

# MIXED LIGAND NICKEL(II) COMPLEXES OF BENZOYLTRIFLUOROACETONE WITH 2,2'-BIPYRIDINE, 1,10- PHENANTHROLINE AND TETRAMETHYLETHYLENEDIAMINE: SYNTHESIS, SPECTROSCOPIC AND ANTIMICROBIAL PROPERTIES

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## ABSTRACT

*A series of nickel (II) complexes of 4,4,4-trifluoro-1-phenyl-1,3-butanedione(bta) ligand mixed with 2,2'-bipyridine (bipy), 1,10-phenanthroline (phen), and tetramethylethylenediamine(tmen) were synthesized and characterized by elemental analysis, IR and UV-Vis spectroscopy, magnetic and antimicrobial studies . A distorted square pyramidal nickel(II) complexes with the formular  $[Ni(bta)(N-N)(X)].H_2O$  was formed with the bta ligand, where bta coordinated with the nickel ion through the two oxygen atom, the N-N donor ligand bonding with two nitrogen atom and the fifth coordination position occupied by the  $NO_3$  molecule. On the other hand, the complexes;  $[Ni(bta)_2tmen]$  and  $[Ni(bta)(bzac)bipy].H_2O$  exhibited an octahedral geometry. The microbiostatic activity revealed that the  $[Ni(bta)(phen)(NO_3)].H_2O$  and  $[Ni(bta)(bipy)(NO_3)].H_2O$  lacked antibacterial activity.*

**Keywords:** 4,4,4-trifluoro-1-phenyl-1,3-butanedione, nickel(II), spectral measurements, antimicrobial studies

## INTRODUCTION

$\beta$ -Diketones are of great interest in coordination chemistry and have been studied extensively because of their use in catalysis [Poncelet *et al.*, 2005; Lassahn *et al.*, 2005; Schwieger *et al.*, 2009; Bose *et al.*, 2016].  $\beta$ -Diketones have been found to have remarkable antibacterial, antifungal and antioxidant properties [Omoregie and Olowoake, 2016; Ahumada *et al.*, 2017; Omoregie *et al.*, 2022]. Complexes with  $\beta$ -diketones and aromatic bidentate ligands (bipy, phen and related molecules) play a key role in many biochemical systems [Malekshah *et al.*, 2018]. There is significant progress in the design of anticancer agents in medicinal inorganic field over the past decades [Ma *et al.*, 2012; Abbasi *et al.*, 2017; Malekshah *et al.*, 2018;] and literature has revealed the sensitivity of human cancer cell

lines to benzoylacetone complexes than methyl acetoacetate complexes [Malekshah *et al.*, 2018]. In continuation of our work on  $\beta$ -diketones [Woods *et al.*, 2009a; Woods *et al.*, 2009b; Omoregie and Woods, 2010; Omoregie and Woods, 2011; Omoregie *et al.*, 2014; Omoregie *et al.*, 2018; Omoregie 2018a; Omoregie 2018b; Omoregie *et al.*, 2022a; Omoregie *et al.*, 2022b], we have synthesized the mixed ligand nickel(II) complexes of 4,4,4-trifluoro-1-phenyl-1,3-butanedione with the aim of investigating the biological properties of these complexes.

## EXPERIMENTAL

### Materials and physical measurements

The reagents: 4,4,4-trifluoro-1-phenyl-1,3-butanedione(btaH), tetramethylethylenediamine, 1-phenyl-1,3-butanedione(bzach)(Aldrich

chemicals), nickel nitrate, nickel chloride, 2,2'-bipyridine and 1,10-phenanthroline (Analytical grade) were sourced commercially and used as supplied. The % metals in the mixed-ligand complexes were determined by titrimetric method with EDTA. The infrared spectra of the complexes, as pressed KBr disc, were recorded on the Buck 500 Scientific model infrared spectrophotometer in the region 4000-400  $\text{cm}^{-1}$ . The solution spectra of the complexes in methanol were recorded on a Spectro UV-VIS double beam PC scanning spectrophotometer-UVD-2960.

