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Synthesis, Physicochemical Properties and Antimicrobial Activities of Nickel(II) Complexes of 4,4,4-Trifluoro-1-(2-Naphthyl)-1,3-Butanedione and Their Adducts

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Abstract

Nickel(II) complex of 4,4,4-trifluoro-1-(2-Naphthyl)-1,3-butanedione(tfnb) and its 2,2′-bipyridine (bipy), ethylenediamine (en) and 1,10-phenanthroline (phen) adducts were synthesized and characterized by elemental analysis, infrared, electronic spectral, magnetic susceptibility measurements and antimicrobial activities. Three other nickel(II) compounds were synthesized along with these compounds. The bands in the 1651-1513 cm^{-1} have been assigned as ν(C=O) + ν(C=C). The infrared and electronic spectral measurements are indicative of a probable six-coordinate octahedral geometry for all the nickel(II) complexes and adducts. The antimicrobial activity shows that $[Ni(tfnb)_2]2H_2O$ complex and its adducts have no antibacterial and antifungal activities.

Keywords: 4,4,4-trifluoro-1-(2-Naphthyl)-1,3-butanedione(tfnb), 1,10-phenanthroline, 2,2′-bipyridine, spectra, antimicrobial

1. Introduction

β-diketones are highly versatile reagents for organic synthesis. They have been found to have remarkable application in metal extractions (Wenzel *et al.*, 1985), as catalysts (Bose *et al.*, 2016) and as precursors for Chemical Vapour Deposition (CVD) application (Banger *et al*., 2001). β-diketones and their complexes have attracted the attention of researchers because of their biological activities (Omoregie, 2012; Omoregie *et al*; 2016; Omoregie *et al*., 2017) and electrochemical features (Ahumada *et al.*, 2018).

The use of β-diketones in drug synthesis in pharmaceutical industry is well documented (Kawashi, *et al*., 1981). Metal complexes of β-diketones are also components of the materials used for charging electrostatographic toners to provide adequate negative charge (Hiroshi and Katsuhiko, 1987). A survey of the literature indicates that a number of β-diketones have been extensively studied whereas there is no report on nickel complex of 4,4,4-trifluoro-1-(2-Naphthyl)-1,3-butanedione and 2,2′-bipyridine, ethylenediamine and 1,10-phenanthroline adducts and no attempt has been made to investigate and compare the results of their infrared, solution spectra and magnetic properties. Hence, there is the need to synthesize the metal(II) complexes and their adducts. In continuation of studies on *β*-diketones and their derivatives (Omoregie, 2012; Omoregie *et al*; 2016; Omoregie *et al*., 2017), a report is presented on the synthesis, magnetic properties, spectral measurements and the antimicrobial activity of nickel(II) complexes of 4,4,4-trifluoro-1-(2-napthyl)-1,3-butanedione (tfnb) and their 2,2′-bipyridine (bipy), ethylenediamine (en) and 1,10-phenanthroline (phen) adducts.

2. Materials and Methods

2.1 Reagents

Reagents used include nickel nitrate, 1,10-phenanthroline, 2,2 ́-bipyridine, ethylenediamine, 4,4,4-trifluoro-1-(2-napthyl)-1,3-butanedione, methanol, chloroform and methanol. The reagents which were of analytical grade obtained from Aldrich Chemicals and British Drug House (BHD) Chemicals Limited were utilized without further purification.

2.2 Preparation of $[Ni(tfnb)_2]2H_2O$

$NiNO_3\,6H_2O$ (0.218 g, 0.75 mmol) dissolved in water (1.2 mL) was added to 4,4,4-trifluoro-1-(2-napthyl) 1,3-butanedione (0.400 g, 1.50 mmol) in methanol (5 mL). The mixture was stirred for 1 hr. and the green solid product was collected by filtration, washed with water and methanol, and dried *in vacuo*. Anal. Calc. for $C_{28}H_{20}F_6NiO_6$: C, 53.80; H, 3.22; Ni, 9.39. Found C, 53.75; H, 3.60; Ni, 9.83.

