SIC1002 Inorganic Chemistry I Practical Manual

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Laboratory Safety Rules

Safety is the primary concern in any chemical laboratory. Chemicals, particularly organic chemicals, are almost all potentially hazardous. Fortunately, with sensible and correct precautions, the risks can be minimized if certain basic safety practices are followed. The responsibility for laboratory safety lies with everyone working in the particular laboratory. Sensible laboratory conduct does not mean memorizing a list of rules! The true test is the actual conduct in the laboratory and safety rules apply to all laboratory activities. Each person's safety is affected by the action of fellow workers in the laboratory. Therefore, it is in everyone's best interest to follow safety work practices. The guidelines below are recommended for working safely in the laboratory.

- Know the location of all exits for the laboratory and the building.
- Know the location of the alarm and fire extinguishers and how to operate them.
- Know the location and use of safety showers, eye-washes and safety aid boxes.
- Know the location of the nearest telephone that can be used during an emergency.
- Never work alone in the laboratory. If you must work alone, make someone is aware of your location and let him or her call or check on you periodically.
- **Safety glasses or goggles must be worn at all times.**
- You might find them a nuisance to wear, but your eyes are very precious.
- If you wear contact lenses, try to avoid wearing them in the laboratory. If you must wear contact lenses, your goggles must seal particularly well to your face.
- Do not eat, drink or smoke in the laboratory.
- Wear protective clothing in the laboratory. Basically this includes laboratory coats, safety glasses, proper shoes and gloves (if necessary).
- Long hair should be tied back. Other articles of clothing that may become entangled should also be secured.
- Do NOT smell or taste chemicals. If your need to determine the odor of any chemical, waft it gently towards your nose with your hand – do not stick your nose in the container and inhale.
- Know the potential hazards of the materials and equipment with which you will work.
- Follow good housekeeping practices, that is, clean up as you go. Work areas must be kept clean. Do not clutter the work areas, aisles and exits.
- Store away apparatus that are not in immediate use, either in a cupboard or storeroom. Wash hands carefully before leaving the laboratory.
- **Do not wear laboratory coats, gloves or other personal protective clothing out of the laboratory and into non-laboratory areas**. This clothing may have become contaminated.
- **Report all accidents and injuries**, however small, immediately to the Lecturer-in charge or demonstrator or the laboratory assistants.
- In the interest of safety and security, work is permitted only during scheduled laboratory periods.
- Dispose of organic chemicals only in designated waste bottles. Chemical wastes are segregated into three groups and stored separately, viz, halogenated wastes (examples are chloroform, dichloromethane, carbon tetrachloride), non-halogenated wastes (examples are acetone, alcohol, toluene, xylene) and other wastes such as mercury and organometallics.

Additional Guidelines for Students

Remember that in a laboratory you have fellow students opposite you and by the side of you. They do not know what you are doing, but they hope and expect that what you are doing is sensible and safe. Always think carefully about what you are about to do.

- Know the Lecturer-in-charge and the laboratory workers of the laboratory.
- Undergraduates are not allowed to work or even be in any of the teaching laboratories at any time outside of the specified laboratory hours, unless they have explicit permission from the Lecturer-in-charge. This includes before and after class and during lunch hour.
- Come to laboratory periods on time and be prepared by studying the experiment and planning your activities before you come to the laboratory.

- Write everything you do and see in your notebook so that you can trace your actions and make corrections if necessary.
- Do not use cracked or broken glassware.
- Check glassware before using it.
- Never use open flames, unless instructed by the Lecturer-in-charge. If flames are permitted, plan your experiments so that you never leave your flame unattended. There are other sources of heat such as steam-baths and hot plates.
- **Handle all chemicals with care and read labels before attempting to get them**.
- Use a spatula to get solid chemicals. Never use your fingers.
- **Be careful not to contaminate reagents with your spatulas or droppers. If you take too much of a chemical or reagent, give it to a fellow student - do no return it to the bottle**.
- Do not wander off with the only bottle of a reagent that everyone needs; keep it in its assigned location. Do not pipette by mouth. Use only mechanical pipetting devices.
- Never look directly into the mouth of a flask containing a reaction mixture.
- Never point a test tube or reaction flask towards yourself or your neighbor.
- When using a separating funnel, vent frequently and remove the stopper immediately upon setting it upright for separation.
- **Never use a thermometer as a stirrer**! If a mercury thermometer breaks, immediately contact the Lecturer-in-charge or the demonstrator.
- Turn off water, burners or electrical equipment when not in use.
- Wash your glassware at the end of the laboratory day. You will have clean and dry glassware ready to go for the next laboratory class.
- Make sure glassware or equipment is kept in the correct locker - your personal locker or the common locker.
- Clean your work area and equipment used before leaving the laboratory.

