

Synthesis, Physicochemical Studies and Biological Properties of Mixed-Ligand Nickel(II) Complexes Containing β -Diketones With N-donor Ligands

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Abstract

Some mixed ligand nickel(II) complexes of thenoyltrifluoroacetone (tta-H) with 2,2'-bipyridine (bipy), 1,10-phenanthroline (phen) and tetramethylethylenediamine (tmen) $[\text{Ni}(\text{tta})(\text{N-N})(\text{NO}_3)]$; N-N= bipy, phen, or tmen] have been synthesized and characterized by molar conductance measurements, elemental analysis, mass spectrometry, spectral measurements and antimicrobial activities. Attempt to prepare the mixed ligand nickel(II) complexes containing acetylacetone (acacH), benzoylacetone (bzacH), dibenzoylmethane (dbm-H) and thenoyltrifluoroacetone (tta-H) with ethylenediamine associated with NO_3 counter ion led to the formation of $[\text{Ni}(\beta\text{-diketone})_2\text{en}]$. The β -diketones enolized acting as bidentate ligand, coordinating to the metal with carbonyl and enolic oxygen, ethylenediamine (en) coordinates to the metal using N, N chromophores. The nickel complexes of the type $[\text{Ni}(\text{tta})(\text{N-N})\text{NO}_3]$ are five-coordinate square pyramidal in geometry while those of the type $[\text{Ni}(\beta\text{-diketone})_2\text{en}]$ are six-coordinate octahedral geometry. The conductivity measurement in nitromethane revealed that the $[\text{Ni}(\text{tta})(\text{N-N})\text{NO}_3]$ complexes are non-electrolyte. The complexes were screened for their in-vitro antimicrobial activity against six microorganisms. A comparison of the complexes with gentamicin showed that most of the complexes are resistant to tested organisms except $[\text{Ni}(\text{tta})_2(\text{en})]\cdot\text{H}_2\text{O}$, $[\text{Ni}(\text{bzac})_2(\text{en})]$ and $[\text{Ni}(\text{bta})_2(\text{en})]$ which compared favourably well in *Staphylococcus aureus*.

Key words: β -Diketones, mixed-ligand, spectral, geometry, antimicrobial

1. Introduction

There has been growing interest in the metal derivatives of β -diketones during the past two decades and a lot of articles have been published on the synthesis of these metal complexes (Ahumada *et al.*, 2018; Omoregie *et al.*, 2017; Omoregie *et al.*, 2016; Omoregie, 2014; Omoregie, 2012; Arpita and Raman, 2010). Metal complexes of β -diketones are being focused on in medicinal chemistry and research has revealed that anticancer activity against tumoral cells is exhibited by Platinum(II) complexes of β -diketones (Wilson and Lippard, 2012).

Extensive literature is available on the application of β -diketonate complexes in catalysis and as precursors for MOCVD (Bose *et al.*, 2016; Poncelet *et al.*, 2005; Lassahn *et al.*, 2005; Nable *et al.*, 2003.). Studies revealed that β -diketones were used in the design of many luminescence compounds (Ahumada *et al.*, 2018) and the metal complexes were used to inhibit the growth of K562 cell (Almeida *et al.*, 2015).

Mixed-ligand nickel(II) complexes containing acac and bzac have been synthesized but there is no report on the synthesis and biological activity of the $[\text{Ni}(\text{tta})(\text{N-N})(\text{NO}_3)]$ and ethylenediamine series. Hence the need to synthesize the mixed-ligand complexes in ratio 1 of β -diketone to 1 of 2,2'-bipyridine, 1,10-phenanthroline, ethylenediamine/tetramethylethylenediamine and investigate their antimicrobial activity. In the process of synthesizing these, ratio 2 of β -diketone and 1 of ethylenediamine were formed.

2. Experimental

2.1 Materials and Physical Measurements

The following reagents were used: benzoylacetone (bzacH), dibenzoylmethane (dbmH), benzoyltrifluoroacetone (btaH), thenoyltrifluoroacetone (ttaH) (Aldrich chemicals), nickel nitrate hexahydrate, ethylenediamine, 2,2'-bipyridine and 1,10-phenanthroline (analytical grade).