2.3 Preparation of Ni(tfnb)$_2$phen

$NiNO_3$ $6H_2O$ (0.5462 g, 1.88 mmol) dissolved in water (1.2 mL) was added to a mixture of 4,4,4-trifluoro-1-(2-napthyl) 1,3-butanedione (0.50 g, 1.88 mmol) and 1,10-Phenanthroline(0.3723 g, 1.88 mmol) in methanol (5 mL). The mixture was stirred for one hr. and the green solid product was collected by filtration, washed with water and methanol, and dried *in vacuo*. Anal. Calc. for $C_{40}H_{24}F_6N_2NiO_4$: C, 62.45; H, 3.14; N, 3.64; Ni, 7.63. Found C, 62.29; H, 3.70; N, 3.72 Ni, 7.38.

2.4 Preparation of Ni(tfnb)$_2$bipy

$NiNO_3$ $6H_2O$ (0.5462 g, 1.88 mmol) dissolved in water (1.2 mL) was added to a mixture of 4,4,4-trifluoro-1-(2-napthyl) 1,3-butanedione (0.50 g, 1.88 mmol) and 2,2-bipyridine (0.2933 g, 1.88 mmol) in methanol (5 mL). The mixture was stirred for 1hr. and the green solid product was collected by filtration, washed with water and methanol, and dried in vacuo. Similar procedure was used for the preparation of ethylenediamine adduct of the complex. Anal. Calc. for $C_{38}H_{24}F_6N_2NiO_4$: C, 61.24; H, 3.25; N, 3.76; Ni, 7.63. Found C, 61.37; H, 3.60; N, 3.77 Ni, 7.88.

2.5 Preparation of [Ni(tfnb)(bzac)2H_2O]H_2O

Nickel chloride hexahydrate (0.36 g, 1.50 mmol) dissolved in 1.8 mL water was added to a mixture of benzoylacetone (0.24 g, 1.54 mmol) and 4,4,4-trifluoro-1-(2-napthyl) 1,3-butanedione (0.4 g, 1.54 mmol) in 5 mL methanol. The reaction mixture was stirred at room temperature for 1 h. The green solid product was collected by filtration, washed with water and methanol, and dried in vacuo. Anal. Calc. for $C_{24}H_{23}F_3NiO_7$: C, 53.47; H, 4.30; Ni, 10.89. Found C, 53.68; H, 3.67; Ni, 10.78.

2.6 Preparation of [Ni(tfnb)(bzac)(phen)]

Nickel chloride hexahydrate (0.36 g, 1.50 mmol) dissolved in 1.8 mL water was added to a mixture of benzoylacetone (0.24 g, 1.50 mmol) and 4,4,4-trifluoro-1-(2-napthyl) 1,3-butanedione (0.4 g, 1.50 mmol) in 5 mL methanol. The reaction mixture was stirred at room temperature for 1 h. 1,10-phenanthroline (0.30 g, 1.50 mmol) was added to the reaction and stirred for 1 hr. The green solid product was collected by filtration, washed with water and methanol, and dried in vacuo. Similar procedure was used for the preparation of ethylenediamine adduct of the complex. Anal. Calc. for $C_{40}H_{24}F_6N_2NiO_4$: C, 62.45; H, 3.14; N, 3.64; Ni, 7.63. Found C, 62.29; H, 3.70; N, 3.72 Ni, 7.38.

2.7 Microbiological Studies

2.7.1 Methodology

Ten microorganisms were used in the screening using a disc diffusion test. This test was performed to screen and determine the complex potentials according to their inhibition zone diameter. A mass of the culture medium (Nutrient agar and Sabouraud Dextrose agar) was weighed and dissolved in distilled water. The mixing was heated to homogenize and sterilized in an autoclave for 15 minutes before the mixture was then poured into Petri dishes and left to solidify. The compounds were weighed and dissolved in 2mL of methanol, making a concentration of 50 mg/mL, from which serial dilution of about four more concentrations were prepared.