Hereby, I, _____, with matric no. _____ have read, understood and will obey the safety rules in the laboratory.

Signed by,

.....

Name: _____

Date: _____

Witnessed by,

.....

Lecturer in-charge: _____ **(Official stamp)**

Date: _____

(Note to student: Print TWO copies of Pages i & ii, sign and return ONE copy to the Lecturer in-charge; and keep the other copy to yourself)

Lab Report Guidelines & Marking Scheme for Practical

Section 1 Lab Performance (Total 20%)

1. Pre-entering lab (5%)

Score	Criteria
0	No preparation of experimental procedure, no proper attire-shoes; goggle; lab coat.
1-2	Summary of procedures too brief, lack of details and confusing; incomplete safety attire.
3-5	Presents easy to follow steps in lab experimental, logical and adequately detailed; safety attire checked.

2. Skill & Techniques (15%)

Score	Criteria
0	No skill is demonstrated.
1-5	Wrong glassware used, wrong technique, spillage and wasting of chemicals.
6-10	Right glassware used, incorrect or lack of lab technique.
10-15	Presents correct lab skill, clean and tidy.

Section 2: Lab report (Total 60%)

Section	Total Mark	Rubric	
Title	5	0-1	No title, or Too brief (e.g. “Lab report”; “Mercury in fish”; Ascorbic acid in fruits”, etc).
		2-3	Too long, or Does not identify the complete subject of study (E.g “Determination of mercury”; “Determination of lead”, etc).
		4-5	Identify the complete subject of study and encapsulates the purpose of the report/study.
Objective	15	0	Section missing completely.
		1 – 7	Be too vague, ambitious or broad in scope. Just repeat each other in different terms. Just be a list of things related to the topic. Contradict with methods. Does not identify subject of study.
		8 – 15	Concise and brief. Be interrelated and describes how to achieve that objective. Clearly identify the subject of study. Related to the experiment that has been done.
Introduction	10	0	Section missing completely.
		1 – 5	Background info only from lab manual

		6 - 10	Clearly written, well structured, with evidence of extra reading. Clear outline of study's hypotheses. Does show something novel in it as compared to the supplied handout/laboratory manual. Does include the rationale for performing the experiment.
Experimental	10	0	Section missing completely.
		1 - 5	One or more subsections (e.g. chemicals or instrumentation) are missing. Confusing statement. Parts have been included under the wrong sub-section.
		6-10	Contains all of the relevant information about the method used; clearly and systematically described in such a way that a reader could replicate the study from the description.
Results	20	0	No Discussion section.
		1-6	Very lack attempt to relate experiment findings and collected data.
		6-12	Showing attempt to discuss the findings and collected data, but using inaccurate theories and justifications.
		13-20	Able to demonstrate analysis skill in discussing the results, including the inaccuracies of data, using logic and appropriate statements to justify the experiment outcome.
Discussion	20	0	No Discussion section.
		1-6	Very lack attempt to relate experiment findings and collected data.
		6-12	Showing attempt to discuss the findings and collected data, but using inaccurate theories and justifications.
		13-20	Able to demonstrate analysis skill in discussing the results, including the inaccuracies of data, using logic and appropriate statements to justify the experiment outcome.
Safety caution	5	0	Section is not present.
		1-3	Sentences are not in complete, focusing on minor or lack important steps.
		4-5	Tabulate at least 3 major and most important safety caution.
Conclusions	10	0	Section missing completely
		1 – 5	Conclusion is drawn but not supported by experimental evidence. No sensible conclusion is drawn.

