

2016

## A Guided Inquiry-Based Laboratory Experiment: Synthesis and Characterization of Various Phthiocol Complexes

Turina Jangula  
*South Dakota State University*

Follow this and additional works at: <http://openprairie.sdstate.edu/jur>

 Part of the [Educational Methods Commons](#), [Higher Education Commons](#), [Inorganic Chemistry Commons](#), and the [Science and Mathematics Education Commons](#)

---

### Recommended Citation

Jangula, Turina (2016) "A Guided Inquiry-Based Laboratory Experiment: Synthesis and Characterization of Various Phthiocol Complexes," *The Journal of Undergraduate Research*: Vol. 14 , Article 9.  
Available at: <http://openprairie.sdstate.edu/jur/vol14/iss1/9>

This Article is brought to you for free and open access by the Division of Research and Economic Development at Open PRAIRIE: Open Public Research Access Institutional Repository and Information Exchange. It has been accepted for inclusion in The Journal of Undergraduate Research by an authorized editor of Open PRAIRIE: Open Public Research Access Institutional Repository and Information Exchange. For more information, please contact [michael.biondo@sdstate.edu](mailto:michael.biondo@sdstate.edu).

---

# A Guided Inquiry-Based Laboratory Experiment: Synthesis and Characterization of Various Phthiocol Complexes

## **Cover Page Footnote**

I would like to thank the Department of Chemistry & Biochemistry for the Undergraduate Research opportunity. I would like to also thank the East Family Summer Research Fellowship for sponsoring me for my summer research.

# **A guided inquiry-based laboratory experiment: Synthesis and characterization of various phthiocol complexes**

Author: Turina Jangula

Faculty Sponsor: Dr. Tanya Gupta

Department: Chemistry & Biochemistry

## **ABSTRACT**

Despite proven effectiveness of constructivist guided-inquiry based instruction for college chemistry courses, teaching of advanced chemistry courses such as inorganic chemistry remains very much traditional. This persistence of traditional instructional approaches could be attributed to the lack of inquiry-based curriculum materials for these courses. In this paper a guided-inquiry based inorganic laboratory experiment on the synthesis and characterization of phthiocol complexes with four different metals is presented along with suggestions for implementation as a laboratory activity. The experiment is unique in terms of its development based on a social constructivist framework and has student discussion and conceptual understanding of inorganic chemistry at its core.

Keywords: guided-inquiry, phthiocol complexes, laboratory activity

## **INTRODUCTION**

Coordination chemistry is the study of compounds formed between metal ions that act as electron-acceptors (Lewis base), and other neutral or negatively charged molecules, which act as electron-donors or ligands (Lewis Bases). Coordination metal complexes are popular because of their multipurpose applications that extend to biomolecules chelating agents as therapeutics, coloring agents and analytical reagents (Strohfeltd, 2015). The metal

## A GUIDED-INQUIRY BASED EXPERIMENT

complexes are formed as a result of interaction of the positively charged metal center with ligands that surround it, which in turns determines the properties of these complexes. Chemists are seeking ways to develop highly efficient and selective catalysts in order to meet the ever-increasing demands for affordable drugs, foods or materials for a sustainable society. While new catalysts are being developed some existing compounds that act as ligands have also gained attention of chemists for their potential as homogenous catalysts.

Conventionally, chemists have relied on various properties of ligands such as steric hindrance and electronic properties to alter its catalytic behavior. However ligands in traditional sense have often been used as spectator species and metal centers have been an active site for the reactions to occur (Housecroft & Sharpe, 2012). Recently, chemists have approached this ligand behavior innovatively by using more chemically active ligands that can play a significant role in early or elementary bond activation stages in the catalytic cycle and thus have both the metal and ligand in a complex work synergistically to enhance a chemical reaction as homogenous catalysts (Volodymyr & Bruin, 2012)

Redox active ligands belong to this class of homogenous catalysts that are being tailored to meet the specific needs of various industries. A redox active or a non-innocent ligand participates in the catalytic cycle either by only accepting or donating its electrons or by actively participating in the bond formation or bond breaking of substrate covalent bonds (De Bruin, Bill, Bothe, Weyhermüller, & Wieghardt, 2000)