A colony from a 24-hour fresh culture of the various organisms was dissolved in 5mL of sterile distilled water which was spread on the prepared culture agar. Sizeable hole was bored on the culture plate and the different concentrations of the complex were added into each hole. The Petri dishes were then incubated for 24 hours for the bacteria and 48 hours for fungi before the inhibition zone diameter was measured. The results are presented in Table 5.

2.8 Physical Measurements

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

Infrared spectra were measured using nujol on Perkin Elmer Spectrophotometer 11 FT-IR. The electronic spectra of the compounds in methanol and 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, Nigeria.

3. Results and Discussion

Table 1 shows the analytical data, colour, percentage yield and room temperature magnetic moments (μ_{eff}) of the prepared nickel(II) complexes. Percentage metal analysis was in significant agreement with those calculated for the proposed formula. Nickel(II) compounds were obtained as various shades of green.

3.1 Magnetic Moment

Literature has indicated that the magnetic moment of nickel(II) complexes have values between 2.8-4.2 B.M. The expected spin only ($\mu_{s.o}$) value for nickel(II) complexes is 2.83 B.M. but there are always deviations which are attributed to both orbital contributions and spin-orbit coupling of the $^3A_{2g}$ and $^3T_{2g}(F)$ terms depending on the stereochemistry (Patel and Woods, 1990; Greenwood and Earnsaw, 1997). Experimental moments of 2.9-3.3 B.M. were observed for octahedral nickel due to spin-orbit coupling of the $^3A_{2g}$ and $^3T_{2g}(F)$ terms while moments of 3.2-4.1 B.M. were observed for tetrahedral nickel due to orbital contributions giving higher moments than the spin-only value of 2.83 B.M. (Patel and Woods, 1990).

The room temperature magnetic moment of the synthesized nickel(II) compounds were in the range 2.86-3.07 B.M. which is indicative of octahedral geometries except for $[Ni(tfnb)_2Phen]H_2O$ with lower moment of 2.36 B.M. which may be due to interconversion of stereochemistries and/or dimerization (Osowole et al, 2000).

3.2 Infrared Spectra

The relevant infrared bands are presented in Table 2. Assignments of the infrared bands were made by comparing the spectra of the compounds with those already reported in the literature on similar systems (Omoregie, 2012; Omoregie *et al.*, 2018). Literature review has shown that infrared spectra of β-diketones shows an appreciable coupling of different vibrational modes due to overlap of absorption frequencies (Nakamoto et al., 1961, Woods *et al*; 2009a; Woods *et al*., 2009b; Omoregie *et al.*, 2014; Omoregie and Woods, 2011). The metal-free ligand, HL, which consists of the uncoordinated C=O+C=C stretching vibrations has a double band in the 1603-1516 cm^{-1} region**.** Hypsochromic shifts of $\nu_{as}(C=O)+ \nu_{as}(C=C)$ vibrations were observed in all the adducts relative to the parent complex except in $[Ni(tfnb)_2Bipy]$ which had a bathochromic shift.

CH deformation band of 2,2′-bipyridine was observed as strong band at 759 cm^{-1} region while the phenanthroline adducts bands were observed around 726-729 cm^{-1} and 847 cm^{-1} region. The presence of bands due to (M—O) and (M—N) in the range 420-696 cm^{-1} in the 2,2′-bipyridine and 1,10- phenanthroline adducts is further evidence of coordination (Patel and Woods, 1990b).