			No clear evidence of a thorough understanding of the experiment and/or theory behind the experiment.
		6 – 10	Conclusion is drawn and supported by experimental evidence. Sensible conclusion is drawn. Shows clear evidence of a thorough understanding of the experiment and/or theory behind the experiment.
References	5	0	Reference not included in the report
		1 - 3	Incomplete references to the books or any other sources used in report.
		4 - 5	References in the text and in the reference list conform in all respects to the formatting convention (e.g. APA format). Complete references to the books or any other sources used in report. References in text are matched with references in reference list (e.g. no missing references).
Total Mark	100		

Section 3 Assessment of Understanding/Revision on conducted experiments (20%)

Score	Criteria
0	Unable to answer any questions.
1-5	Very little attempt to answer question correctly.
6-10	Most answers are incorrect, and some are irrelevant to the question type.
11-15	Some answers maybe very short or incomplete.
16-20	Questions are answered to the best of abilities and answers match the question types.

Late Report -1 marks / day

*For Section 3 Assessment - it is up to the lecturer in-charge to decide whether he/she wants to carry out a simple test or not. If he/she chooses not to, the 20% marks will be allocated back to Section 2 - Lab report.

List of Common Equipment and Glassware

Beaker

Conical Flask

Buchner Funnel

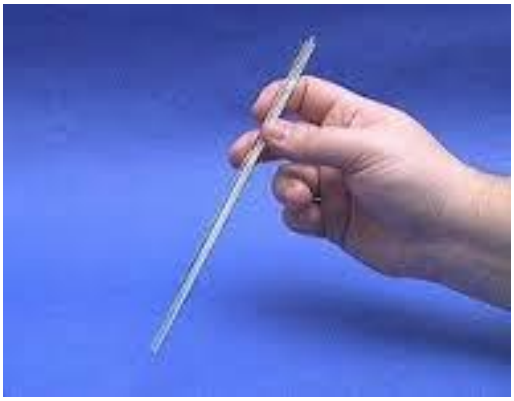
Pipette Filler

Pipette Bulb

Measuring Cylinder

Filter Flask

Wash Bottle



Glass Rod

Spatula

Volumetric Flask

Watch Glass

Weighing Bottle

Bulb

Glass Funnel

Thermometer

Experiment 1 Inorganic Chemistry I

CALIBRATION OF A 25-ML PIPETTE

1. INTRODUCTION

The graduation mark on a pipette being usually made at 20°C (whereas room temperature is much higher than this), the volume of the pipette must be calibrated before any volumetric analysis is carried out. Otherwise, the error in the graduation mark may exceed the error allowed in a measurement. A pipette is designed to deliver only one fixed volume of a liquid and it is calibrated for this volume only. Accuracy to two decimal places in mL is generally possible.

The pipette is calibrated by weighing distilled water in it at room temperature, and then calculating the volume from the weight of water in air. A correction for the buoyancy of air is included. The formula allows for the determination of the weight in vacuum, W after correction, where W_a is the weight in air. The volume is calculated from the weight and the density.

$$W = Wa + 1.06\ Wa/1000$$

Table 1: Density of water at various temperatures.

Temperature / (°C)	Density of water / (g.mL^{-1})
25	1.0000
26	0.99681
27	0.99654
28	0.99626
29	0.99597
30	0.99567
31	0.99537
32	0.99503
33	0.99473
34	0.99440

2. LEARNING OUTCOME

1. Able to handling a pipette, including -
   selecting the appropriate pipette for a given task, and using the selected pipette in the proper manner.
2. Able to understand the accuracy of pipettes in the laboratory by relating pipetted volumes to mass measurements.

HOW TO USE A PIPETTE

1. Rinse a 25-mL pipette with two or three small volumes of distilled water, and then with a complete volume of distilled water.
2. Do not immerse the tip of the pipette too deep into the water. The tip should not be above the water level so as to avoid any mishap during the suction of the water into the pipette via the pipette filler or suction bulb.
3. Draw the solution into the pipette until it reaches a level above the graduated mark.
4. Take the pipette away and wipe its tip with a piece of tissue paper.
5. Hold the pipette upright, and slowly release the pressure of the suction bulb or finger until the meniscus level is on the graduated mark. Touch the tip of the pipette on the dry side of the container until the drop at the end has been drained off.
6. Drain the contents to another container. Hold the pipette upright until the solution has been completely drained. Allow the liquid to flow out and then wait for 15 seconds after the last drop has emerged from the tip.
7. During the delivery, ensure that the pipette tip is always above the level of the solution in the receiver.