The starting material 2-methyl-1,4-naphthoquinone (menadoine) used in this experiment, is one such redox active ligand. The coordination chemistry of this ligand is comparatively less explored as compared to other redox-active species. However, these ligands have found applications as biologically active agents (antifungal, antiviral and antimalarial agents). Phthiocol (2 hydroxy-3-methyl-1,4-naphthoquinone) also known as the hydroxy analog of vitamin K<sub>3</sub> was synthesized from menadoine for this experiment (Gaikwad, Kankariya & Shah, 2014). This analog can act as a monodentate or bidentate ligand for metal complexes possibly forming redox-active materials. Hence it is important to develop laboratory activity on redox-active complexes for an undergraduate laboratory course. While it may not be possible for students in an undergraduate laboratory course to explore all aspects of an inorganic concept such as redox-active ligands during a three-hour laboratory periods for a given week of semester, it is still possible for students to engage in

## A GUIDED-INQUIRY BASED EXPERIMENT

synthesis and characterization of these metal complexes to understand the richness of redox-active species.

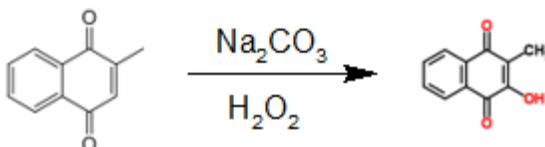
The next section describes experimental procedure for the synthesis and of Phthiocol and its metal complexes followed by Results, Discussions & conclusions based on melting points, Infrared (IR) and Nuclear Magnetic Resonance ( $^1\text{H-NMR}$ ) characterizations of these complexes.

It is important to note that for the development of this laboratory activity comparative published literatures of the synthesis of Phthiocol with metal complexes were first reviewed (see references) in order to develop an optimized guided-inquiry based procedure. The goal was to improve experimental yield, establish purity by melting point, and use of analytical characterization methods such as IR and  $\text{H}^1$  NMR and to establish these procedures for successfully implementing it as a laboratory experiment to be completed in a three-hour timeframe (one laboratory meeting). Hence several experiments were performed until we were satisfied with the methods of synthesis and convinced that this could be implemented in an undergraduate laboratory course.

Different metals for the synthesis of coordination complexes were used anticipating that synthesizing a range of metal complexes with ligand would promote discussion of structure and properties of coordination compounds among students in addition to testing different routes of synthesis for the ligand by adjusting quantities of starting materials.

Figure 1 illustrates a general method for the synthesis of Phthiocol (ligand).

**Figure 1:** General scheme for synthesis of Phthiocol



## METHODS

The following synthesis was slightly modified from original reported (Gaikwad, Kankariya & Shah, 2014; Kathwate et. al., 2013 & 2015).

**Table 1:** Materials and Instruments used (all chemicals used were AR grade)

Materials	Equipment & Instruments
2-methyl-1,4-naphthalenedione	pH-meter
Anhydrous Sodium Carbonate	Hot plate
Anhydrous methanol	Ice-bath
Concentrated H <sub>2</sub> SO <sub>4</sub>	Melt-temp
Ammonium thiosulfate	IR spectrometer
Cadmium (II) acetate	<sup>1</sup> H-NMR spectrometer (60 MHz)
Zinc (II) acetate	Hot plate
Sodium hydroxide	Conical flask (100 mL)
Potassium hydroxide	Beaker 100 mL, 250 mL
Aqueous Ammonia	Glass rod
30% H <sub>2</sub> O <sub>2</sub>	Magnetic stirrer and stir bard
Ethyl acetate	Glass rod

### Synthesis of Phthiocol (2-hydroxy-3-methyl-1,4-naphthaquinone)

The starting material, 1.0 g (5.8 mmols) of 2-methyl-1, 4-naphthalenedione, was dissolved in 10 mL of hot ethanol solution and placed on an ice-bath to chill. A peroxide solution was made using 0.2 g of anhydrous sodium carbonate in 1 mL of 30% H<sub>2</sub>O<sub>2</sub> and 5 mL of deionized water. The peroxide solution was added to the 2-methyl-1,4-naphthalenedione solution and the reaction mixture was maintained at 0°C. Next about 50 mL of cold deionized water was added that led to the formation of colorless to white crystals of 2-methyl, 1, 4-naphthaquinone oxide. The crystals were collected by vacuum filtration, rinsed with cold water and dried. The dried solid was transferred to a beaker and treated with 5 mL of concentrated H<sub>2</sub>SO<sub>4</sub> and allowed to stand for 10-15 minutes. This solution turned to a dark red color. Next on adding about 20 mL of cold water to the reaction mixture, yellow colored crystals precipitated. The precipitate was filtered using vacuum filtration. The crude ligand was then recrystallized with methanol.

