

Synthesis and Physicochemical Studies of Nickel(II) Complexes of Various 2-Alkyl-1-phenyl-1,3-butanediones and Their 2,2'-Bipyridine and 1,10-Phenanthroline Adducts

Oluwatola Omoregie (Corresponding author)

Department of Chemistry, Faculty of Science, University of Ibadan

Ibadan, Oyo-State, Nigeria

E-mail: tolaomoregie@yahoo.co.uk

Joseph Woods

Department of Chemistry, Faculty of Science, University of Ibadan

Ibadan, Oyo-State, Nigeria

E-mail: jaowoods@yahoo.com

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Abstract

Some 2-substituted-1-phenyl-1,3-butanedionato nickel(II) complexes and their 2,2'-bipyridine (bipy) and 1,10-phenanthroline (phen) adducts have been synthesized and characterized by elemental analyses, infrared, electronic spectral studies, conductance, and magnetic susceptibility measurements. The electronic spectral data have been interpreted in terms of the $\pi_3 \rightarrow \pi_4^*$ and other transitions and the effect of the substituents at β -position on the different transitions determined. The infrared spectra of the nickel(II) complexes showed that the frequencies of the asymmetric C=O + C=C stretching vibrations were lowered from their ligand values.

Keywords: Nickel(II) complexes, 1-phenyl-1,3-butane, 2,2'-bipyridine (bipy) and 1,10-phenanthroline, Magnetic susceptibility, Conductometry

1. Introduction

β -diketone and their metal complexes are among the most widely studied coordination compounds since they have wide application in the industries as catalyst (Schwieger et al, 2009; Xingbang et al, 2009; Ferreira et al, 2002; Poncelet et al, 2005; Lassahn et al, 2005; Campelo et al, 2006) and also as precursors for chemical vapour deposition (Zhang et al, 2006; Nable et al, 2003; Banger et al, 2001; Dela Rosa et al, 2003). Thermal transfer printing materials containing metal β -diketonates exhibit good whiteness and image stability (Miura et al, 1993) and it has been found that toner containing metal complexes of β -diketones are stable, controllable and capable of producing clear colour images even at high temperature and high humidity without producing copier stain (Hiroshi and Katsuhiko, 1987).

Studies on the effect of substituents on the spectra properties of metal β -diketonates have been reported (Nakamoto et al, 1959; Nakamoto et al, 1962; Patel and Woods, 1990; Woods and Patel, 1994) but there is dearth of information on 2-substituted-1-phenyl-1,3-butanedionato nickel(II) complexes and their 2,2'-bipyridine and 1,10-phenanthroline adducts. In continuation of our studies on β -diketones and their derivatives (Woods et al, 2009a; Woods et al, 2009b), we report the results of our investigations on bis (2-substituted-1-phenyl-1,3-butanedionato) nickel(II) complexes $\text{Ni}(\text{R-bzac})_2$ (R = H, Me, Et, n-Pr, i-Pr and n-Bu) and their adducts with 2,2'-bipyridine and 1,10-phenanthroline.

2. Experimental

2.1 Reagents

1-phenyl-1,3-butanedione (bzacH) (Aldrich chemicals), potassium carbonate, nickel(II) acetate, nickel(II) chloride, methyl iodide, ethyl iodide, n-propyl iodide, i-propyl iodide, n-butyl iodide (Aldrich chemicals), 2,2'-bipyridine and 1,10-phenanthroline (Analytical grade).

2.2 Preparation of the Ligands

The 2-alkyl-1-phenyl-1,3-butanediones were prepared according to a literature procedure (Patel and Woods, 1990).

2.3 Preparation of $\text{Ni}(\text{Me-bzac})_2 \cdot 2\text{H}_2\text{O}$

3 mL 2-Me-1-phenyl-1,3-butanedione (3.39 g, 19.2 mmol) dissolved in 10 mL methanol was added to nickel(II) chloride hexahydrate (2.28 g, 9.60 mmol) in 3 mL water, while stirring and the pH was raised to 8. The precipitated solids were washed with 40% methanol and dried over silica gel. Similar procedure was used for preparation of the other nickel(II) complexes.

2.4 Preparation of $\text{Ni}(\text{Me-bzac})_2\text{bipy}$

The 2,2'-bipyridine adduct of $\text{Ni}(\text{Me-bzac})_2 \cdot 2\text{H}_2\text{O}$ was prepared by mixing 1.2 mL Me-bzacH (1.25 g, 7.07 mmol) and 2,2'-bipyridine (0.55 g, 3.53 mmol) in 80 mL methanol. Nickel(II) acetate tetrahydrate (0.88 g, 3.53 mmol) dissolved in 12 mL 50% methanol was added dropwise to the mixture and stirred for 1 hour. The precipitates obtained were filtered, washed with few drops of acetone and dried in vacuo. Similar procedure was used for preparation of the other 2,2'-bipyridine and 1,10-phenanthroline adducts except 2,2'-bipyridine and 1,10-phenanthroline adducts of $\text{Ni}(\text{bzac})_2 \cdot 2\text{H}_2\text{O}$ which were prepared by mixing the base with the complex prior to dissolution in the solvent.

2.5 Physical Measurements

Elemental analyses for C, H and N were determined by the analytical laboratory of Geological Survey of Ethiopia while nickel was determined using a complexometric method (Vogel, 1986). The analytical and physical data are presented in Tables 1 and 2. The room temperature magnetic susceptibilities of the compounds were measured by MSB-AUTO (Sherwood scientific).

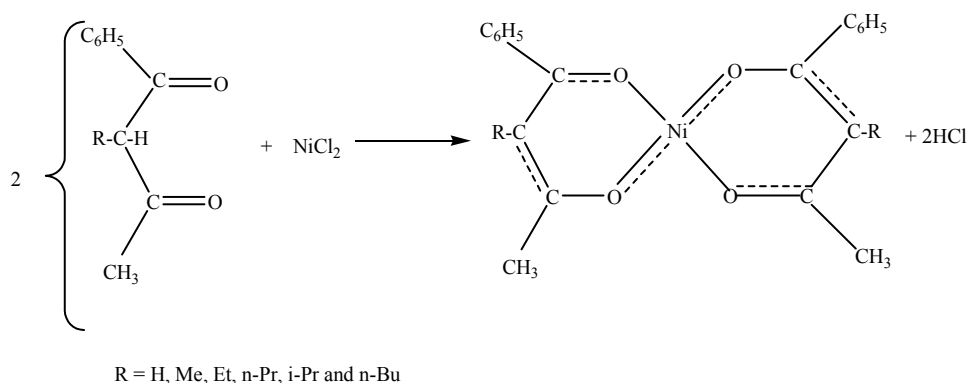
The electrolytic conductivities of the soluble compounds in nitromethane at room temperature were determined using Digital conductivity meter (Labtech).