8. Finally, touch the tip of the pipette on the side of the container until the meniscus level in the pipette tip does not fall any farther.
9. **Do not blow or force out whatever remains in the tip.**

3. METHODOLOGY

1. Take the weight of the weighing bottle and its cover. (The volume of the weighing bottle must exceed 30 mL).
2. Transfer the distilled water in a 25-mL pipette into the weighing bottle.
3. Weigh the bottle, and repeat the weighing until two consecutive readings do not differ by more than 0.005 g.
4. Record the room temperature.
5. Calculate the volume of the pipette.

4. QUESTIONS

1. Does the pipette empty from full volume to zero or from zero to full volume?
2. Is the pipette designed to be emptied by gravity with the tip in contact with the vessel or to be expelled by blowing out with pipette filler? Explain.

VOLUMETRIC ANALYSIS

1. INTRODUCTION

Volumetric analysis is a quantitative analytical process whereby volumes are measured. A volume of reagent known as the standard solution of known concentration is chemically reacted with a solution of unknown concentration in order to determine the concentration of the unknown.

The solution that is used as a standard is called the **titrant**, which is delivered from a burette and is added until the reaction is complete. Titrations are based on acid-base neutralization, oxidation-reduction, and complex formation or precipitation reactions. The end-point of the titration, i.e., when the **analyte** has been completely reacted, is noted by observing the change of color of an **indicator**. The titrant is standardized against a **primary standard**, which is a pure reagent. In a **direct titration**, the titrant is added to analyte until the end point is observed whereas in a **back titration**, a known excess of a standard reagent is added to the analyte. A second standard reagent is then used to titrate the excess of the first reagent. Back titrations are used when the end point of the back titration is clearer than the end point of the direct titration or when excess of the first reagent is required. The experiment starts with preparation of potassium permanganate solution as primary standard. The equation for the standardization experiment is given in the ionic form as

$$5\ C_2O_4^{2-} + 2\ MnO_4^- + 16\ H^+ \longrightarrow 10\ CO_2 + 2\ Mn^{2+} + 8\ H_2O$$

The potassium permanganate solution now becomes the “standard solution” after it has been standardized against sodium oxalate, i.e., the molarity is known exactly.

This solution is then used to determine the quantity of nitrite ions in a back titration in the first part. A known volume of potassium permanganate solution is added to the nitrite solution under acid conditions. Because there is an excess, the excess is titrated against iron(II) ammonium sulfate solution. The back titration is explained by the equations

$$5\ NO_2^- + 2\ MnO_4^- + 6\ H^+ \longrightarrow 5\ NO_3^- + 2\ Mn^{2+} + 3\ H_2O$$

$$2\ MnO_4^- + 10\ Fe^{2+} + 16\ H^+ \longrightarrow 10\ Fe^{2+} + 2\ Mn^{2+} + 8\ H_2O$$

The second set of titration experiments involve the uses of sodium thiosulfate pentahydrate (formula weight of $Na_2S_2O_3{\cdot}5H_2O$ is 248.21), which is similarly standardized against the iodine in potassium iodate. Potassium iodate reacts with potassium iodide to release iodine. The reactions are represented by the ionic equations

$$IO_3^- + 5\ I_2^- + 6\ H^+ \longrightarrow 3\ I_2 + 3\ H_2O$$

$$2\ S_2O_3^{2-} + I_2 \longrightarrow S_4O_6^{2-} + 2\ I^-$$

The standardized sodium thiosulfate solution is used to determine the amount of chlorine in *chlorox*:

$$OCl^- + 2\ I^- + 2\ H^+ \longrightarrow I_2 + Cl^- + H_2O$$

2. LEARNING OUTCOME

1. Able to understand the concept of volumetric analysis.
2. Capable of using the standardized solution to determine the unknown solution concentration via titration.

3. METHODOLOGY

Part A

1. Weigh about 0.3 g sodium oxalate (Formula weight = 133.99 g/mol) and transfer it into a 100 mL volumetric flask. Add 20 mL distilled water followed by 50 mL dilute sulfuric acid (2.5 M), and then top up to the calibration mark with distilled water.
2. Weigh between 0.8 to 1.0 g of potassium permanganate and transfer it in a 250-mL volumetric flask. Add 50 mL distilled water to the flask and shake it to dissolve the solid, and then top up to the calibration mark with distilled water. Place some of the solution in the burette.
3. Pipette 25 mL of the sodium oxalate solution into a 250-mL conical flask. Heat the solution to 80°C, and titrate the hot solution with the potassium permanganate solution. Repeat two more times.
4. Calculate the molarity of the potassium permanganate solution.
5. Transfer 10 mL of the potassium permanganate solution into a 400-mL beaker and add dilute sulfuric acid (1:5) to the 100-mL mark.
6. Warm the beaker to 40°C, and then add 8 mL of the nitrite solution from a burette. The tip of the burette must be below the surface of the solution. Rinse the tip of burette with distilled water. Cool the solution to room temperature.
7. Place the standard iron(II) ammonium sulfate solution in a burette and titrate it against your nitrite solution. Repeat two more times.
8. Calculate the molarity of the nitrite solution.