2.2 Synthesis of $[Ni(tta)(bipy)(NO_3)]$

$NiNO_3 \cdot 6H_2O$ (1.324 g, 4.504 mmol) dissolved in water (2 mL) was added to a mixture of thenoyltrifluoroacetone (1.009 g, 4.504 mmol) and 2,2'-bipyridine (0.3723 g, 1.88 mmol) in methanol (10 mL). NaOH was added to the mixture and stirred for one hour and the green solid product was collected by filtration, washed with water and methanol, and dried in vacuo. A similar procedure was used for the preparation of $[Ni(tta)(phen)(NO_3)]$ and $[Ni(tta)(tmen)(NO_3)]$. Anal. Calc. for $C_{18}H_{12}F_3N_3NiO_5S$: C, 43.41; H, 2.43; N, 8.44; Found C, 43.64; H, 2.84; N, 7.96 MS: Calc. for $[Ni(TTA)bipy]^+$ m/z 436.06, found: 435.0

2.3 Synthesis of $[Ni(bzac)_2(en)]$

$NiNO_3 \cdot 6H_2O$ (0.538 g, 1.85 mmol) dissolved in water (1 mL) was added to a mixture of benzoylacetone (0.30 g, 1.85 mmol) and ethylenediamine (0.12 mL, 1.85 mmol) in methanol (5 mL). Na_2CO_3 (0.098 g, 9.248 mmol) dissolved in water (2 mL) was added to the mixture and stirred for one hour and the green solid product was collected by filtration, washed with water and methanol, and dried in vacuo. Anal. Calc. for $C_{22}H_{26}N_2NiO_4$: C, 59.90; H, 5.94; N, 6.35; Ni, 13.30. Found C, 59.42; H, 6.00; N, 5.85 Ni, 13.13

2.4 Synthesis of $[Ni(bta)_2(en)]$

$NiNO_3 \cdot 6H_2O$ (0.404 g, 1.389 mmol) dissolved in water (1.2 mL) was added to a mixture of benzoyltrifluoroacetone (0.30 g, 1.389 mmol) and ethylenediamine (0.093 mL, 1.389 mmol) in methanol (5 mL). Na_2CO_3 (0.098 g, 9.248 mmol) dissolved in water (2 mL) was added to the mixture and stirred for one hour and the green solid product was collected by filtration, washed with water and methanol, and dried in vacuo. Anal. Calc. for $C_{22}H_{20}F_6N_2NiO_4$: C, 48.12; H, 3.67; N, 5.10; Ni, 10.69. Found C, 48.21; H, 4.13; N, 5.29 Ni, 10.78

2.5 Synthesis of $[Ni(dbm)_2(en).H_2O]$

$NiNO_3 \cdot 6H_2O$ (0.389 g, 1.338 mmol) dissolved in water (2 mL) was added to a mixture of dibenzoylmethane (0.30 g, 1.338 mmol) and ethylenediamine (0.09 mL, 1.338 mmol) in methanol (10 mL). Na_2CO_3 (0.098 g, 6.689 mmol) dissolved in water (2 mL) was added to the mixture and stirred for one hour and the green solid product was collected by filtration, washed with water and methanol, and dried in vacuo. Anal. Calc. for $C_{32}H_{32}N_2NiO_5$: C, 65.89; H, 5.53; N, 4.80; Ni, 10.06 Found C, 65.21; H, 5.18; N, 4.88 Ni, 10.72

2.6 Synthesis of $[Ni(tta)_2(en)].H_2O$

$NiNO_3 \cdot 6H_2O$ (0.393 g, 1.350 mmol) dissolved in water (1.2 mL) was added to a mixture of thenoyltrifluoroacetone (0.30 g, 1.350 mmol) and ethylenediamine (0.09 mL, 1.350 mmol) in methanol (5 mL). Na_2CO_3 (0.072 g, 6.751 mmol) dissolved in water (2 mL) was added to the mixture and stirred for one hour and the green solid product was collected by filtration, washed with water and methanol, and dried in vacuo. Anal. Calc. for $C_{18}H_{18}F_6N_2NiO_5S_2$: C, 37.33; H, 3.13; N, 4.84; Ni, 7.63. Found C, 37.34; H, 3.17; N, 4.28 Ni, 7.38.