The solution spectra of the compounds in methanol and chloroform were recorded on a Unicam UV-Visible Spectrophotometer using 1 cm glass cell.

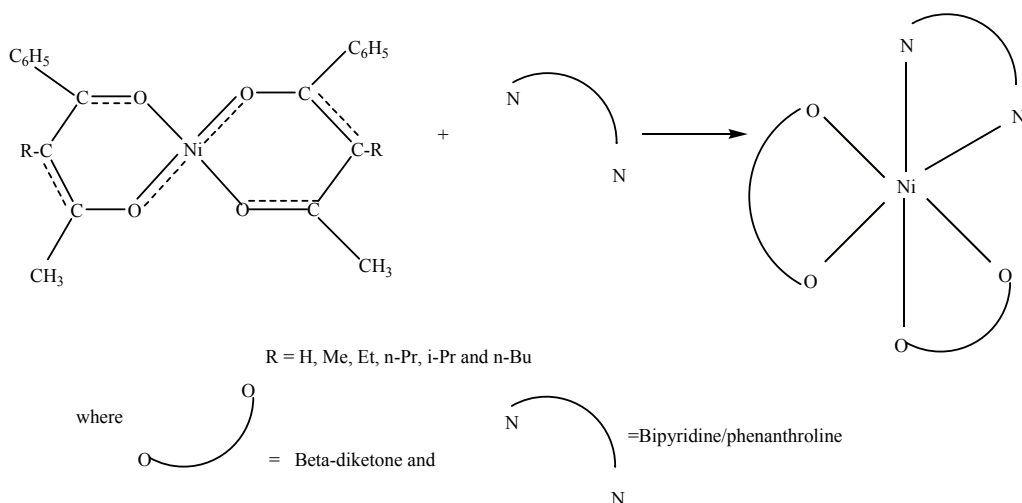
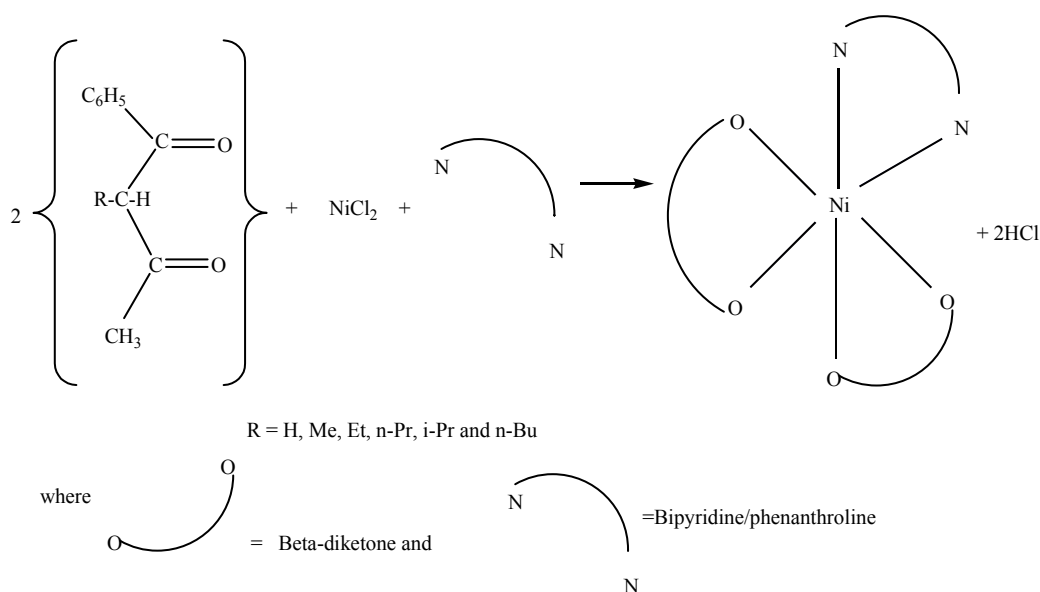
The reflectance spectra of the nickel(II) complexes were recorded on a Perkin Elmer Lambda 950 UV/VIS spectrophotometer at the Department of Chemical Engineering, Faculty of Technology, Addis Ababa University, Ethiopia using calcium carbonate as reference. The infrared spectra of the compounds as pressed KBr disc were recorded on Perkin Elmer Spectrophotometer BX FT-IR.

3. Results and Discussion

The reaction of 2-alkyl-1-phenyl-1,3-butanedione with metal salt is represented by equation 1 while that of the metal complex and base is represented by equation 2. The adducts prepared directly from metal salt are represented by equation 3. Reasonable yield of the precipitates were obtained.



EQUATION 1: PREPARATION OF NICKEL(II) COMPLEX

**EQUATION 2: PREPARATION OF NICKEL(II) ADDUCT****EQUATION 3: PREPARATION OF NICKEL(II) ADDUCT**

The analytical data, colours, %yields, melting points/decomposition temperatures and room temperature magnetic moments (μ_{eff}) of the complexes are given in Table 1. All the compounds were obtained as various shades of green except few that were pinkish in colour. They exhibited good solubility in chloroform and methanol except few. The microanalytical data for the compound agree with the calculated values (Table 2).