3.3 Electronic Spectra

The electronic spectra of the ligand, the complexes and their adducts in chloroform and methanol are presented in Table 3. The ultraviolet spectra of the nickel compounds were characterised by four characteristic peaks at 27,248-30,120, 33,003-34,129, 37,037-40,161 and 44,053-49,020 cm^{-1} assigned as n-π*/π-d, π_3-π_4*, Benzenoid band/σL-3dxy/π-π (bipy.phen) and π_3-π_5* respectively. Three transitions were expected for an octahedral nickel(II) ion in the region 7,000-13,000 cm^{-1}, 11,000-20,000 cm^{-1} and 19,000-27,000 cm^{-1} ; and they were assigned to the $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$, $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ and $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ respectively. The visible spectra of the nickel complexes and adducts had two absorption bands each in the range 11,628-13038 cm^{-1} and 15,432-17,361 cm^{-1} assigned to $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$, $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ transitions of an octahedral geometry (Lever, 1986).

3.4 Antimicrobial Activities

The antimicrobial screening of the synthesized complexes was investigated using Gram-positive bacteria, *Staphylococcus aureus* and *Bacillus subtilis*, Gram-negative bacteria, *Pseudomonas aeruginosa, Salmonella typhi, Escherichia coli* and *Klebsiella pneumoniae* and four fungi (*Candida albicans*, *Aspergillus niger, Penicillium notatum, Rhizopus stolonifer*). The results of antibacterial activity are presented in Table 5. The ligand indicated a significant activity against the tested organisms except *Aspergillus niger, Penicillium notatum, Rhizopus stolonifer* in relation to which it indicated moderate activity. $[Ni(tfnb)_2 2H_2O]$, $[Ni(tfnb)_2(Phen)]$ $[Ni(tfnb)_2(Bipy)]$ and $[Ni(tfnb)_2(en)]$ lacked antibacterial and antifungal activities. This may be attributed to poor organism uptake (Omoregie *et al.*, 2015). $[Ni(tfnb)(bzac)2H_2O]$, [Ni(tfnb)(bzac)(Phen)] and [Ni(tfnb)(bzac)(en)] revealed moderate antibacterial activity but lacked antifungal activity except [Ni(tfnb)(bzac)(Phen)] which was sensitive to *Aspergillus niger* and *Penicillium notatum.*

4. Conclusion

Nickel(II) complexes of 4,4,4-trifluoro-1-(2-Naphthyl)-1,3-butanedione and adducts were found to be octahedral in geometry as corroborated by magnetic, electronic and IR spectra measurements. The antimicrobial activities showed that ligand is significantly sensitive to all the tested bacteria but moderately sensitive to the fungi (*Aspergillus niger, Penicillium notatum, Rhizopus stolonifer*). $[Ni(tfnb)(bzac)2H_2O]$ H_2O, [Ni(tfnb)(bzac)(en)] and [Ni(tfnb)(bzac)(Phen)] had good comparison with the standard drugs, Gentamycin and Ketoconazole and therefore can be used as disinfectant on inanimate materials.

Table 1. Analytical and physical data of nickel(II) complexes of 4,4,4-trifluoro-1-(2-napthyl)1,3-butanedione and their adducts

Compounds	Mol. wt. (g mol^{-1})	Colour	M.pt(^oC)	%Metal Exp (Cal)	Yield%	μ_{eff} (BM)
$[Ni(tfnb)_2]2H_2O$	625.13	Light Green	213-215	9.83(9.39)	32.70	3.07
$[Ni(tfnb)_2(Phen)]$	769.33	Deep Green	239-241	7.38(7.63)	21.00	2.36
$[Ni(tfnb)_2(Bipy)]$	745.32	Green	237-239	7.21(7.87)	80.30	2.86
$[Ni(tfnb)_2(en)]$	649.23	Green	201-203	9.22(9.04)	67.60	3.02
$[Ni(tfnb)(bzac)]3H_2O$	539.13	Green	215-217	10.78(10.89)	21.34	2.85
$[Ni(tfnb)(bzac)(Phen)]\ 2H_2O$	701.32	Green	268-270	7.19(8.37)	45.67	2.81
[Ni(tfnb)(bzac)(en)]	545.18	Light green	278-280	10.78(10.77)	39.81	