2.7 Biological Studies

2.7.1 Antimicrobial Susceptibility Testing

The antimicrobial activity of the complexes prepared was determined by agar cup diffusion method using each compound in decreasing concentration of 200 mg/mL-6.25 mg/mL dissolved in methanol against isolated organisms. Plate cultures were prepared either by seeding (bacteria/fungi) or spreading using 0.1 mL of 10^{-2} dilution from 18-12 hours. 3 drops of dissolved compound was used to fill each of the well dug in the set agar media, pre-incubation diffusion period of 1 hour on bench was observed. Incubation of the cultured plates were observed at 37° C for 24 hours (for bacteria) and 28 ° C for 24-72 hours (for fungi). Observation of the plates for zones of growth inhibition, measured in mm was recorded (Omorieg *et al.*, 2015).

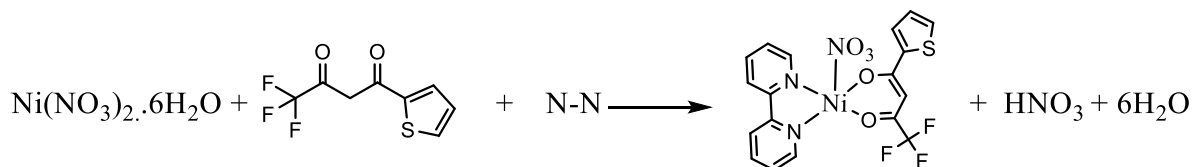
2.8 Physical Measurements

The elemental analysis for CHNS were determined at the School of Chemistry and Physics, University of KwaZulu-Natal, South Africa. The percentage metal in the nickel(II) compounds were determined titrimetrically using EDTA. The magnetic susceptibilities of the compounds at room temperature were measured using the Sherwood magnetic susceptibility balance.

The infrared spectra were measured using nujol on Perkin Elmer spectrophotometer 11 FT-IR. The electronic spectra of the compounds in chloroform were recorded on a Perkin Elmer Lambda Double Beam UV/VIS spectrophotometer using 1cm glass cell at the Department of Chemistry, University of Ibadan, Ibadan, Nigeria.

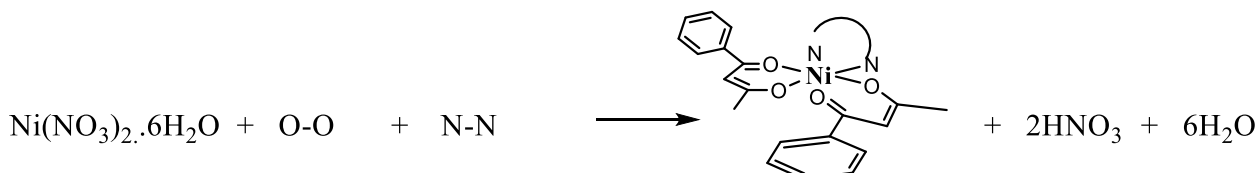
3. Results and Discussion

The formation of the complexes were confirmed by microanalysis. Scheme 1 is for the formation of $[Ni(tta)(N-N)NO_3]$ series while $[Ni(\beta\text{-diketone})_2en]$ complexes are formed using scheme 2.



N-N = 1,10-phenanthroline, 2,2'-bipyridine and tetramethylethylenediamine.