The principal infrared bands are presented in Table 3. Infrared studies on diketones have shown that electron releasing substituents give rise to low $\nu_{\text{as}}(\text{C}=\text{O}) + \nu_{\text{as}}(\text{C}=\text{C})$ (Hancock and Thornton, 1969; Patel and Adimado, 1979). Infrared spectra of 2-substituted-1-phenyl-1,3-butanediones studied showed a decrease in frequency as the electron in the system increases except Me-bzacH and i-Pr-bzacH which showed an increase in frequency. Reduction of the frequency of $\nu_{\text{as}}(\text{C}=\text{O}) + \nu_{\text{as}}(\text{C}=\text{C})$ is an indication of the positive inductive effect of the alkyl group on the system. Single bands of $\nu_{\text{s}}(\text{C}-\text{O}) + \delta\text{C}-\text{H}$ were observed in all the ligands except i-Pr-bzacH had two distinct bands. Reduction of the coupled $\nu_{\text{s}}(\text{C}-\text{O}) + \delta\text{C}-\text{H}$ vibrations of 2-substituted-1-phenyl-1,3-butanedione was observed as the length of the alkyl group increases except i-Pr-bzacH which had increased frequency. The reduction in frequency also indicates the positive inductive effect of the alkyl group on the system. The methyl deformation bands of the ligands studied were observed in the $1421\text{--}1359\text{ cm}^{-1}$ region (Wood and Patel, 1994; Patel and Woods, 1990; Tanaka et al,

1969; Koshimura et al, 1973; Nakamoto et al, 1961). These bands were observed as double bands except n-Bu-bzacH which had a single absorption band. The infrared spectra of the nickel(II) complexes showed that the frequencies of the asymmetric C=O + C=C stretching vibrations were lowered from the ligand values in the order: $\text{Ni}(\text{n-Pr-bzac})_2 \cdot 2\text{H}_2\text{O} > \text{Ni}(\text{Me-bzac})_2 \cdot 2\text{H}_2\text{O} > \text{Ni}(\text{Et-bzac})_2 \cdot 2\text{H}_2\text{O} > \text{Ni}(\text{i-Pr-bzac})_2 \cdot 2\text{H}_2\text{O} > \text{Ni}(\text{bzac})_2 \cdot 2\text{H}_2\text{O} > \text{Ni}(\text{n-Bu-bzac})_2 \cdot 2\text{H}_2\text{O}$. The order shows that the substituted Ni(II) complexes have larger bathochromic shift than the unsubstituted complex except $\text{Ni}(\text{n-Bu-bzac})_2 \cdot 2\text{H}_2\text{O}$ which had hypsochromic shift. The larger bathochromic shift probably indicates the positive inductive effect of the alkyl group on the system. The $\nu_{\text{as}}(\text{C=O}) + \nu_{\text{as}}(\text{C=C})$ stretching vibrations of these nickel(II) complexes were observed in the range $1560\text{--}1726\text{ cm}^{-1}$ while $\nu_{\text{s}}(\text{C-O}) + \delta\text{C-H}$ were observed in the $1450\text{--}1492\text{ cm}^{-1}$ region with $\text{Ni}(\text{bzac})_2 \cdot 2\text{H}_2\text{O}$, $\text{Ni}(\text{Et-bzac})_2 \cdot 2\text{H}_2\text{O}$, and $\text{Ni}(\text{n-Bu-bzac})_2 \cdot 2\text{H}_2\text{O}$ having multiple bands. A decrease in $\nu_{\text{s}}(\text{C-O}) + \delta\text{C-H}$ of 2-substituted-1-phenyl-1,3-butanedionato nickel(II) complexes was observed as the length of the alkyl group increases except $\text{Ni}(\text{Et-bzac})_2 \cdot 2\text{H}_2\text{O}$ and $\text{Ni}(\text{n-Bu-bzac})_2 \cdot 2\text{H}_2\text{O}$ which had increased frequency. The decrease in frequency indicates the positive inductive effect of the alkyl group on the system (Bamkole and Ogunkoya, 1981). The frequency of the methyl deformation band decreases as the length of the alkyl group increases with the exception of $\text{Ni}(\text{n-Pr-bzac})_2 \cdot 2\text{H}_2\text{O}$ and $\text{Ni}(\text{n-Bu-bzac})_2 \cdot 2\text{H}_2\text{O}$ which had increased frequency.

The $\nu_{\text{as}}(\text{C=O}) + \nu_{\text{as}}(\text{C=C})$ vibrations of most of the adducts were observed at higher frequency relative to the parent complexes except $\text{Ni}(\text{Me-bzac})_2\text{bipy}$, $\text{Ni}(\text{Et-bzac})_2\text{bipy}$, $\text{Ni}(\text{n-Pr-bzac})_2\text{bipy}$, $\text{Ni}(\text{n-Bu-bzac})_2\text{bipy}$, $\text{Ni}(\text{n-Bu-bzac})_2\text{phen}$ and $\text{Ni}(\text{bzac})_2\text{phen} \cdot 2\text{H}_2\text{O}$ which had bathochromic shifts while $[\text{Ni}(\text{bzac})(\text{bipy})(\text{H}_2\text{O})_2](\text{bipy})$ and $\text{Ni}(\text{Et-bzac})_2\text{phen}$ had no shift. The strength of the bonds in the adducts and the electron density on nickel(II) ion could be inferred from these shifts (Holtzclaw and Collman, 1957). The $\nu_{\text{as}}(\text{C=O}) + \nu_{\text{as}}(\text{C=C})$ vibrational modes of metal β -diketonate observed at lower frequency shifts could be an indication of stronger Ni-O bonds in the chelate ring due to increased electron delocalization as a result of weaker Ni-N (base) interactions (Patel and Woods, 1990). The $\nu_{\text{s}}(\text{C-O}) + \delta\text{C-H}$ and $\delta_{\text{as}}(\text{CH}_3) + \delta_{\text{s}}(\text{CH}_3)$ of the 2,2'-bipyridine adduct of $\text{Ni}(\text{R-bzac})_2$ were observed at higher frequency relative to the $\text{Ni}(\text{bzac})_2$ adducts except $\delta_{\text{as}}(\text{CH}_3) + \delta_{\text{s}}(\text{CH}_3)$ of the $\text{Ni}(\text{n-Pr-bzac})_2\text{bipy}$ which occurred at lower frequency. This is attributed to the positive inductive effect of the alkyl groups. The magnitude of the shift of $\nu_{\text{s}}(\text{C-O}) + \delta\text{C-H}$ was the same for methyl, ethyl and n-Propyl substituents ($\Delta\nu = +34$) while those of the i-Propyl and n-Butyl were ($\Delta\nu = +2$) and ($\Delta\nu = +29$) respectively. Higher frequency shifts of varying magnitude were observed in the $\delta_{\text{as}}(\text{CH}_3) + \delta_{\text{s}}(\text{CH}_3)$ of the 2,2'-bipyridine adducts relative to $[\text{Ni}(\text{bzac})(\text{bipy})(\text{H}_2\text{O})_2](\text{bzac})$ with the exception of $\text{Ni}(\text{iPr-bzac})_2\text{bipy}$ which had lower frequency shift. The shift is in the order: $\text{Ni}(\text{nPr-bzac})_2\text{bipy}(\Delta\nu = +5) > \text{Ni}(\text{Me-bzac})_2\text{bipy} = \text{Ni}(\text{Et-bzac})_2\text{bipy}(\Delta\nu = +3) > \text{Ni}(\text{iPr-bzac})_2\text{bipy}(\Delta\nu = -14)$. Higher frequency shift of $\delta_{\text{as}}(\text{CH}_3) + \delta_{\text{s}}(\text{CH}_3)$ of the 1,10-phenanthroline adducts of 2-substituted-1-phenyl-1,3-butanedionato nickel(II) complexes relative to $\text{Ni}(\text{bzac})_2\text{phen}$ were also observed. Strong bands in the $773\text{--}774\text{ cm}^{-1}$ region were assigned to CH deformation bands of 2,2'-bipyridine while the 1,10-phenanthroline adducts had very prominent bands around $714\text{--}732\text{ cm}^{-1}$ and $849\text{--}856\text{ cm}^{-1}$ region which were attributed to CH deformation bands. The coupled vibrations of Ni-O and Ni-N stretching vibrational modes appeared below 700 cm^{-1} in all the adducts.