## A GUIDED-INQUIRY BASED EXPERIMENT

### Recrystallization of phthiocol

The crude product was dissolved in hot methanol in a beaker. Minimum amount of solvent was to ensure better product yield. The solution was then gradually heated. Once the solution appeared clear, it was slowly cooled to room temperature. Fine yellow crystals of phthiocol were formed and were collected using vacuum filtration followed by rinsing with cold methanol (Figure 2). This purified ligand was used for synthesizing metal complexes with various salts of sodium, potassium, zinc and cadmium as described in the next section. The final product collected from recrystallization was analyzed using FTIR,  $H^1$  NMR, and melting point analysis.

**Table 2:** Purified Phthiocol ligand



### Synthesis of Metal Complexes

#### Zinc Complex

About 2 mmol (~0.38 grams) of the ligand (was dissolved in 25 mL of anhydrous methanol. A solution of 1.0 mmol (~0.22 grams) of zinc acetate was prepared separately in 10 mL of anhydrous methanol. The dissolved zinc solution was added dropwise to the ligand solution over a period of 30 minutes while stirring continuously using magnetic stirrer. A deep red colored complex was formed. The product was collected using vacuum filtration. It was washed with methanol followed by diethyl ether and dried.

## A GUIDED-INQUIRY BASED EXPERIMENT

### **Cadmium Complex**

About 2.0 mmol (~0.38 grams) of the ligand was dissolved in approximately 25 mL of methanol. Next 1.0 mmol of the aqueous solution Cadmium (II) acetate was added. The solution's pH was adjusted to the range 5.0-5.5 using an aqueous ammonia solution (1:20 v/v). This solution was refluxed in an oil bath for approximately 90 minutes. The reddish violet colored product was cooled to room temperature, and finally washed thoroughly with cold water, vacuum filtered and dried in a desiccator.

### **Potassium Complex**

For synthesis of potassium complex about 1 mmol of ligand, it was dissolved in 10 mL of anhydrous methanol. A separate solution was made using 1 mmol of KOH in 10 mL of anhydrous methanol. The dissolved ligand solution and the KOH solution were mixed together and stirred vigorously for 30 minutes. The pH was tested and adjusted to be close to the value of 7. A reddish orange colored product appeared which was filtered vacuum filtration and washed with diethyl ether and air-dried.

### **Sodium complex**

About 1 mmol, of Phthiocol ligand was dissolved in 10 mL of anhydrous methanol. A separate solution was made using 1 mmol of NaOH and 10 mL of anhydrous methanol. The ligand solution and NaOH solutions were mixed together and vigorously stirred for 30 minutes. The pH was adjusted in order to have a value of 7. The reddish-orange product was collected using vacuum filtration and finally rinsed with diethyl ether and air-dried.

## **Characterization of phthiocol ligand and complexes using melting point, IR, NMR**

**Melting point determination:** melting point is the temperature at which the solid and liquid phases of pure substances coexist. Melting points of a solid helps to identify compounds and also establishes their purity, which is indicated by a high and sharp melting point (narrow range of melting temperature). For determining melting point of ligands and complexes Melt-Temp was used. A small sample of ligands and metal complexes was transferred to a capillary tube and placed in Melt-Temp. The samples were heated slowly

## A GUIDED-INQUIRY BASED EXPERIMENT

and temperature was noted at the onset of melting till the entire solid sample melted to a clear liquid. A sharp temperature range was indicative of the purity of the sample.