Part B

1. Fill up a burette with sodium thiosulfate solution.
2. Weigh about 0.2 g potassium iodate and dissolve it with distilled water in a 100 mL volumetric flask.
3. Pipette 25 mL of the potassium iodate solution into a 250 mL conical flask. Add 1 g of potassium iodide followed by 5 mL of sulfuric acid (1 M).
4. Titrate the liberated iodine with the thiosulphate solution immediately. When the color of the mixture is pale yellow, add about 100 mL distilled water followed by 1 to 2 mL of freshly prepared starch solution.
5. Continue the titration until the solution is colorless.
6. Repeat the titration one more time and calculate the average molarity of the thiosulphate solution.
7. Pipette 10 mL of the *chlorox* solution into a conical flask. Add 40 mL of distilled water, 1 g of potassium iodide and 10 mL of sulfuric acid (1 M).
8. Titrate the liberated iodine with standard sodium thiosulphate solution.
9. Repeat the titration, and calculate the average concentration of hypochlorite in the chlorox (expressed in % Cl_2).

4. QUESTIONS

1. What is an acid base indicator and how it is been used for?
2. How to determine the equivalence point of a titration?

Experiment 3 Inorganic Chemistry I

GRAVIMETRIC DETERMINATION AND IR CHARACTERIZATION OF NICKEL COMPLEX

1. INTRODUCTION

The quantity of nickel ions in a salt is determined by precipitating the ions with dimethylglyoxime in an ammoniacal solution. The product is bis(dimethylglyoximato)nickel(II), chemical formula $Ni(C_4H_7O_2N_2)_2$. It is an insoluble red precipitate. The precipitate is collected and dried, and is then weighed. Dimethylglyoxime behaves as a monobasic acid in the reaction:

$$Ni^{2+} + 2\ C_4H_8O_2N_2 \rightarrow Ni(C_4H_7O_2N_2)_2 + 2H^+$$

Precipitation is effected by the addition of dilute ammonium hydroxide to a hot solution of the nickel salt and dimethylglyoxime.

The prepared compound will be characterized by Fourier Transform Infrared Spectroscopy, also known as FTIR Analysis or FTIR Spectroscopy, which is an analytical technique used to characterize organic, polymeric, and in some cases, inorganic materials. The FTIR analysis method uses infrared light to scan test samples and observe chemical properties. It is used by chemists to determine functional groups in molecules. FTIR Spectroscopy measures the vibrations of atoms, and based on this it is possible to determine the functional groups. Generally, stronger bonds and light atoms will vibrate at a high stretching frequency (wavenumber).

2. LEARNING OUTCOME

1. Able to understand the concept of gravimetric analysis.
2. Able to derive the mass of nickel calculated from the mass of precipitate.

3. METHODOLOGY

Part A

1. Pipette 25 mL of a nickel(II) sulfate solution into each of the two 400-mL beakers.
2. Add 5 mL of dilute hydrochloric acid (2 M) and dilute the solution to 200 mL with distilled water. Heat the solution to 60 – 80°C.
3. Add about 20 mL of dimethylglyoxime solution (1%) to the hot solution, followed by dropwise addition, with stirring, of ammonium hydroxide until precipitation occurs and the solution is slightly basic (about 2 mL of excess ammonium hydroxide). Test for completeness of precipitation by adding more dimethylglyoxime solution (1%).
4. Warm the precipitate for 30 minutes. Cool to room temperature and filter through a weighed No. 4 sintered glass crucible previously dried at 110°C.
5. Wash the precipitate with cold distilled water until it is free from any chloride (test with silver nitrate solution) and dry overnight.
6. Cool the crucible in a desiccator and weigh it to constant weight.
7. From the weight of the precipitate and a knowledge of the theoretical quantity of nickel in bis(dimethylglyoximato)nickel(II), calculate the concentration (in g L^{-1}) of nickel(II) ions in the solution.