The solution spectra of the Me-bzacH in methanol showed a splitting of the $\pi_3\text{--}\pi_4^*$ transition whereas bzacH, Et-bzacH, n-Pr-bzacH, i-Pr-bzacH, n-Bu-bzacH had just a single band. The electronic spectra of 2-substituted-1-phenyl-1,3-butanedione in chloroform showed $\pi_3\text{--}\pi_4^*$ transition in the $32,258\text{--}35,336\text{ cm}^{-1}$ region which had hypsochromic shift in methanol except Me-bzacH and i-Pr-bzacH with bathochromic shift while bzacH had no shift.

The phenyl-substituted ligands had bands in the $38,000\text{--}46,000\text{ cm}^{-1}$ region, which may be attributed to primary bands in the benzene π system (Ogden and Selbin, 1968). The synthesized ligands showed similar bands in the region $39,526\text{--}44,248\text{ cm}^{-1}$ which have been assigned to $\pi\text{--}\pi^*$ transition of the phenyl ring.

Hypsochromic shifts of the $\pi_3\text{--}\pi_4^*$ transition bands were 1,10-observed on substituting the 2-position of 1-phenyl-1,3-butanedione with methyl, ethyl, n-propyl and i-propyl in chloroform and methanol. Hypsochromic shifts of the $\pi_3\text{--}\pi_4^*$ transition bands could be attributed to the positive inductive effect of the alkyl groups leading to higher frequency shift (Graddon and Schulz, 1965).

The solution spectra in methanol showed that the $\pi_3\text{--}\pi_4^*$ transitions of the nickel(II) complexes are lowered from the ligand values in the order: $\text{Ni}(\text{Et-bzac})_2 \cdot 2\text{H}_2\text{O}(\Delta\nu = +2958) > \text{Ni}(\text{i-Pr-bzac})_2 \cdot 2\text{H}_2\text{O}(\Delta\nu = +2515) > \text{Ni}(\text{Me-bzac})_2 \cdot 2\text{H}_2\text{O}(\Delta\nu = +2501) > \text{Ni}(\text{n-Pr-bzac})_2 \cdot 2\text{H}_2\text{O}(\Delta\nu = +1331) > \text{Ni}(\text{bzac})_2 \cdot 2\text{H}_2\text{O}(\Delta\nu = +1202)$. The order shows that the substituted Ni(II) complexes have larger bathochromic shift than the unsubstituted complex, $\text{Ni}(\text{bzac})_2 \cdot 2\text{H}_2\text{O}$ except $\text{Ni}(\text{n-Bu-bzac})_2 \cdot 2\text{H}_2\text{O}$ which had $\pi_3\text{--}\pi_4^*$ hypsochromic shifts in chloroform and methanol.

Upon adduct formation $\pi_3\text{--}\pi_4^*$ hypsochromic shifts were observed in all the adducts in methanol except $\text{Ni}(\text{n-Pr-bzac})_2\text{bipy}$, $\text{Ni}(\text{n-Bu-bzac})_2\text{bipy}$ and $\text{Ni}(\text{n-Bu-bzac})_2\text{phen}$ which had bathochromic shifts. $\pi_3\text{--}\pi_4^*$

hypsochromic shifts were also observed in the adducts in chloroform except $\text{Ni}(\text{Et-bzac})_2\text{bipy}$, $\text{Ni}(\text{Et-bzac})_2\text{phen}$ and $\text{Ni}(\text{n-Bu-bzac})_2\text{bipy}$ which had bathochromic shifts and $\text{Ni}(\text{n-Me-bzac})_2\text{bipy}$ which had no shift. The hypsochromic shifts probably indicate weaker bonds in the chelate ring due to reduced electron delocalization as a result of stronger Ni-bipy or Ni-phen interaction. The ligand field spectra of the 2,2'-bipyridine and 1,10-Phenanthroline adducts of the nickel(II) complexes studied had bands in the $11,494\text{--}12,853\text{ cm}^{-1}$, $13,158\text{--}19,305\text{ cm}^{-1}$ and $20,080\text{--}20,161\text{ cm}^{-1}$ in which have been assigned to ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})$, ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$, ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$ respectively for octahedral geometry (Lever, 1986; Osowole et al, 2000).