Table 2. Relevant infrared bands (cm^{-1}) of nickel(II) complexes of 4,4,4-trifluoro-1-(2-napthyl)1,3-butanedione and their adducts

Formula	ν(OH)	ν(NH)	C=O, C=C	ν_s(C-H) phen/bipy
Tfnb			1603s,1516w	
$[Ni(tfnb)_2.2H_2O]$	3415b		1610s, 1537m, 1519w	
$[Ni(tfnb)_2(Phen)]$			1612s, 1532m, 1518m	847w, 726m
$[Ni(tfnb)_2(Bipy)]$			1609s, 1574w, 1524w	759m
$[Ni(tfnb)_2(en)]$		3368m, 3288m	1613s, 1532	
$[Ni(tfnb)(bzac)]3H_2O$	3384b		1651w,1609s,1599w,1575,1537s,1519m	
[Ni(tfnb)(bzac)(Phen)]			1613s, 1588m	848s, 729s
[Ni(tfnb)(bzac)(en)]		3366s, 3292s	1611vs,1585m,1577s,1531s,1513s	

Table 3. Relevant electronic solution spectra of nickel(II) complex of 4,4,4-trifluoro-1-(2-napthyl)1,3-butanedione and their 2,2′-bipyridine, ethylenediamine and 1,10-phenanthroline

Tfnb / Methanol	$[Ni(tfnb)_2.2H_2O$	$[Ni(tfnb)_2Phen]$ H_2O	$[Ni(tfnb)_2Bipy]$	$[Ni(tfnb)_2en]$	Tentative Assignment
46,948		46,729 42,918	46,729 40,650	46,948 39,063	π_3-π_5*
40,161		37,594	37,736	37,879	Benzenoid band
34,247		34,247	32,573	34,364	π_3-π_4*
30,120		29,762 27,248	29,586 27,248	29,762	n-π*/π-d
	15,873br 13,123	15,798 12,970	15,924 13,228	16,155 14,327	$^3A_2g\leftarrow{}^3T_1g(F)$ $^3A_2g\leftarrow{}^3T_2g(F)$
Chloroform 47,847	41,494	44,843	45,662	43,860 41,667	π_3-π^*_5
39,370 37,453	39,063	39,216 37,453	39,841	39,841 38,911	Benzenoid band/$\sigma_L 3d_{xy}$/π-π(bipy.phen)
34,364	34,965	34,483	34,247	34,843,	π_3-π^*_4
28,986	28,653 27,248	28,902	29,155	29,155	n-π*/π-d
	16,393 13,123	17,331br 12,165	17,241br 13,038	-	$^3A_2g\leftarrow{}^3T_1g(F)$ $^3A_2g\leftarrow{}^3T_2g(F)$

Table 4. Relevant electronic solution spectra of nickel(II) complex of 4,4,4-trifluoro-1-(2-napthyl)1,3-butanedione/benzoylacetone and their 2,2′-bipyridine, ethylenediamine and 1,10-phenanthroline(cm^{-1})

Tfnb / Chloroform	$[Ni(tfnb)(bzac)2H_2O]H_2O$	[Ni(tfnb)(bzac)(Phen)]	[Ni(tfnb)(bzac)(en)]	Tentative Assignment
47,847 42,553			41,322	π_3-π^*_5
39,370 37,453		37,453	39,683	Benzenoid band/$\sigma_L 3d_{xy}$/π-π(bipy.phen)
34,364	34,513	34,513	34,513	π_3-π^*_4
28,986	29,851	28,818	29,070	n-π*/π-d
	11,919	17,215	17,361	$^3A_2g\leftarrow{}^3T_1g(F)$ $^3A_2g\leftarrow{}^3T_2g(F)$