Notes:

1. Carry out the analysis twice.
2. After the experiment, clean the crucibles by washing with hydrochloric acid followed by distilled water.
3. Keep the precipitate for IR analysis.

Part B

Solid FTIR sample preparation

Method 1: As Nujol mulls

Simplest method to obtain IR spectra of solids are Nujol (mineral oil) mulls between KBr plates.

Good results are obtained by this method only if the average particle size of the solid is somewhat less than the wavelength of light the particles are to transmit. Samples should therefore be grounded in a mortar to reduce the average particle size to 1 to 2 microns.
About 5 to 10 mg of finely ground sample are then placed onto the face of a KBr plate, a small drop of mineral oil is added and the second window is placed on top.
With a gentle circular and back-and-forth rubbing motion of the two windows, evenly distribute the mixture between the plates. The mixture should appear slightly translucent, with no bubbles, when properly prepared.
Place the sandwiched plates in the spectrometer and obtain a spectrum. Ideally, the strongest band should have a transmission of 0 to 10% and should not be totally absorbing for more than 20 cm^{-1}.

Attention:
If the bands are distorted (show fronting or tailing) the particle size is too large and some radiation incident on the mull has been scattered out of the sample beam. If a better spectrum is required, reduce the particle size further.

The KBr plates must be thoroughly cleaned after this procedure to prevent contamination of future samples. Wipe the windows with a tissue, then wash several times with methylene chloride, then ethanol. Use the polishing kit in the lab to polish the window surface. Wear gloves to prevent fogging. The cleaned surface should be clear and free from scratches.

Notes:
Remember that Nujol by itself shows a characteristic spectrum!

Method 2: KBr pellets/disks
Sample/KBr ratio
The concentration of the sample in KBr should be in the range of 0.2% to 1%. The pellet is much thicker than a liquid film, hence a lower concentration in the sample is required (Beer's Law). Too high a concentration usually causes difficulties in obtaining clear pellets. The IR beam is absorbed completely, or scattered from the sample which results in very noisy spectrum.

Although a homogeneous mixture will give the best results, excessive grinding of the potassium bromide is not required. The finely powdered potassium bromide will absorb more humidity (it is hygroscopic) from the air and therefore lead to an increased background in certain ranges. Make sure to work fast. Transfer some KBr out of the oven. Add about 1 to 2% of your sample, mix and grind to a fine powder in mortal with pestle. For very hard samples, add the sample first, grind, add KBr and then grind again. The sample must be very finely ground as in the Nujol mulling technique to reduce scattering losses and absorption band distortions.

Place just enough sample (fine powder) to cover the bottom of the pellet die. Place in press and press at 5000-10000 psi. Check pellet press brochure for details. Carefully remove the pressed sample from die and place in the FTIR sample holder. The pressed disc should be nearly clear if properly made. If it is translucent, regrind and repress. Insert into the IR sample holder and attach with scotch tape. Run the spectrum.

After use, the mortar and pistil should be cleaned with acetone and double distilled water, and be put back on top of the oven for drying.

4. QUESTIONS

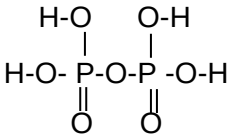
1. Draw the structural formula of bis(dimethylglyoximato)nickel(II) and describe the type of hybridization involved.
2. Explain why the temperature of the solution must be maintained at 60 – 80°C during the precipitation process.

Experiment 4 Inorganic Chemistry I

SODIUM ACID SALT OF HEPTAOXODIPHOSPHORIC ACID

1. INTRODUCTION

Heptaoxodiphosphoric acid (common name: pyrophosphoric acid) is a tetrabasic dinuclear oxoacid. Its chemical formula is $H_4P_2O_7$ and its structural formula is shown below.



Heptaoxodiphosphoric acid forms acid salts with metal ions. The general formula of the acid salts of Group 1 metal ions is $M_xH_{4-x}P_2O_7$ (the value of x is 1, 2 or 3).