**Infrared spectroscopy (IR)** was used to characterize ligands and the metal complexes (usually from  $400\text{ cm}^{-1}$ -  $4000\text{ cm}^{-1}$ ). In order to obtain IR spectra for metal and complexes a small amount of sample was dissolved in dichloromethane and IR was collected using a Fischer Scientific model with peak values in  $\text{cm}^{-1}$ . The characterization was done based the structure of starting materials as reported in the literature (Gaikwad, Kankariya, & Shah; 2014)

IR study is useful to gain information about vibrational motions of molecules. When molecules absorb energy, specific bonds in the molecule become excited and they vibrate. When the frequency of incident radiation equals the vibrational frequency of a bond, it is excited to a higher vibrational state and the amplitude of vibration also increases. Structurally, different molecules absorb different amounts of energy hence different patterns of IR absorption are obtained as each functional group of a compound has a specific absorption wavelength. Due to these structural differences, motions among different group of molecules on a ligand or a metal complex also differ causing these functional groups to, absorb different amounts of energy. Thus the signals in IR spectrum represent characteristics or specific functional groups.

IR serves two purposes - first to identify the presence or absence of function groups in a molecule and secondly IR is used as a fingerprint for molecule identification. In order to analyze the IR spectrum it is divided into two parts. The signals in the  $4000\text{-}1500\text{ cm}^{-1}$  provide information about the functional groups, whereas the range from  $1500\text{-}400\text{ cm}^{-1}$  is called the fingerprint region because the peaks observed in this region are specific to a compound just like fingerprints of humans (positive identification if fingerprint region is a match).

**Nuclear Magnetic Resonance Spectroscopy:** is another widely used technique for characterization of ligands and metal complexes due to its ability to reveal specific positions of atoms and their connectivity by mainly looking at the nuclear spin. NMR is used to determine the exact location functional groups and protons in a compound and it provides information about not just the carbon skeleton but also the complete molecule.

## A GUIDED-INQUIRY BASED EXPERIMENT

When a compound containing a positive charged nucleus such as  $^1\text{H}$  is placed in a magnetic field, the spin of the nucleus aligns with that of the applied magnetic spin (nuclear spin is  $+\frac{1}{2}$  in this situation). Next when radio frequency is applied it causes that nucleus to move or flip to a higher energy state, which is against the magnetic field (changes nuclear spin to  $-\frac{1}{2}$ ). Due to this small difference in energy (equal to that of low energy radio frequency) the protons undergo resonance. There is a decline or lag of signal observed for a time period while the proton spins back and aligns again with the magnetic field. The raw signal or oscillation curve is transformed to NMR spectrum using Fourier Transform mathematical function (Padias, 2015).

For  $^1\text{H}$ -NMR the hydrogen nucleus is used and hence it is also called proton-NMR and such as spectrum uses a scale of 1-10. The position of NMR peaks is expressed as  $\delta$ , which equals the signal frequency in Hertz divided by the frequency of NMR instrument (usually in Mega Hertz) and multiplied by  $10^6$ . The NMR spectrum has an x-axis usually expressed in parts per million (ppm) and the NMR peaks expressed as  $\delta$ , which is determine by the environment of the proton (and to determine the structure of molecule. Usually protons that are in same chemical environment are considered equivalent and give one signal whereas non-equivalent protons produce different signals. The area under the peaks in a NMR (also called integration) is directly proportional to the number of proton peaks or the relative number of protons of each type in a molecule. When there is a splitting or multiplicity of peaks, it is often determined by the number protons on the neighboring carbon plus one ( $n+1$ ). In general related multiplets in a  $^1\text{H}$ -NMR have same coupling constant ( $J$ ) which is the distance between the peaks.

For conducting  $^1\text{H}$ -NMR measurements, the ligand and the metal complexes were dissolved in DMSO and chemical shifts ( $\delta$ ) were recorded for each.