The reflectance spectra of the ligands showed single bands of the $\pi_3\text{--}\pi_4^*$ transition in bzacH, n-Pr-bzacH, and n-Bu-bzacH while Me-bzacH and Et-bzacH had multiple bands, these occurred in the $31,056\text{--}35,587\text{ cm}^{-1}$ region. A split of $\pi_3\text{--}\pi_4^*$ band was also observed in $\text{Ni}(\text{Et-bzac})_2\cdot 2\text{H}_2\text{O}$ and $\text{Ni}(\text{n-Pr-bzac})_2\cdot 2\text{H}_2\text{O}$ while $\pi\text{-d/C.T.}$ transition was observed in $\text{Ni}(\text{Et-bzac})_2\cdot 2\text{H}_2\text{O}$ at $30,395\text{ cm}^{-1}$. $\pi_3\text{--}\pi_4^*$ bathochromic shifts were observed in the Nickel(II) complexes except $\text{Ni}(\text{bzac})_2\cdot 2\text{H}_2\text{O}$, $\text{Ni}(\text{Et-bzac})_2\cdot 2\text{H}_2\text{O}$ and $\text{Ni}(\text{n-Pr-bzac})_2\cdot 2\text{H}_2\text{O}$ which had hypsochromic shifts.

Upon adduct formation $\pi_3\text{--}\pi_4^*$ hypsochromic shifts were observed in $[\text{Ni}(\text{bzac})(\text{bipy})(\text{H}_2\text{O})_2](\text{bipy})$, $\text{Ni}(\text{bzac})_2\text{bipy}$, $\text{Ni}(\text{Me-bzac})_2\text{bipy}$, $\text{Ni}(\text{Et-bzac})_2\text{bipy}$, $\text{Ni}(\text{n-Pr-bzac})_2\text{phen}$, $\text{Ni}(\text{n-Bu-bzac})_2\text{bipy}$ and $\text{Ni}(\text{n-Bu-bzac})_2\text{phen}$. The hypsochromic shifts probably indicate weaker bonds in the chelate ring due to reduced electron delocalization as a result of stronger Ni-phen or Ni-bipy interaction. $[\text{Ni}(\text{phen})_2(\text{H}_2\text{O})_2](\text{bzac})_2$, $\text{Ni}(\text{bzac})_2\text{phen}$, $\text{Ni}(\text{Me-bzac})_2\text{phen}$, $\text{Ni}(\text{Et-bzac})_2\text{phen}$ and $\text{Ni}(\text{n-Pr-bzac})_2\text{bipy}$ had bathochromic shifts. The reflectance spectral data showed that the visible bands of the adducts were also typical of octahedral geometry (Lever, 1986; Osowole et al, 2000).

The room temperature magnetic moments of the synthesized nickel(II) complexes were in the range 3.02–3.26 B.M. which is indicative of octahedral geometries except $\text{Ni}(\text{n-Pr-bzac})_2\cdot 2\text{H}_2\text{O}$ with moment of 4.11 B.M. The spin only ($\mu_{s.o}$) value of 2.83 B.M is expected for nickel(II) complexes. Experimental moments of 2.9–3.3 B.M. are normally observed for octahedral nickel(II) complexes due to spin-orbit coupling of the ${}^3\text{A}_{2g}$ and ${}^3\text{T}_{2g}(\text{F})$ terms while moments of 3.2–4.1 B.M. are observed for tetrahedral nickel due to orbital contributions. In the synthesized complexes, lower moments were observed on substituting the 2-position of $\text{Ni}(\text{bzac})_2$ with alkyl groups except $\text{Ni}(\text{Et-bzac})_2\cdot 2\text{H}_2\text{O}$ and $\text{Ni}(\text{n-Pr-bzac})_2\cdot 2\text{H}_2\text{O}$ which had increased moments. Reduction in moment may probably be due to increase in electron density around the metal leading to reduction in orbital contribution and therefore lower moment. The adducts studied displayed effective magnetic moments (μ_{eff}) in the range 1.81–3.31 B.M. The magnetic moments between 1.81–2.65 B.M. which are lower than the spin only value were observed for $\text{Ni}(\text{Me-bzac})_2\text{phen}$ and $\text{Ni}(\text{n-Bu-bzac})_2\text{phen}$. The lowering of the moment observed in the compounds studied may be attributed to interconversion of stereochemistries and /or dimerization (Osowole et al, 2000). A decrease in moment was observed on comparing the 2,2'-bipyridine adduct of 2-substituted-1-phenyl-1,3-butanedionato nickel(II) complexes with $\text{Ni}(\text{bzac})_2\text{bipy}$. The 1,10-phenanthroline adduct of nickel(II) 2-substituted-1-phenyl-1,3-butanediones also exhibited decreased moment. Reduction in moment is attributed to increase electron density around the nickel leading to reduction in orbital contribution and therefore lower moments are obtained.

The molar conductivities of these complexes are very low with Λ_m values of $14.1\text{--}32.5\text{ ohm}^{-1}\text{cm}^2\text{mole}^{-1}$, which suggests that they are non-electrolytes. The molar conductances of the soluble adducts in nitromethane clearly indicate that they were non-electrolytes except $[\text{Ni}(\text{bzac})(\text{bipy})(\text{H}_2\text{O})_2](\text{bzac})$, $[\text{Ni}(\text{phen})_3](\text{bzac})_2$ and $[\text{Ni}(\text{phen})_2(\text{H}_2\text{O})_2](\text{bzac})_2$ which are electrolytes.

4. Conclusion

The electronic and infrared spectral data and magnetic measurement are consistent with the adoption of an octahedral geometry for the Nickel(II) compounds except $\text{Ni}(\text{n-Pr-bzac})_2\cdot 2\text{H}_2\text{O}$ which has tentative four coordinate tetrahedral geometry. The conductance measurement showed that the compounds are non-electrolytes except $[\text{Ni}(\text{bzac})(\text{bipy})(\text{H}_2\text{O})_2](\text{bzac})$ which is a 1:1 electrolyte, $[\text{Ni}(\text{phen})_3](\text{bzac})_2$ and $[\text{Ni}(\text{phen})_2(\text{H}_2\text{O})_2](\text{bzac})_2$ are 1:2 electrolytes.