Table 5a. Antimicrobial activity of ligand, $[Ni(tfnb)_2]2H_2O$ and adducts

Compounds	*S. aur*	*E. coli*	*B. sub*	*P. aer*	*S. typhi*	*K. pne*	*Ca*	*An*	*Pen*	*Rs*
Tfnb	S	S	S	S	S	S	S	MS	MS	MS
$[Ni(tfnb)_2]2H_2O$	R	R	R	R	R	R	R	R	R	R
$[Ni(tfnb)_2Phen]$	R	R	R	R	R	R	R	R	R	R
$[Ni(tfnb)_2Bipy]$	R	R	R	R	R	R	R	R	R	R
$[Ni(tfnb)_2en]$	R	R	R	R	R	R	R	R	R	R
Gentamycin/ *Tioconazole	MS	MS	MS	MS	ND	MS	MS	MS	ND	ND
Methanol	No activities									

Table 5b. Antimicrobial activity of $[Ni(tfnb)(bzac)]3H_2O$ and adducts

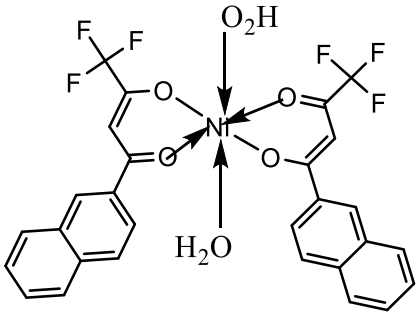
Compounds	*S. aur*	*E. coli*	*B. sub*	*P. aer*	*Ca*	*An*	*Pen*
Tfnb	S	S	S	S	S	MS	MS
$[Ni(tfnb)(bzac)2H_2O]H_2O$	MS	MS	MS	MS	–	–	–
[Ni(tfnb)(bzac)(Phen)]	MS	MS	MS	MS	–	S	S
[Ni(tfnb)(bzac)(en)]	MS	MS	MS	MS	–	MS	–
N,N-dimethylformamide (-c)	-	-	-	-	–	–	–
Gentamycin/ *Ketoconazole (+c)	MS	MS	MS	MS	MS	MS	S

S. aur = Staphylococcus aureus; B. sub = Bacillius subtilis; K. pne = Klebsiella pneumonia;

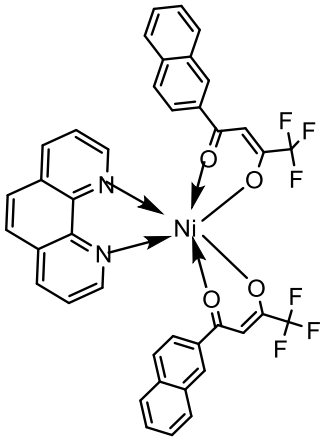
E. coli = Escherichia coli; S.typi=Salmonella typhi; P. aer = Pseudomonas aeruginosa;

Ca = Candida albicans; An = Aspergillus niger; Pen = Penicillium notatum

Rs=Rhizopus stolonifer; R= organism resistant to the extract; MS=organism moderately sensitive to extract; S=organism adequately sensitive to extract; ND=not done



$[Ni(tfnb)_2.2H_2O$



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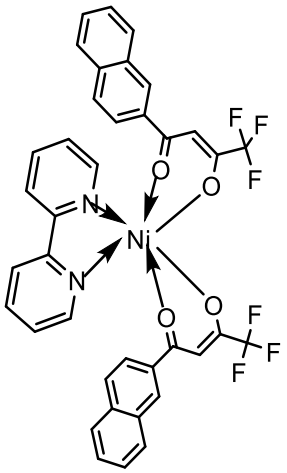

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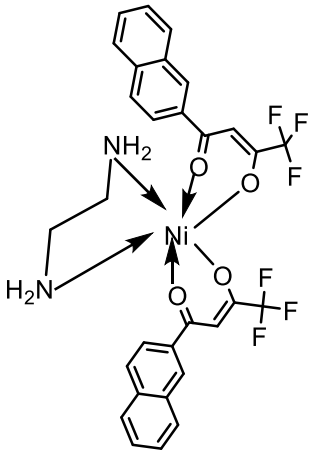