## Results

Table 2, 3 & Table 4 provide physical properties of ligand and its metal complexes and the IR and NMR characterizations for ligand and each of the four metal complexes (Zn, Cd, K, Na)

## A GUIDED-INQUIRY BASED EXPERIMENT

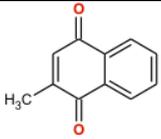
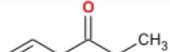
**Table 2:** Physical properties of starting material, intermediate, ligand and metal complexes

Sample	Appearance	Melting Point (°C)
2-methyl 1,4-naphthalenedioine (menadoine)	Yellow powder	103.7-105.5
2-methyl, 1, 4-naphthaquinone oxide (intermediate)	Colorless to white crystalline precipitate	95.1 - 96.2
2-hydroxy-3-methyl-1,4-naphthaquinone (Phthiocol)	Yellow crystals	172.4 - 174.2
Zn-Phthiocol complex	Dark red crystals	--
Cd-Phthiocol complex	Reddish violet solid	--
Na-Phthiocol complex	Reddish orange solid	--
K-Phthiocol complex	Reddish-orange solid	--

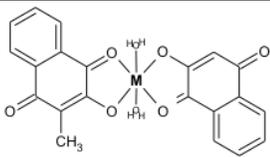
**Table 3:** IR spectral characterization for starting material, ligand and metal complexes

Sample	IR Characterization					
	Ketone (cm <sup>-1</sup> )	Aromatic C=C (cm <sup>-1</sup> )	Aromatic C-H (cm <sup>-1</sup> )	Methyl C-H (cm <sup>-1</sup> )	Alcohol (cm <sup>-1</sup> )	Metal
<b>2-methyl-1,4-naphthoquinone</b>	1664	1597, 1626	3055	1301, 1352, 1327	N/A	N/A
<b>Phthiocol</b>	1660	1590	3050	1342	3333	N/A
<b>Zn-Phthiocol</b>	1635	1436	2915.56, 3200	1314	3260	538
<b>Cd-Phthiocol</b>	1638	1525 1585	2914, 3150	1313	3424	704
<b>K-Phthiocol</b>	1655	1437	2913, 3001	1311	3460	700
<b>Na-Phthiocol</b>	1641	1523, 1586	2915, 2993	1315	3472	702

**Table 4:** NMR spectral characterization of ligand and metal complexes

Compound Name	Chemical shift (ppm)	Characterization	Structure
2-methyl-1,4-naphthoquinone (starting material)	2.190	singlet, 3H	
	6.831	doublet, 2H	
	7.615	triplet, 2H	
	8.042	triplet, 2H	
2-hydroxy-3-methyl-1,4-naphthaquinone or	2.101	singlet, 3H	
	8.173	doublet, 2H	

## A GUIDED-INQUIRY BASED EXPERIMENT

Phthiocol ligand	7.935	triplet, 2H	 <p style="text-align: center;">M=Zn, Cd</p>
	7.800	triplet, 2H	
	7.963	doublet, 2H	
	11.302	singlet, Ar-OH	
Zinc-Phthiocol complex	2.101	singlet, 3H	
	1.821	Singlet, 3H	
	7.493	Triplet, 2H	
	7.582	Triplet, 2H	
	7.620	Doublet, 2H	
	7.849	Triplet, 2H	
	Cadmium-Phthiocol complex	1.910	
8.151		Doublet, 2H	
7.686		Doublet, 2H	
7.725		Triplet, 2H	
7.462		Triplet, 2H	
Sodium-Phthiocol complex	1.998	Singlet, 3H	
	8.101	Doublet, 2H	
	7.834	Doublet, 2H	
	7.630	Doublet, 2H	
	7.598	Triplet, 2H	
	7.753	Triplet, 2H	
Potassium-Phthiocol complex	1.871	Singlet, 3H	
	7.880	Doublet, 2H	
	7.773	Doublet, 2H	
	7.599	Doublet, 2H	
	7.513	Triplet, 2H	
	7.725	Triplet, 2H	

## DISCUSSION AND CONCLUSIONS

As mentioned above, the preliminary structural characterization of phthiocol and its complexes was done using melting point IR, and  $H^1$  NMR. The expected melting point for pure Phthiocol is between 172-174 °C. The experimental melting point observed for the synthesis of Phthiocol was 172.4-174.2 °C (Table 1). The melting points of these products indicate that the ligand and corresponding complexes had minimal impurities. The experimental yield for the product was 0.5673 g and overall a 95.3%. In comparison, the published literatures reported 84%, 80%, and 76% yields respectively. These procedures dissolved their starting material in methanol rather than ethanol used in this experiment. The reported procedures also used column chromatography to separate their product

## A GUIDED-INQUIRY BASED EXPERIMENT

(Kathwate et. al., 2013). In this experiment, a column separation technique was not used. The purity of product as established by its melting point, IR and  $^1\text{H-NMR}$  and an improved yield of product ascertained that column chromatography (though a valuable technique) can be skipped all together and bring down the amount of time needed to complete this activity from three laboratory periods (nine hours) to within one laboratory meeting (less than three hours).