Scheme 1



O-O = benzoylacetone, benzoyltrifluoroacetone, dibenzoylacetone, thenoyltrifluoroacetone

N-N = 1,10-phenanthroline, 2,2'-bipyridine and tetramethylethylenediamine

Scheme 2

The analytical data, colours, percentage yields, melting points, molar conductivities, and room temperature magnetic moments of the complexes are presented in Table 1. The molar conductivities of the $[\text{Ni}(\text{tta})(\text{N-N})\text{NO}_3]$ complexes in nitromethane were in the range $23\text{-}53 \text{ ohm}^{-1} \text{ cm}^2 \text{ mole}^{-1}$, showing that they are non-electrolytes. A value of $60\text{-}115 \text{ ohm}^{-1} \text{ cm}^2 \text{ mole}^{-1}$ is expected for a 1:1 electrolyte (Geary, 1970)

3.1 Infrared

The relevant infrared bands are presented in table 2. The ν_{NH} bands in the $[\text{Ni}(\beta\text{-diketone})_2\text{en}]$ complexes were observed as three bands in range $3173\text{-}3364 \text{ cm}^{-1}$. The $\nu_{\text{as}}\text{C}=\text{O} + \nu_{\text{as}}\text{C}=\text{C}$ vibrations occurred in the $1593\text{-}1621 \text{ cm}^{-1}$ in all the nickel complexes. The $\nu_{\text{as}}\text{C}=\text{O} + \nu_{\text{as}}\text{C}=\text{C}$ vibrations of the metal free ligand tta-H were observed at $1643\text{-}1663 \text{ cm}^{-1}$ (Woods *et al.*; 2009). These shifted to $1608\text{-}1620 \text{ cm}^{-1}$ in the $[\text{Ni}(\text{tta})(\text{N-N})\text{NO}_3]$ complexes indicating the coordination of ligand to the metal. The shift in the $[\text{Ni}(\beta\text{-diketone})_2\text{en}]$ series as compared to the ligands (Woods *et al.*, 2009; Omoregie, 2012; Omoregie and Woods, 2011) is also an indication of coordination. Studies have revealed that ionic nitrate bands are categorized as $\nu_1 = 1050 \text{ cm}^{-1}$ (N-O stretch, infrared-inactive), $\nu_2 = 831 \text{ cm}^{-1}$ (NO, deformation), $\nu_3 = 1390 \text{ cm}^{-1}$ (NO, asymmetric stretch), and $\nu_4 = 720 \text{ cm}^{-1}$ (planar rock) (Gatehouse *et al.*, 1957; Wheeler and Walmsley, 1986). In the $[\text{Ni}(\text{tta})(\text{N-N})\text{NO}_3]$ series, the bands in the range $1384\text{-}1385 \text{ cm}^{-1}$ have been assigned as NO_2 asymmetric stretch while bands in $1056\text{-}1062 \text{ cm}^{-1}$ have been assigned as N-O stretch. NO deformation occurred in the range $819\text{-}846 \text{ cm}^{-1}$ while planar rock bands occurred in the range $717\text{-}726 \text{ cm}^{-1}$. The 2,2'-bipyridine CH deformation band was observed as strong band at 766 cm^{-1} region. This occurred in the phenanthroline around 726 cm^{-1} and 846 cm^{-1} region.

3.2 Electronic Spectra, Magnetic Moment and Mass Spectroscopy

The electronic spectra of the nickel complexes in chloroform are presented in Table 3. There is a clear relationship between five coordinate high spin square pyramidal complexes and tetragonal six coordinate complexes through removal of one ligand from an octahedral complexes (Lever, 1986). Bands similar to octahedral complexes were observed in the $[\text{Ni}(\text{tta})(\text{bipy})(\text{NO}_3)]$, $[\text{Ni}(\text{tta})(\text{phen})(\text{NO}_3)]$ and $[\text{Ni}(\text{tta})(\text{tmen})(\text{NO}_3)]$ and are tentatively square pyramidal in geometry (Lever, 1986). This is supported by the X-ray structure of $[\text{Ni}(\text{bta})(\text{phen})(\text{Cl})]$ (Omoregie, 2018). The $[\text{Ni}(\beta\text{-diketone})_2\text{en}]$ series had absorption bands typical of six-coordinate octahedral geometry which is corroborated by the observed magnetic moment.