In this experiment, you will first prepare a sodium acid salt of heptaoxodiphosphoric acid ($Na_xH_{4-x}P_2O_7$) from tetrasodium heptaoxodiphosphate (common name: tetrasodium pyrophosphate; chemical formula: $Na_4P_2O_7$). You will then determine its chemical formula as follows:

In the first step of the determination, an aqueous solution of silver nitrate ($AgNO_3$) is added to the acid salt to completely precipitate the normal salt, tetrasilver heptaoxodiphosphate, $Ag_4P_2O_7$. The equation is:

$$Na_xH_{4-x}P_2O_7(s) + 4\ Ag^+(aq) \longrightarrow Ag_4P_2O_7(s) + (4 - x)H^+(aq) + xNa^+(aq)$$

The number of moles of hydrogen ion (n_{H+}) present in the solution containing the precipitated $Ag_4P_2O_7$ is determined by neutralisation titration with a standard solution of sodium hydroxide.

$$H^+(aq) + NaOH(aq) \longrightarrow Na^+(aq) + H_2O(l)$$

In the second step of the determination, the precipitated $Ag_4P_2O_7$ is dissolved in nitric acid. The number of moles of Ag^+ ion ($n_{Ag}+$) present in the solution is determined by titration with a standard solution of thiocyanate (SCN^-).

$$Ag^+(aq) + SCN^-(aq) \longrightarrow AgSCN(s)$$

The value of x in the chemical formula $Na_xH_{4-x}P_2O_7$ is then calculated from the following relationship:

$$\frac{nAg+}{nH+} = \frac{4}{(4-x)}$$

2. LEARNING OUTCOME

1. Able to calculate the ion moles in chemical compound.
2. Able to determine chemical formula from ion moles quantification.

3. METHODOLOGY

A. Preparation of $Na_xH_{(4-x)}P_2O_7$

1. Weigh 5.0 g of tetrasodium heptaoxodiphosphate monohydrate ($Na_4P_2O_7.H_2O$) in a 250 mL conical flask. Add about 20 mL of distilled water and heat the mixture on a hot plate at 80 ^oC until a clear solution is obtained.
2. Add an equal volume of glacial ethanoic acid to the hot solution. Maintain the temperature of the mixture at 80 ^oC for several minutes until a white crystalline solid separates out.

3. Add 25 mL of ethanol to the hot mixture, filter the white crystalline solid under suction, and wash it with ethanol and finally with acetone. Allow the solid to dry in air and record its yield.

B. Number of moles of H^+ ions (Analysis is to be done in duplicate)

1. Accurately weigh 0.15 - 0.20 g of the white solid prepared in (A) into a small conical flask, and dissolve it in 50 mL of distilled water.
2. Add 1 g of sodium ethanoate crystals, stir to dissolve the crystals, and then add with continuous stirring, 18 mL (excess) of silver nitrate solution (5%). Continue stirring vigorously until the white precipitate formed coagulated (do not heat the solution).
3. Filter the precipitate on a sintered glass funnel attached to a Buchner flask and wash the precipitate thoroughly with cold distilled water. Combine the washed water with the filtrate in the Buchner flask.
4. Detach the sintered glass funnel containing the precipitate from the Buchner flask and **keep it for the analysis of Ag^+ ion in C.**
5. Add sodium chloride aqueous solution (5%) to the filtrate in the Buchner flask until all silver ions are precipitated as silver chloride. Add a few drops of phenolphthalein to the mixture and titrate it with a standard solution of sodium hydroxide (the molarity of sodium hydroxide solution is about 0.1 M) until the color changes from white colour to pale pink.
6. Calculate the number of moles of H^+ ions.

C. Number of moles of Ag^+ ions (Analysis is to be done in duplicate)

1. Discard the content of the Buchner flask from **B** and wash it three times with distilled water. Attach the sintered glass funnel containing the dry precipitate [from **B**] to the clean Buchner flask.
2. Dissolve the precipitate using several 10 mL portions of hot nitric acid (3M). Finally, wash the sintered glass funnel three times with cold distilled water. Combine the washed water with the filtrate in the Buchner flask.
3. Add 2 mL concentrated iron(III) alum solution and then titrate it with a standard solution of ammonium (or potassium) thiocyanate (the molarity of the standard solution is about 0.1 M) until a permanent reddish color is formed even after the flask is shaken vigorously.
4. Calculate the number of moles of Ag^+ ions.