#### **Synthesis of [Ni(bta)(phen)NO<sub>3</sub>].H<sub>2</sub>O.CH<sub>3</sub>OH**

Nickel nitrate hexahydrate (1.320 g, 4.54 mmol) was dissolved in water (2 mL) and added directly to a mixture of 1,10-phenanthroline (0.90 g, 4.54 mmol) and btaH (0.981 g, 4.54 mmol) in methanol (10 mL). The pH of the reaction mixture was adjusted to 8 using sodium hydroxide solution and stirred at room temperature for 1 h. The green solid product was collected by filtration, washed with water and methanol, and dried in vacuo. [Ni(bta)(bipy)NO<sub>3</sub>].H<sub>2</sub>O was prepared using similar procedure. Yield, 0.8710 g, 35.93%.  $\mu_{\text{eff}}=2.60$ B.M., Anal. Calc. for C<sub>23</sub>H<sub>20</sub>F<sub>3</sub>N<sub>3</sub>NiO<sub>7</sub>: C,48.80; H, 3.56; N, 7.42; Ni,10.99. Found C,49.04 H,4.06 N,6.70Ni, 10.59%. IR [(v(OH) 3434b, [(v(C=O)+v(C=C)1627vs, 1600vw, 1578vs, 1533s, 1517m]; (NO<sub>2</sub>)<sub>str</sub>, 1384s;N-O, 872m;γ(C-H), 851vs, 721s.

#### **Synthesis of [Ni(bta)(bzac)bipy].H<sub>2</sub>O**

Nickel(II) chloride (0.59 g, 2.47 mmol) dissolved in 2mL of distilled water was added to a mixture of 1-phenyl-1,3-butanedione (0.40 g, 2.47 mmol), 4,4,4-Trifluoro-1-phenyl-1,3-butanedione (0.53 g, 2.47 mmol) and 2,2'-bipyridine (0.59 g, 2.47 mmol) in methanol (10mL). The mixture was stirred at room temperature for an hour. The precipitated solids were washed with 50% methanol and dried in

vacuum dessicator. Yield, 0.8233 g, 40.43%  $\mu_{\text{eff}}=2.81$ , Anal. Calc. for C<sub>30</sub>H<sub>25</sub>F<sub>3</sub>N<sub>2</sub>NiO<sub>5</sub>: C,59.15; H, 4.14; N, 4.60; Ni, 9.63. Found C,58.34; H,3.36 N,5.19 ; Ni,8.98. IR [(v(C=O)+v(C=C)1626w, 1608m, 1574w,1532w]γ(C-H)760s

#### **Synthesis of [Ni(bta)2tmen]**

Nickel(II) chloride hexahydrate (0.4398 g, 1.85mmol) dissolved in 2mL of distilled water was added to a mixture of tetramethylethylenediamine (0.2156 g, 1.85 mmol) and btaH (0.4010 g, 1.85 mmol) in methanol (10 mL). Yield: 0.4036 g, 36.05%  $\mu_{\text{eff}}=3.22$ , Anal. Calc. for C<sub>20</sub>H<sub>16</sub>F<sub>3</sub>N<sub>3</sub>NiO<sub>6</sub>: C, 51.60; H, 4.66; N, 4.63; Found C,51.65 H,4.99 N,5.46. IR [(v(C=O)+v(C=C)1631w,1612w, 1597w, 1577m]; γ(C-H), 759s.

#### **Biological Studies**

The antibacterial test was carried out at the Department of Pharmaceutical Microbiology, University of Ibadan, Ibadan, Nigeria.

#### **Antimicrobial Susceptibility Testing**

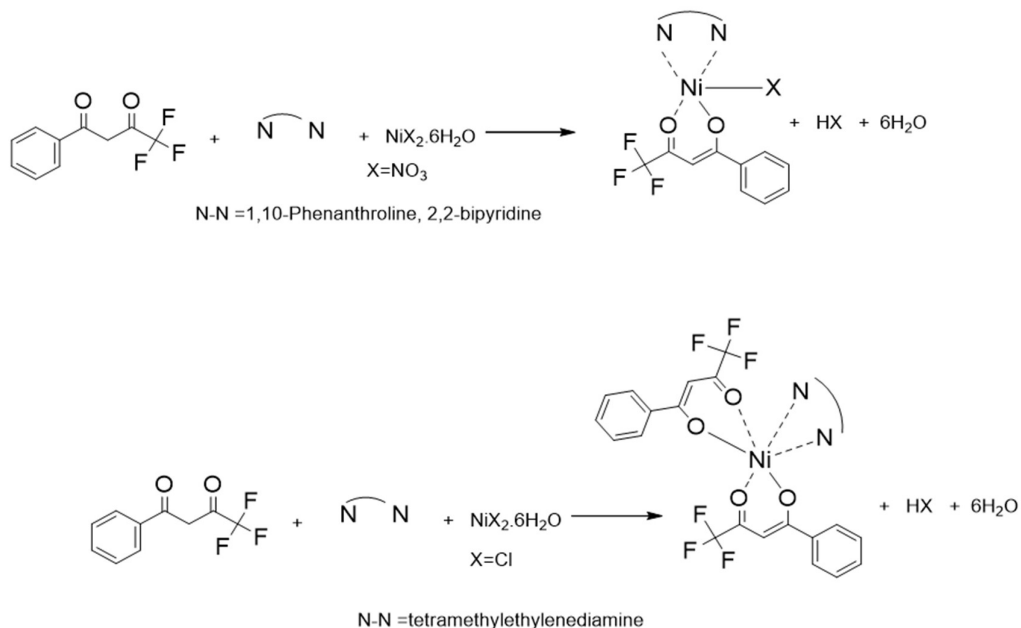
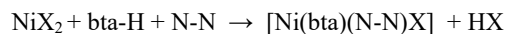
The antimicrobial activity of the complexes was determined by agar cup diffusion method using each compound in decreasing concentration of 200 mg/mL-6.25 mg/mL dissolved in methanol against isolated organisms. Plate cultures were prepared either by seeding (bacteria/fungi) or spreading using 0.1 mL of 10<sup>-2</sup> 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 was 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 [Omereg *et al.*, 2015; Omereg 2018a; Omereg 2018b].

