. subtilis* and *Y. enterecolitica* but are inactive against *P. aeruginosa* and *S. aureus*; activity of H_2L^2 is higher than the other ligands. The highest inhibitory zones are measured in Fe(III) complexes against *P. aeruginosa*, while the ligands and the Ni(II) complexes

Table 4. Antibacterial activities of the compounds and some standard antibiotics against test bacteria.

	Bacterium									
	P. aeruginosa		*S. aureus*		*E. coli*		*B. subtilis*		*Y. enterecolitica*	
Conc. (μg)	500	1000	500	1000	500	1000	500	1000	500	1000
Compound										
H_2L^1	–	–	–	–	4	6	9	9	7	7
H_2L^2	–	–	–	–	8	9	17	20	16	18
H_2L^3	–	–	–	–	5	8	11	14	9	12
L^1Ni	–	–	–	–	–	–	3	5	5	5
L^2Ni	5	6	–	7	6	7	6	9	6	8
L^3Ni	–	–	–	–	–	–	3	3	5	5
L^1Fe	15	19	4	4	–	–	5	5	3	3
L^2Fe	12	14	4	4	–	–	8	8	7	7
L^3Fe	17	20	–	–	–	–	9	6	9	10
Erythromcin*	R**		17		R		29		R	
Vancomcin*	R		8		R		19		R	

*These values are taken from Ref. [23] for comparison (Erythromycin 30 μg, Vancomycin 15 μg).
**Resistant.

(except NiL^2) are inactive. Additionally, *P. aeruginosa* is resistant to Erythromycin and Vancomycin as shown in table 4.

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