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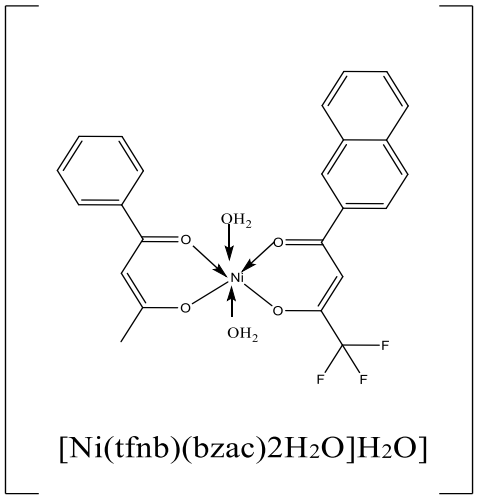
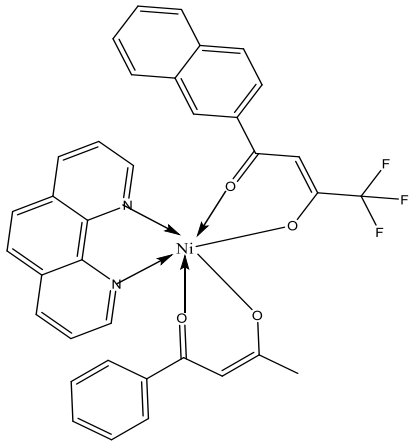
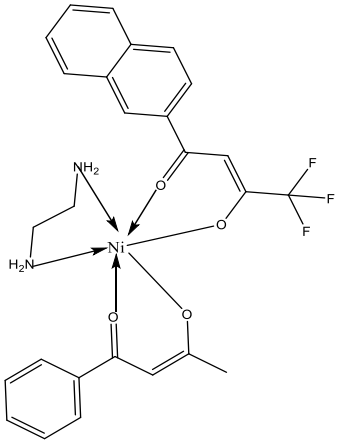


[Ni(tfnb)(bzac)2H$_2$O]H$_2$O]



[Ni(tfnb)(bzac)(phen)]



[Ni(tfnb)(bzac)(en)]

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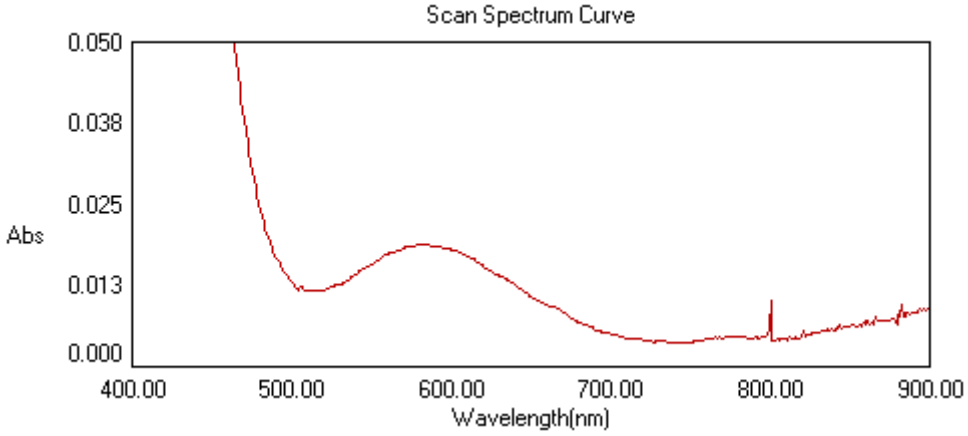
Appendix