The peak observed at m/z 435 corresponded to $[\text{Ni}(\text{tta})(\text{bipy})]^+$ and this shows that NO_3 has been lost.

3.3 Antimicrobial Activities

The ligands showed good activity at 200 mg/mL in the tested bacteria except in *Klebsiella pneumonia* in which bta-H and bza-H showed resistance and moderate activity respectively. dbm-H showed pronounced activity in all the tested bacteria while tta-H only showed sensitivity in E-coli. But it had moderate sensitivity in *Staphylococcus aureus*; *Bacillus subtilis* and *Salmonella enterica*. All the nickel complexes showed good activity in *Staphylococcus aureus* except $[\text{Ni}(\text{tta})(\text{bipy})(\text{NO}_3)]$ and $[\text{Ni}(\text{tta})(\text{phen})(\text{NO}_3)]$ that were resistant and $[\text{Ni}(\text{dbm})_2(\text{en})]\text{H}_2\text{O}$ showed moderate

activity. All the complexes were resistant in *Klebsiella pneumonia* except $[\text{Ni}(\text{tta})_2(\text{en})]\cdot\text{H}_2\text{O}$ and $[\text{Ni}(\text{bzac})_2(\text{en})]$ which were moderately sensitive and sensitive respectively. The complexes were also resistant in *Escherichia coli* except $[\text{Ni}(\text{bzac})_2(\text{en})]$ and $[\text{Ni}(\text{bta})_2(\text{en})]$ with moderate sensitivity. Comparison of the ligands with the standard drug (Tioconazole) showed that the ligands have mild antifungi properties. $[\text{Ni}(\text{bzac})_2(\text{en})]$ and $[\text{Ni}(\text{bta})_2(\text{en})]$ compared favourably well with Tioconazole while $[\text{Ni}(\text{tta})_2(\text{en})]$ and $[\text{Ni}(\text{dbm})_2(\text{en})]\cdot\text{H}_2\text{O}$ were resistant. There is a good comparison of the ligands with the gentamicin except tta-H which was mild. Comparison of the complexes with gentamicin showed that most of the complexes are resistant except $[\text{Ni}(\text{tta})_2(\text{en})]\cdot\text{H}_2\text{O}$, $[\text{Ni}(\text{bzac})_2(\text{en})]$ and $[\text{Ni}(\text{bta})_2(\text{en})]$ which compared favourably well in *Staphylococcus aureus*.

4. Conclusion

A tentative five-coordinate square pyramidal geometry is proposed for the $[\text{Ni}(\text{tta})(\text{NN})(\text{NO}_3)]$ complexes while the $[\text{Ni}(\beta\text{-diketone})_2(\text{en})]$ series have probable six-coordinate octahedral geometry. This is corroborated by magnetic, infrared and electronic spectral measurements. The antibacterial studies revealed that $[\text{Ni}(\text{bzac})_2(\text{en})]$ shows a broad spectrum of antimicrobial activity against *Staphylococcus aureus*, *Klebsiella pneumonia*, *Escherichia coli*, *Candida albicans*, *Aspergillus niger* and *Penicillium notatum*. A similar trend is observed in the antimicrobial activity of $[\text{Ni}(\text{tta})(\text{bipy})(\text{NO}_3)]$ and $[\text{Ni}(\text{tta})(\text{phen})(\text{NO}_3)]$ which shows that the antimicrobial activity is not affected by the presence of any substituent as compared with $[\text{Ni}(\text{tta})_2(\text{en})]$.