#### **RESULT AND DISCUSSION**

The nickel (II) complexes were obtained by reaction of appropriate amounts of nickel (II) salts with

4,4,4-trifluoro-1-phenyl-1,3-butanedione and the respective ligand, 1,10-phenanthroline or 2,2'-bipyridine and tetramethylethylenediamine, in good

yields (36-40.43%). The formation of the complexes is given by the equation below:



**Scheme 1. Synthesis of the nickel (II) complexes. (X = NO<sub>3</sub><sup>-</sup> and N-N = 1,10-phenanthroline or 2,2'-bipyridine and tetramethylethylenediamine)**

## SPECTROSCOPIC / STRUCTURAL CHARACTERIZATIONS

### 3.1.1. Infrared

The assignments of the infrared bands were made by comparing the spectra of synthesized compounds with those reported in the literature [Schilt *et al.*, 1959; Omereg, 2012; Omereg *et al.*, 2022b]. The infrared spectra of the nickel(II) complexes showed the  $\nu(\text{C}=\text{O})+\nu(\text{C}=\text{C})$  band in the range 1631-1517  $\text{cm}^{-1}$ , which are the prominent bands in  $\beta$ -diketones. The slight shifts observed in the  $\nu(\text{C}=\text{O})+\nu(\text{C}=\text{C})$  of the complexes from the free ligand value of 1604  $\text{cm}^{-1}$  support the coordination through the oxygen of the carbonyl group [Omereg, 2012]. The absorption at 1382  $\text{cm}^{-1}$  in

$[\text{Ni}(\text{bta})(\text{bipy})(\text{NO}_3)].\text{H}_2\text{O}$  signifies the presence of nitrate in the complexes. This band occurred at 1384  $\text{cm}^{-1}$  in  $[\text{Ni}(\text{bta})(\text{phen})(\text{NO}_3)].\text{H}_2\text{O}$ . In the prepared bipyridine complexes, bands at 760  $\text{cm}^{-1}$  region are out of plane bending of ring hydrogens. Several studies have revealed that 1,10-phenanthroline and its complexes normally have two strong bands around 725 and 825  $\text{cm}^{-1}$  which are out of plane motion of the hydrogen atoms on the heterocyclic rings and hydrogen atoms on the middle ring respectively [Schilt *et al.*, 1959; Omereg *et al.*, 2015]. In the prepared 1,10-Phenanthroline complex, these bands were observed at 721  $\text{cm}^{-1}$  and 851  $\text{cm}^{-1}$ .

### Electronic Spectra/Magnetic

The electronic solution spectra of the complexes in methanol and the solid reflectance are presented in Table 1. Similar patterns are observed in the electronic spectra of metal  $\beta$ -diketonates and these complexes are known to exist in highly distorted stereochemistries [Odunola *et al.*, 2003]. The assignments of bands were made by comparing the spectra with those reported in literature [Odunola *et al.*, 2003, Silverstein *et al.*, 2005; Omereg, 2012]. Literature has shown that five coordinate high spin square pyramidal complexes are related to the tetragonal six coordinate complexes through the removal of one ligand from an octahedral arrangement. It is known that their spectra are similar to the six-coordinate tetragonal system [Lever, 1986].

The visible spectra of [Ni(bta)(phen)(NO<sub>3</sub>)]<sub>2</sub>.H<sub>2</sub>O and [Ni(bta)(bipy)(NO<sub>3</sub>)]<sub>2</sub>.H<sub>2</sub>O showed single broad

band in the region 16,658-17,131 cm<sup>-1</sup>, which is consistent with a square-pyramidal geometry for nickel(II) complexes [Preer and Gray 1970; Lever, 1986] while [Ni(bta)<sub>2</sub>tmen].H<sub>2</sub>O and [Ni(bta)(bzac)(bipy)] had bands in the region 13,029-17,182 cm<sup>-1</sup> assigned as octahedral geometry.