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Table 1. Analytical and physical data of Nickel(II) complexes of 2-substituted-1-phenyl-1,3-butanedione and their adducts

Compound	Colour	M.P. (°C)	Yield (%)	μ_{eff} (B.M.)
$\text{Ni}(\text{bzac})_2 \cdot 2\text{H}_2\text{O}$	Light green	165-167	93.90	3.11
$[\text{Ni}(\text{bzac})(\text{bipy})(\text{H}_2\text{O})_2] (\text{bipy})$	Light green	210-212	38.76	3.04
$\text{Ni}(\text{bzac})_2\text{bipy}$	Green	215	6.79	3.29
$[\text{Ni}(\text{phen})_3](\text{bzac})_2$	Pink	106-108	28.94	3.44
$[\text{Ni}(\text{phen})_2(\text{H}_2\text{O})_2](\text{bzac})_2$	Pink	205	37.00	3.09
$\text{Ni}(\text{bzac})_2\text{phen}$	Y green	237-239	52.26	3.05
$\text{Ni}(\text{Me-bzac})_2 \cdot 2\text{H}_2\text{O}$	Light green	158-160	11.34	3.10
$\text{Ni}(\text{Me-bzac})_2\text{bipy}$	Light green	310-312	55.03	2.99
$\text{Ni}(\text{Me-bzac})_2\text{phen}$	D.Pink	323-325	30.04	2.65
$\text{Ni}(\text{Et-bzac})_2 \cdot 2\text{H}_2\text{O}$	Light green	179-181	54.29	3.26
$\text{Ni}(\text{Et-bzac})_2\text{bipy}$	Light green	296-298	30.33	3.11
$\text{Ni}(\text{Et-bzac})_2\text{phen}$	Light green	240-242	35.97	3.31
$\text{Ni}(\text{i-Pr-bzac})_2 \cdot 2\text{H}_2\text{O}$	Light green	222-224	91.96	3.02
$\text{Ni}(\text{i-Pr-bzac})_2\text{bipy}$	D.green	146-148	46.54	3.03
$\text{Ni}(\text{i-Pr-bzac})_2\text{phen}$	Dark green	168-170	42.50	3.04
$\text{Ni}(\text{n-Pr-bzac})_2 \cdot 2\text{H}_2\text{O}$	Light green	304	50.56	4.11
$\text{Ni}(\text{n-Pr-bzac})_2\text{bipy}$	Light green	301-303	42.50	3.15
$\text{Ni}(\text{n-Pr-bzac})_2\text{phen}$	Dark green	336-338	46.90	3.28
$\text{Ni}(\text{n-Bu-bzac})_2 \cdot 2\text{H}_2\text{O}$	Bright green	155-157	59.40	3.23
$\text{Ni}(\text{n-Bu-bzac})_2\text{bipy}$	Dark green	279-281	25.40	3.07
$\text{Ni}(\text{n-Bu-bzac})_2\text{phen}$	Dark green	255-257	13.69	1.81

D=Dirty, Y=yellowish %=percentage

Table 2. Microanalytical data of nickel(II) complexes of 2-substituted-1-phenyl-1,3-butanedione and their adducts

Compound	M.Wt	% Calculated (Observed)			
		C	H	N	Ni
Ni(bzac) ₂ ·2H ₂ O	417.13	57.58 (57.33)	5.33 (5.13)	- -	14.07 (13.79)
[Ni(bzac)(bipy)(H ₂ O) ₂] (bipy)	573.30	62.85 (62.94)	5.29 (4.99)	4.88 (5.10)	10.24 (10.00)
Ni(bzac) ₂ bipy	537.27	67.06 (66.91)	4.89 (5.14)	5.21 (5.08)	10.93 (10.34)
[Ni(phen) ₃](bzac) ₂	957.73	70.22 (70.16)	4.85 (4.62)	8.77 (8.62)	6.13 (5.56)
[Ni(phen) ₂ (H ₂ O) ₂](bzac) ₂	777.53	67.96 (67.74)	4.94 (4.82)	7.20 (7.16)	7.55 (7.98)
Ni(bzac) ₂ phen	561.29	68.47 (68.25)	4.68 (4.56)	4.99 (5.02)	10.46 (10.39)
Ni(Me-bzac) ₂ ·2H ₂ O	445.19	59.35 (59.33)	5.90 (5.82)	- -	13.19 (13.02)
Ni(Me-bzac) ₂ bipy	565.33	67.98 (68.05)	5.36 (5.07)	4.95 (5.21)	10.39 (10.60)
Ni(Me-bzac) ₂ phen	589.35	69.29 (69.01)	5.14 (4.89)	4.75 (4.53)	9.96 (10.18)
Ni(Et-bzac) ₂ ·2H ₂ O	473.25	60.91 (60.69)	6.40 (6.13)	- -	12.41 (12.30)
Ni(Et-bzac) ₂ bipy	593.39	68.81 (68.58)	5.78 (5.57)	4.72 (4.42)	9.89 (10.10)
Ni(Et-bzac) ₂ phen	617.41	70.03 (69.78)	5.56 (5.34)	4.54 (4.31)	9.51 (9.79)
Ni(i-Pr-bzac) ₂ ·2H ₂ O	501.39	62.29 (62.59)	6.85 (7.11)	- -	11.71 (11.73)
Ni(i-Pr-bzac) ₂ bipy	621.45	69.57 (69.68)	6.18 (6.30)	4.51 (4.66)	9.45 (9.69)
Ni(i-Pr-bzac) ₂ phen	645.47	70.71 (70.96)	5.95 (6.16)	4.34 (4.46)	9.10 (8.82)
Ni(n-Pr-bzac) ₂ ·2H ₂ O	501.39	62.29 (62.59)	6.85 (7.11)	- -	11.71 (11.73)
Ni(n-Pr-bzac) ₂ bipy	621.45	69.57 (69.68)	6.18 (6.30)	4.51 (4.66)	9.45 (9.69)
Ni(n-Pr-bzac) ₂ phen	645.47	70.71 (70.96)	5.95 (6.16)	4.34 (4.46)	9.10 (8.82)
Ni(n-Bu-bzac) ₂ ·2H ₂ O	529.37	63.52 (63.39)	7.25 (7.08)	- -	11.09 (11.32)
Ni(n-Bu-bzac) ₂ bipy	649.51	70.27 (70.01)	6.53 (6.38)	4.31 (4.59)	9.04 (8.80)
Ni(n-Bu-bzac) ₂ phen	673.53	71.33 (71.03)	6.30 (6.07)	4.16 (3.95)	8.72 (8.94)