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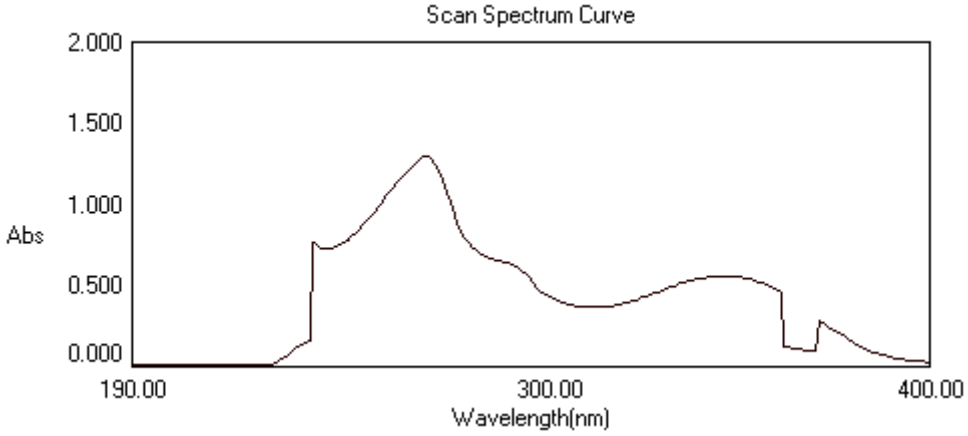

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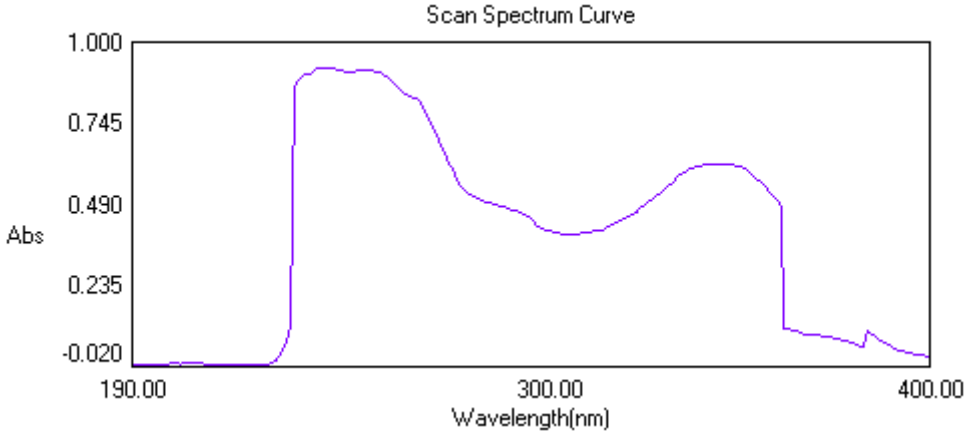

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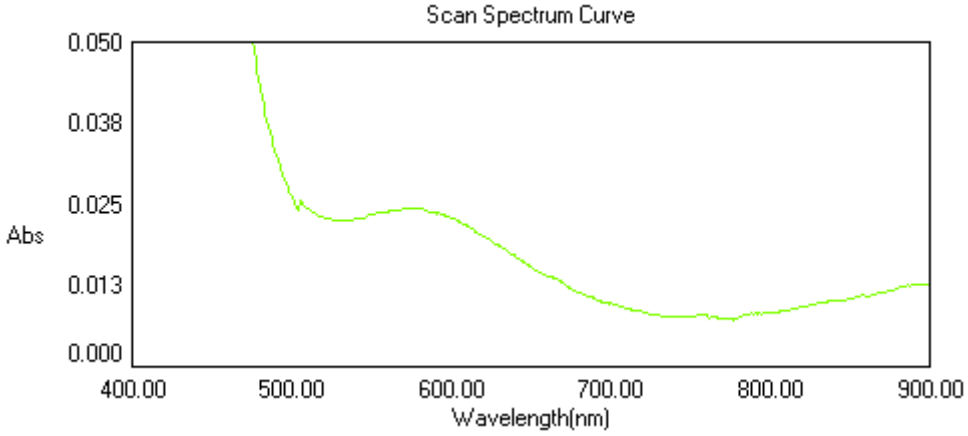

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