The intraligand  $\pi$ - $\pi^*$  and ligand to metal charge transfer (LMCT) transitions are observed in the region between 47,619-33,333 cm<sup>-1</sup> for the nickel(II) complexes [Preer and Gray 1970; Ogden and Selbin, 1968; Lever, 1986; Omereg and Woods, 2011]. [Ni(bta)<sub>2</sub>tmen].H<sub>2</sub>O and [Ni(bta)(bzac)(bipy)] had absorption bands typical of octahedral geometry which is corroborated by observed magnetic moments [Omereg, 2018a].

**Table 1: Relevant electronic solution spectra of nickel (II) mixed ligand complexes of 4,4,4-trifluoro-1-phenyl-1,3-butanedione with 2,2'-bipyridine, 1,10-phenanthroline and tetramethylethylenediamine**

[Ni(bta)phenNO <sub>3</sub> ]. H <sub>2</sub> O	[Ni(bta)bipyNO <sub>3</sub> ]. H <sub>2</sub> O	[Ni(bta) <sub>2</sub> tmen]. H <sub>2</sub> O	[Ni(bta)(bzac)(bipy)]	Tentative Assignment
<u>Methanol</u>	<u>Methanol</u>	<u>Methanol</u>	<u>Methanol</u>	$\pi_3$ - $\pi^*_5$
43,478 40,000	39,215		40,650	Benzenoid band/ $\sigma_L$ -3d <sub>xy</sub> / $\pi$ - $\pi$ (bipy.phen)
33,333	33,333	36,363		$\pi_3$ - $\pi^*_4$
30,303	30,303		30,864	$\pi$ -d
16,949	17,131	15,625	17,182 13,029	d-d

### Antimicrobial Activities

The microbial growth inhibitory activity of the ligands and compounds was evaluated in- vitro against Gram-positive bacteria, *Staphylococcus aureus* and *Bacillus subtilis*, Gram-negative bacteria, *Pseudomonas aeruginosa*, *Salmonella enterica*, and *Klebsiella pneumoniae*. The results are

shown in Table 2. The solvent methanol was used as negative control. [Ni(bta)<sub>2</sub>(tmen)] had pronounced activity on the tested bacteria (*Staphylococcus aureus*, *Bacillus subtilis*, *Klebsiella pneumoniae*, *Escherichia coli*, *Salmonella enterica*, *Pseudomonas aeruginosa*) except in *Pseudomonas aeruginosa* which showed moderate activity.

[Ni(bta)(phen)NO<sub>3</sub>] and [Ni(bta)(bipy)NO<sub>3</sub>] lacked antibacterial activity. The exceptionally poor activity shown by [Ni(bta)(phen)NO<sub>3</sub>] and [Ni(bta)(bipy)NO<sub>3</sub>] against the bacteria tested could be due to poor bacterial uptake of the compounds

[Omereg *et al.*, 2015]. The antibacterial activity of the [Ni(bta)<sub>2</sub>(tmen)] and [Ni(bta)(bzac)Bipy] compared favorably with that of gentamicin used at 8 and 4 mg/mL [Omereg *et al.*, 2015].

**Table 2: Antimicrobial activity data of  $\beta$ -diketone and the mixed Ligand nickel(II) complexes at 10mg/mL.**

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>
btaH	S	S	R	S	MS	VS
[Ni(bta)(phen)NO <sub>3</sub> ]	R	R	R	R	R	R
[Ni(bta)(bipy)(NO <sub>3</sub> )]	R	R	R	R	R	R
[Ni(bta) <sub>2</sub> (tmen)]	S	S	S	S	S	MS
[Ni(bta)(bzac)Bipy]	S	S	ND	24	ND	S
Gentamicin	MS	MS	MS	MS	MS	MS
Methanol	-	-	-	-	-	-

MS=Moderately Sensitive, S= Sensitive, R= organism resistant to the extract, ND=Not Done

*S. aur* = *Staphylococcus aureus*, *B. sub* = *Bacillus subtilis*, *K. pne* = *Klebsiella pneumonia*

*E. coli* = *Escherichia coli*, *S. ent* = *Salmonella enterica*, *P. aer* = *Pseudomonas aeruginosa*

## CONCLUSION

New nickel (II) complexes of 4,4,4-trifluoro-1-phenyl-1,3-butanedione mixed with 1,10-phenanthroline, 2,2'-bipyridine and tetramethylethylenediamine were prepared and characterized by elemental analysis, spectral and magnetic measurement and antimicrobial studies. The assignment of 5-coordinate, square pyramidal and octahedral geometries for the nickel (II) complexes is corroborated by magnetic, infrared, electronic spectral measurements.

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