Table 3. Relevant Infrared Spectra bands (cm^{-1}) of Nickel(II) complexes of 2-substituted-1-phenyl-1,3-butanedione and their adducts

Formula	C=O, C=C	$\nu_s(\text{C-O})+\delta\text{C-H}$	$\delta_{as}(\text{CH}_3)+\delta_s(\text{CH}_3)$	$\gamma(\text{C-H})\text{Phen/bipy}$
bzacH	1599m, 1540b	1484m	1413m, 1360m	-
Ni(bzac) ₂ .2H ₂ O	1595s, 1560m	1487m, 1452m	1409b	-
[Ni(bzac)(bipy)(H ₂ O) ₂](bzac)	1595s, 1569s	1456m	1414b	773s
Ni(bzac) ₂ bipy	1596s, 1570s	1456s	1415s	773w
[Ni(phen) ₃](bzac) ₂	1652w, 1626w	1482vw	1426m, 1399m	849s, 726s
[Ni(phen) ₂ (H ₂ O) ₂](bzac) ₂	1594s, 1565s	1458m	1400vs	852s, 727s
Ni(bzac) ₂ phen	1711s, 1592w	1486vw, 1460w	1406b	849s, 728m
Me-bzacH	1729m, 1666m	1450s	1408vw, 1372s	-
Ni(Me-bzac) ₂ .2H ₂ O	1584m, 1578s	1472s	1407s, 1354s	-
Ni(Me-bzac) ₂ bipy	1556s	1490vw, 1472vw	1417m, 1342vw	774s
Ni(Me-bzac) ₂ phen	1624w, 1594s	1485w, 1456m	1423vw, 1400vs	850s, 727vs
Et-bzacH	1723m, 1677m	1449m	1420w, 1359m	-
Ni(Et-bzac) ₂ .2H ₂ O	1598s, 1560s	1489m, 1454s	1405s	-
Ni(Et-bzac) ₂ bipy	1557vs	1490vs, 1472vw	1417s	774vs
Ni(Et-bzac) ₂ phen	1598s, 1570s	1456s	1408s	852m, 714m
i-Pr-bzacH	1764w, 1687b	1490vw, 1448vs	1411w, 1373m	-
Ni(i-Pr-bzac) ₂ .2H ₂ O	1654w, 1584m	1472vs	1407vs, 1354vs	-
Ni(i-Pr-bzac) ₂ bipy	1663s, 1600vs	1458w, 1444w	1400s	770vs
Ni(i-Pr-bzac) ₂ phen	1665b, 1628b	1459vw	1424s	851vs, 724vs
n-Pr-bzacH	1720m, 1676m	1458m	1421w, 1384m	-
Ni(n-Pr-bzac) ₂ .2H ₂ O	1569b	1488w	1415b, 1342w	-
Ni(n-Pr-bzac) ₂ bipy	1556b	1490w, 1472w	1419s, 1342m	774s
Ni(n-Pr-bzac) ₂ phen	1570b, 1517w	1498vw	1413s, 1344m	856s, 732s
n-Bu-bzacH	1718w, 1685w	1459w	1421w	-
Ni(n-Bu-bzac) ₂ .2H ₂ O	1726w, 1676w	1492m, 1454m	1410b	-
Ni(n-Bu-bzac) ₂ bipy	1591vw, 1557s	1485w, 1452vw	1416s, 1342w	774s
Ni(n-Bu-bzac) ₂ phen	1593vs, 1568v	1486m, 1459s	1410vs	849s, 728s

Table 4. The electronic solution spectra of Nickel(II) complexes of 2-substituted-1-phenyl-1,3-butanedione and their adducts

Empirical Formula	$\pi_{3, \pi_4}^*(\text{cm}^{-1})$		d-d	
	CHCl_3	CH_3OH	CHCl_3	CH_3OH
bzacH	32,258(7247)	32,258(18259)	-	-
Ni(bzac) ₂ .2H ₂ O	31,646(61419)	31,056(31780)	15,480(37)	19,305(62) 14,286(45)
[Ni(bzac)(bipy)(H ₂ O) ₂](bzac)	33,113(40581)	33,113(27405)	17,182(25) 12,843(5)	17,668(21) 12,853(4) 11,521(7)
Ni(bzac) ₂ bipy	33,3113(29467)	32,895(41812)	17,212(11)	17,361(83) 12,821(47) 11,765(54)
[Ni(phen) ₃](bzac) ₂	34,247(?)	34,014(43955)	20,080(?) 12,500(?)	15,748(71) 13,158(69) 11,765(69)
[Ni(phen) ₂ (H ₂ O) ₂](bzac) ₂	34,247(25953)	34,247(34536)	17,730(18) 12,821(7)	17,483(31) 12,579(13) 11,765(34)
Ni(bzac) ₂ phen	34,014(?)	34,247(22)752	17,241(26) 12,987(7)	17,241(?) 12,626(?) 11,494(?)
Me-bzacH 31,056(?)	34,364(?)	33,557(?)	-	-
Ni(Me-bzac) ₂ .2H ₂ O	-	31,056(29975)	-	20,161(64) 15,853(50)
Ni(Me-bzac) ₂ bipy	33,557(36196)	33,333(21213)	15,974(31)	16,611(6)
Ni(Me-bzac) ₂ phen	34,602*	34,483*	18,149(4)	18,149(22)
Et-bzacH	32,680(?)	34,014(?)	-	-
Ni(Et-bzac) ₂ .2H ₂ O	34,602*	31,056(339)	15,924(20)	15,674(1)
Ni(Et-bzac) ₂ bipy	33,557(3638)	33,333(20555)	15,934(23)	16,474(1)
Ni(Et-bzac) ₂ phen	34,483*	34,483(?)	15,974(23)	16,949(?)
i-Pr-bzacH	35,336(?)	35,088(?)	-	-
Ni(i-Pr-bzac) ₂ .2H ₂ O	-	32,573(?)	-	14,430(?)
Ni(i-Pr-bzac) ₂ bipy	32,895(?)	34,602(?)	15,823(?)	16,340(?) 13,369(?)
Ni(i-Pr-bzac) ₂ phen	-	33,333*	16,779(?)	15,528(?) 13,369(?)
n-Pr-bzacH	35,088(?)	35,461(?)	-	-
Ni(n-Pr-bzac) ₂ .2H ₂ O	-	34,130(?)	-	14,903(?)
Ni(n-Pr-bzac) ₂ bipy	33,557(25232)	33,223(?)	15,949(20)	16,393(?)
Ni(n-Pr-bzac) ₂ phen	34,247(?)	34,364(?)	15,974(?)	15,480(?)
n-Bu-bzacH	33,003(?)	33,784(?)	-	-
Ni(n-Bu-bzac) ₂ .2H ₂ O	34,602(?)	34,722*	15,674(?)	15,848(?)
Ni(n-Bu-bzac) ₂ bipy	33,557(65368)	33,557(22886)	16,207(27)	17,212(6)
Ni(n-Bu-bzac) ₂ phen	34,965*	34,483(?)	16,892(33)	16,722(?) 13,423(8)