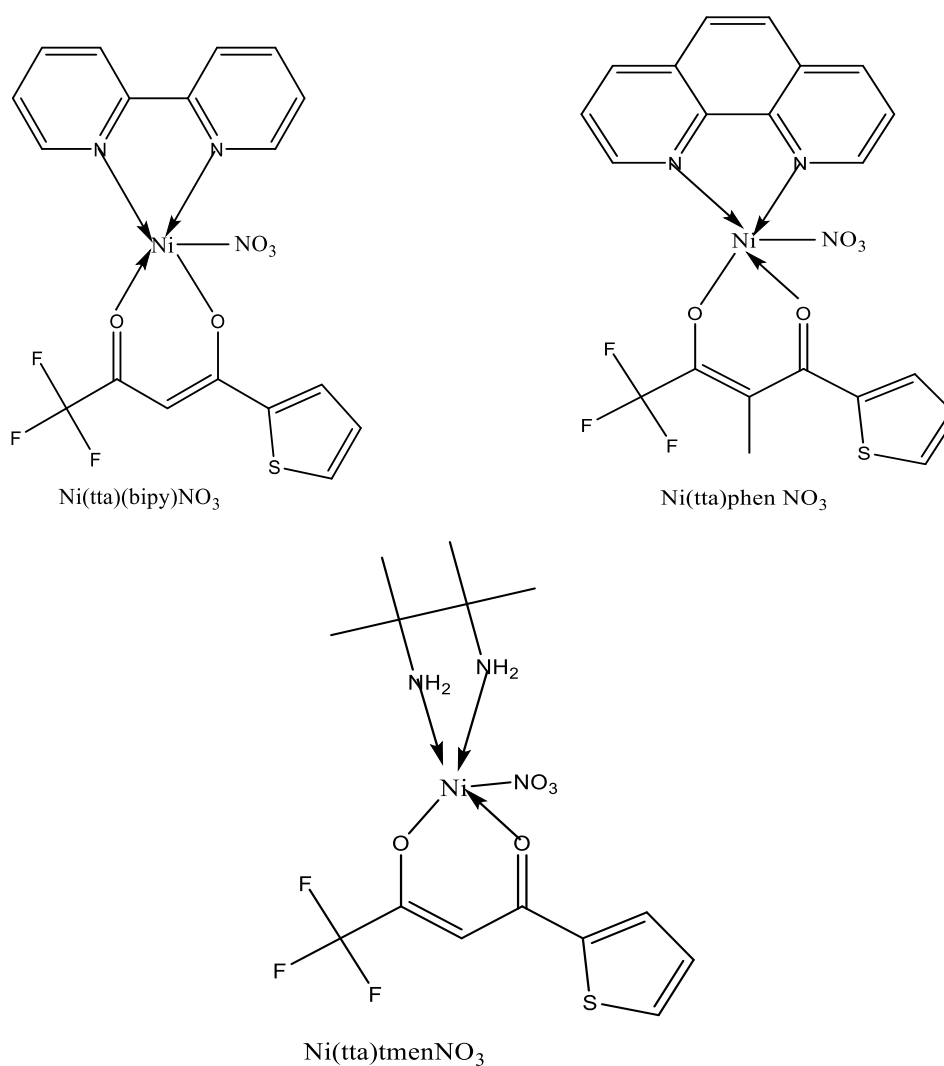


Table 1. Analytical and physical data of mixed ligand nickel(II) complexes

Compounds	Mol. wt. (g mol ⁻¹)	Colour	M.pt(°C)	Yield%	Molar conductance (Ω ⁻¹ cm ² mol ⁻¹)	μ _{eff} (BM)
[Ni(tta)(bipy)(NO ₃)]	498.06	Light Green	165-167	24.41	53	3.12
[Ni(tta)(phen)(NO ₃)]	522.08	Green	268-270	22.67	27	2.86
[Ni(tta)(tmen)(NO ₃)]	458.08	Green	130-132	50.33	23	2.93
[Ni(tta) ₂ (en)].H ₂ O	579.15	Green	280-282	54.05	12	3.17
[Ni(bzac) ₂ (en)]	441.15	Light Green	260-262	57.89	34	3.15
[Ni(bta) ₂ (en)]	549.10	Light Green	276-278	45.78	43	3.10
[Ni(dbm) ₂ (en)]H ₂ O	583.31	Yellow	>300	40.08	35	3.13