These analytical techniques IR and  $^1\text{H-NMR}$  spectroscopy were sufficient to characterize the ligand and the metal complexes and are believed to be important for students to gain a decent understanding of ligand and metal complex structures. These instruments are usually available for an undergraduate level laboratory courses in several institutions and hence lend to a reasonable inquiry for students to understand the synthesis and characterization aspects of inorganic coordination complexes.

The functional groups identified were alcohol, ketone, aromatic bonds, and methyl protons based on IR. The Phthiocol product formed showed an OH peak at  $3333\text{cm}^{-1}$  (Table 2). In the metal complexes synthesized, the broad band at  $\sim 32,60\text{ cm}^{-1}$  in Cd-complex and  $\sim 3424\text{ cm}^{-1}$  in Zn-complexes, and at  $3460$  and  $3472\text{ cm}^{-1}$  indicates the presence of coordinated water in the spectra. This broad peak obscures the  $\nu_{\text{C-H}}$  stretching observed for the aromatic protons for these complexes. The ketone peak ( $\nu_{\text{C=O}}$ ) was observed at  $1660\text{ cm}^{-1}$ . This functional group on the Phthiocol also remained throughout the syntheses of the metal complexes though it shifts to lower energy after complexion with metal (Table 2). Further the IR also showed that  $\nu_{\text{C=C}}$  vibrations also show a downward shift due to perhaps lowering of energy with formation of complexes. The appearance of peaks in the fingerprint region indicates the bonding of metal ion to the ligand.

The  $^1\text{H-NMR}$  of Phthiocol ligand showed three hydrogen's at peak  $2.101\text{ ppm}$  (Table 4). This indicates the presence of the methyl group on aromatic ring in phthiocol and similar environment for the three-methyl protons. This aromatic OH-bond is found at peak  $11.302\text{ ppm}$  as singlet. In the  $^1\text{H-NMR}$  of metal complexes the singlet  $-\text{OH}$  peak is not present. This also indicates the coordination of metal to the ligand. Further shift observed for ring protons in case of complexes as compared to the  $^1\text{H-NMR}$  of ligand provides more evidence of changes in the structure due to metal-ligand bond formation (Table 4).

## A GUIDED-INQUIRY BASED EXPERIMENT

The findings of IR and  $^1\text{H-NMR}$  of Phthiocol are consisted with those reported for similar complexes in literature. However, there was no report of phthiocol successfully bonding to sodium metal. In this synthesis, the formation of the metal complex of Na was successful as observed in IR (peak  $703\text{ cm}^{-1}$ ) and disappearance of O-H peak in Na-complex  $^1\text{H-NMR}$ . The bonding of phthiocol with a very soluble metal such as Na could have potential to be used as a redox-active catalyst similar to other complexes known for phthiocol.

The importance of optimizing procedure for this experiment through several trials indicates that this synthesis can be completed in a reasonable timeframe to further study the characteristics of phthiocol complexes. By using a different solvent for dissolving the starting material and excluding the column chromatography separation technique, this experiment was able to increase the overall yield and also complex with different metals.

## **SUGGESTIONS FOR IMPLEMENTATION FOR GUIDED-INQUIRY TEACHING**

This experiment will help instructors to engage students through guided-inquiry teaching in an undergraduate laboratory (Gunstone & Champagne, 1990; Gupta, Burke, Greenbowe & Mehta, 2015).