Table 5. The electronic solid reflectance spectra of Nickel(II) complexes of 2-substituted-1-phenyl-1,3-butanedione and their adducts

Emperical Formula	$\pi_3, \pi_4^*(\text{cm}^{-1})$	d-d
bzacH	31,056	-
Ni(bzac) ₂ .2H ₂ O	35,211	21,053, 19,417, 11,962
[Ni(bzac)(bipy)(H ₂ O) ₂](bzac)	35,842, 31,250	19,881, 13,605, 12,594
Ni(bzac) ₂ bipy	35,971, 31,153	21,598, 13,680, 12,330
[Ni(phen) ₂ (H ₂ O) ₂](bzac) ₂	31,153	20,877, 14,493, 12,771
Ni(bzac) ₂ phen	32,573	19,493, 13,532, 12,788
Me-bzacH	35,587, 32,680	-
Ni(Me-bzac) ₂ .2H ₂ O	34,965	21,739, 19,608, 11,990
Ni(Me-bzac) ₂ bipy	35,842, 31,056	19,920, 13,514, 12,484
Ni(Me-bzac) ₂ phen	32,258	20,576, 14,771, 12,755
Et-bzacH	35,088, 31,153	-
Ni(Et-bzac) ₂ .2H ₂ O	35,211, 31,153	19,569, 13,333, 12,034
Ni(Et-bzac) ₂ bipy	35,587, 31,055	19,763, 13,532, 12,674
Ni(Et-bzac) ₂ phen	33,223	19,531, 13,495, 12,658
n-Pr-bzacH	33,003	-
Ni(n-Pr-bzac) ₂ .2H ₂ O	33,898, 31,347	19,380, 14,205, 11,696
Ni(n-Pr-bzac) ₂ bipy	31,250	19,841, 13,587, 12,330
Ni(n-Pr-bzac) ₂ phen	34,130	19,608, 13,495, 12,315
n-Bu-bzacH	33,670	-
Ni(n-Bu-bzac) ₂ .2H ₂ O	31,153	21,231, 19,048, 12,121
Ni(n-Bu-bzac) ₂ bipy	31,250	19,881, 13,569
Ni(n-Bu-bzac) ₂ phen	33,113	19,960, 19,011

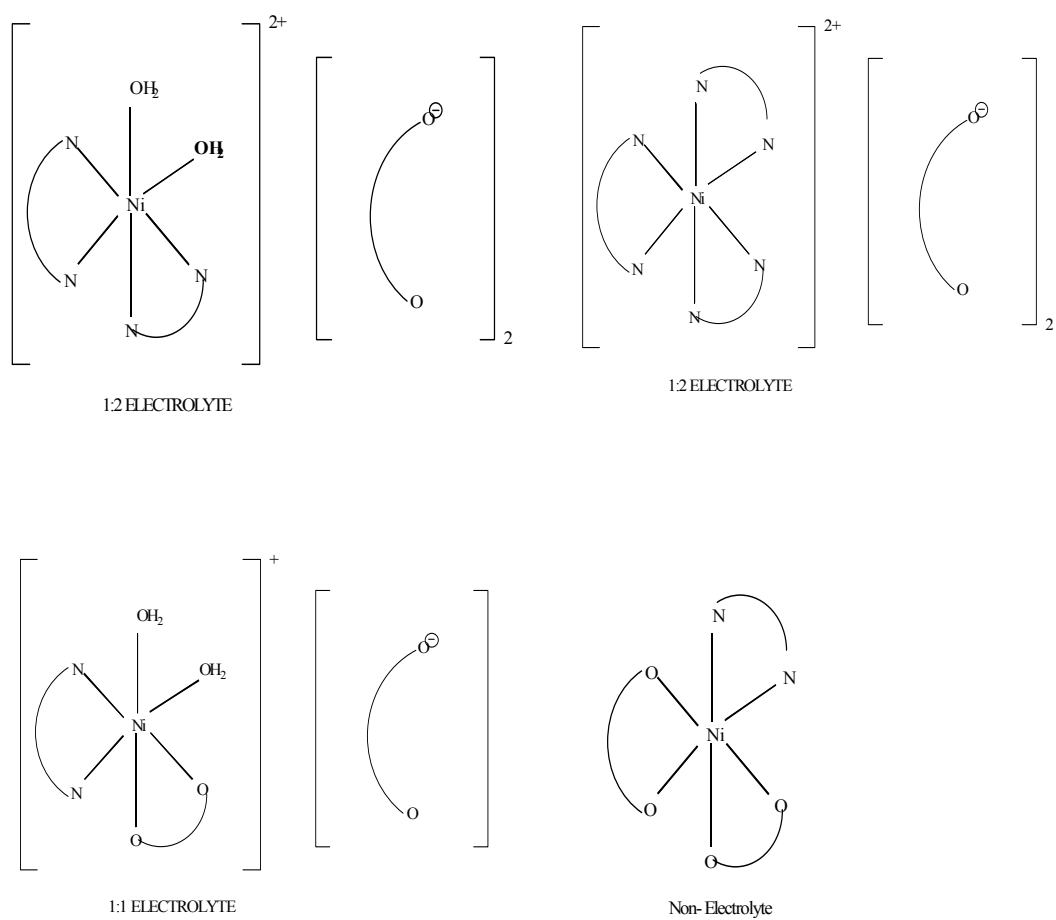


Figure 1. Proposed structures for Nickel adducts