Table 2. Infrared spectral data of mixed ligand nickel(II) complexes

Compounds	v(NH)	C=O, C=C	NO ₂ str	N-Ostr	v _s (C-H) phen/bipy	NO ₂ def	Planar rock
bzaac-H		1599m, 1540b					
bta-H		1604vs					
dbm-H		1598m, 1540b					
tta-H		1663b, 1643w					
[Ni(tta)(bipy)(NO ₃)]		1608s, 1566m, 1537s	1385s	1056s	766vs	819vs	723vs
[Ni(tta)(phen)(NO ₃)]		1612s, 1569w, 1539s	1384m	1060vs	846s 726s		
[Ni(tta)(tmen)(NO ₃)]		1620s, 1565w, 1538m	1384w	1062s		845m	717s
[Ni(tta) ₂ (en)].H ₂ O	3364s, 3286vs, 3173m	1602vs, 1537vs, 1501vs	-				
[Ni(bzac) ₂ (en)]	3353s, 3277s, 3173m	1593, 1570, 1518	-				
[Ni(bta) ₂ (en)]		1621s, 1576s	-				
[Ni(dbm) ₂ (en)]H ₂ O		1610, 1593, 1549	-				

Key: vs = very strong, s = strong, m = medium, b = Broad, w=weak

Table 3. Electronic spectral data of mixed-ligand nickel(II) complexes

Compounds	Electronic spectra in chloroform (cm ⁻¹) d-d
tta-H	36,100br, 42,000
[Ni(tta)(bipy)(NO ₃)]	12,778*, 16,667
[Ni(tta)(phen)(NO ₃)]	13,072*, 17,1821
[Ni(tta)(tmen)(NO ₃)]	12,230*, 16,528
[Ni(tta) ₂ (en)].H ₂ O	17,094
[Ni(bzac) ₂ (en)]	12,897*, 17,123
[Ni(bta) ₂ (en)]	13,018*, 16,779
[Ni(dbm) ₂ (en)]H ₂ O	17,121

Key: * = shoulder, br=broad

Table 4. Antimicrobial activity data of β-diketone and mixed ligand nickel(II) complexes

Compound	<i>S. aur</i>	<i>B. sub</i>	<i>K. pne</i>	<i>E. coli</i>	<i>S. ent</i>	<i>P. aer</i>	<i>C.a</i>	<i>A.n</i>	<i>Pen</i>
bzac-H	24	32	18	32	32	34	16	14	16
bta-H	26	21	R	24	19	30	ND	ND	ND
dbm-H	20	24	24	32	32	30	14	16	18
tta-H	17	16	-	21	17	36	ND	ND	ND
[Ni(tta)(bipy)(NO ₃)]	R	R	R	R	R	R	ND	ND	ND
[Ni(tta)(phen)(NO ₃)]	R	R	R	R	R	R	ND	ND	ND
[Ni(tta) ₂ (en)].H ₂ O	20	ND	19	R	ND	ND	R	R	R
[Ni(bzac) ₂ (en)]	20	ND	20	19	ND	ND	25	25	26
[Ni(bta) ₂ (en)]	30	ND	R	15	ND	ND	25	R	21
[Ni(dbm) ₂ (en)]H ₂ O	15	ND	R	R	ND	ND	R	R	R
Gentamicin (Positive control)	38	38	40	40	40	38	NA	NA	NA
Tioconazole (Positive control)	NA	NA	NA	NA	NA	NA	28	28	28
Methanol (Negative control)	R	R	R	R	R	R	R	R	R

Key: R= organism resistant to the extract

S. aur = *Staphylococcus aureus*; *B. sub* = *Bacillus subtilis*; *K. pne* = *Klebsiella pneumonia*; *E. coli* = *Escherichia coli*; *S. ent* = *Salmonella enterica*; *P. aer* = *Pseudomonas aeruginosa*; *Ca* = *Candida albicans*; *An* = *Aspergillus niger*; *Pen* = *Penicillium notatum*;

ND-not determined NA-not applicable

Growth inhibition zone in millimeters (mm)

10-19-moderate

20-29-active

30 and above- v.active

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