Instead of providing a complete procedure to students, the instructor should facilitate this experiment by providing students a list of materials and equipment and ask students to develop their own procedure. Instructor should only provide a framework for students such that they can generate a complete procedure with minimal guidance. For example, the instructor can ask students to work in groups and choose different quantities of ligand or vary the mass of each metal (limiting reactant problem). Students can also decide the techniques that they would like to use to characterize these complexes. This experiment can be successfully completed in single laboratory session can be used for significant class discussions on fundamental ideas of coordination complexes such as metal-ligand bonding especially redox active ligands, differences between transition and main-group elements, role of solvents in synthesis, and various techniques for characterization of such inorganic solids and their practical applications.

## LIMITATIONS

Overall the synthesis and characterization of phthiocol and its complexes with Zn, Cd, Na and K was successful. However, we could study the metal and ligand further for its structures and properties due to time constraints. It will be worthwhile to conduct  $^{13}\text{C}$ -NMR, Ultra-violet/ Visible spectroscopy and powder-x-ray diffraction for metal complexes to study their structure deeply and to ascertain some properties of these complexes.

## ACKNOWLEDGEMENTS

I would like to thank the Department of Chemistry & Biochemistry for the Undergraduate Research opportunity. I would like to also thank the East Family Summer Research Fellowship for sponsoring me for my summer research.

## REFERENCES

- De Bruin B. & Hindson, K. Special issue on Cooperative & Redox Non-innocent Ligands on Directing Organometallic chemistry, *European Journal of Inorganic Chemistry*, 2012, 3, 340-580.
- De Bruin, B., Bill, E., Bothe, E., Weyhermüller, T. & Wieghardt, K. (2000). Molecular and Electronic Structures of Bis(pyridine-2,6-diimine)metal Complexes  $[\text{ML}_2](\text{PF}_6)_n$  ( $n = 0, 1, 2, 3$ ;  $\text{M} = \text{Mn, Fe, Co, Ni, Cu, Zn}$ ). *Inorganic Chemistry*, 39 (13), 2936–2947
- Gaikwad, S. Kankariya, R., & Shah, D (2014). Ring Isomerism in Cadmium (II) Complexes of Vitamin K3 Analog with Methyl Derivative. *Journal of Applicable Chemistry*, 3 (1), 164-169.
- Gupta, T., Burke, K.A., Mehta, A., Greenbowe, T.J. Impact of Guided-Inquiry-Based Instruction with a Writing and Reflection Emphasis on Chemistry Students' Critical Thinking Abilities, *J. Chem. Educ.*, 2015, 92 (1), 32–38

## A GUIDED-INQUIRY BASED EXPERIMENT

- Gunstone, R.F., and Champagne, A.B. (1990). Promoting conceptual change in the laboratory. In E. Hegarty-Hazel (Ed.), *The student laboratory and the science curriculum* (pp. 159-182). London, England: Routledge.
- Kathawate, L. Gejj, S. P., Yeole, S. P., Verma, P. L., Puranik, V. G., & Gawali, S. S. (2015). The First Naphthosemiquinone Complex of  $K^+$  with Vitamin K3 Analog: Experiment and Density Functional Theory. *Journal of Molecular Structure* (2015): 56-63.
- Kathawate, Laxmi. Synthesis and Molecular Structure of a Zinc Complex of the Vitamin K3 Analogue Phthiocol. *Journal of Molecular Structure* (2013): 223-25.
- Kaim, W., Schwederski, B. (2010). Non-innocent ligands in bioinorganic chemistry—An overview. *Coordination Chemistry Reviews*. 254. (13-14) (13-14): 1580–1588.
- Houskroft, C & Sharpe, A. G. *Inorganic Chemistry*, 4<sup>th</sup> Edition, 2012, Chapter 20, page 665-715. Pearson: New York
- Luca, O. R. & Crabtree, R. H. (2012). "Redox-active Ligands in Catalysis". *Chemical Society Reviews* **42**: 1440–1459.
- Padias, A. B. (2015). *Making the connections: A how to guide to lab techniques*. Hayden McNeil: MI
- Strohfeltd, K. A. *Essentials of Inorganic Chemistry for students of pharmacy, pharmaceutical sciences, and medicinal chemistry*, Chapter 7, page 123-181, 2015, John-Wiley Ltd.: UK
- Volodymyr, L. & Bruin, De Bas (2012). Redox non-innocent ligands: Versatile new tools to control catalytic reactions, *ACS Catalysis*, 2, page 270-279.