4. QUESTIONS

1. Calculate the value of x.
2. Write the chemical formula and IUPAC name of the sodium acid salt of heptaoxodiphosphoric acid.
3. Calculate the percentage yield of the sodium acid salt of heptaoxodiphosphoric acid.

Experiment 5 Inorganic Chemistry I

SYNTHESIS AND STOICHIOMETRIC ANALYSIS OF HEXAAMMINENICKEL(II) CHLORIDE

1. INTRODUCTION

Hexaamminenickel(II) chloride, $[Ni(NH_3)_6]Cl_2$, is a coordination compound whose nickel atom and six ammonia molecules constitute the cation; the anion is the chloride ion.

The amount of ammonia in the compound is determined by adding a known excess quantity of an acid to neutralize the ammonia; the excess acid is determined by back-titration using a standard solution of sodium hydroxide, with bromocresol green as the indicator. This indicator is yellow in acidic solution and blue in basic solution.

The chloride ion in the compound is titrated against mercury(II) nitrate, with diphenylcarbazone as the indicator. The colour of the indicator at the end point is pale purple.

$$Hg^{2+} + 2Cl^- \longrightarrow HgCl_2$$

2. LEARNING OUTCOME

1. Able to synthesis a complex via crystallization and purification methods.
2. Able to quantify chemical component in a chemical complex via back titration method.

3. METHODOLOGY

Procedure for the synthesis of hexaamminenickel(II) chloride

1. Dissolve 4.0 g hydrated nickel(II) chloride in 6 mL distilled water in a 50-mL flask.
2. In the fume cupboard, add 12 mL concentrated ammonia to the above solution and warm the mixture for about 10 minutes on a hot plate.
3. Cool the solution in an ice-bath, and while stirring it with a glass rod, add 6 mL ethanol. Note the formation of a solid.
4. When all of the solid has formed, filter it under suction and wash it with a few mL of cold concentrated ammonia solution, followed with ethanol, and finally with acetone.
5. Record the weight of the solid.

Procedure for the analysis of ammonia (This analysis is to be done in duplicate.)

1. Weigh about 0.2 g of hexaamminenickel(II) chloride and place it in a 250-mL conical flask.
2. Dissolve the compound by adding 25 mL standard hydrochloric acid from a burette. (Note the molarity of the standard acid)
3. Add 3-5 drops of bromocresol green to the solution in the conical flask.
4. Titrate with standard sodium hydroxide solution until the colour of the indicator changes to pale blue.

Procedure for the analysis for chloride ion (This analysis is to be done in duplicate.)

1. Weigh about 0.2 g of hexaamminenickel(II) chloride and dissolved it in 10 mL of distilled water.
2. Add 2 drops of bromophenol blue indicator to the solution, and using a dropper, add nitric acid (1M) until the color of the solution changes to green. Add 5 drops diphenycarbazone and 25 mL 2-propanol to the mixture.

3. Titrate the mixture with standard mercury(II) nitrate solution until the colour of the indicator changes to pale purple.

(Standard Hydrochloric acid 0.270 M)

Calculations

Weight of hexaamminenickel(II) chloride

	Weight / (g)
$[Ni(NH_3)_6]Cl_2$ + watch glass	
Watch glass	
$[Ni(NH_3)_6]Cl_2$	

Analysis for NH_3

Weight of $[Ni(NH_3)_6]Cl_2$/g		
Molarity of HCl		
Initial volume of HCl, $V_{initial}$/mL	25.00	25.00
Molarity of NaOH		
Final burette reading/mL	a1	b1
Initial burette reading/mL	a_2	b_2
Volume of NaOH/mL	a_1-a_2	b_1-b_2
Volume of excess HCl, V_{excess}* /mL	X_1	X_2
Volume of reacted HCl, $V_{reacted}$/ mL	25.00 – X_1	25.00 – X_1
Moles of NH_3#		
Weight of NH_3		
% NH_3 in $[Ni(NH_3)_6]Cl_2$		

***** $V_{(HCl,\ excess)} = M_{(NaOH)}\ V_{(NaOH)} / M_{(HCl)}$

moles of ammonia = moles of HCl = $M_{(HCl)}\ V_{(HCl)reacted} /1000$

Analysis for Cl^- ion

Weight of $[Ni(NH_3)_6]Cl_2$/g		
Molarity of Hg^{2+}		
Final buret reading/mL	a_1	B_1
Initial buret reading/mL	a_2	B_2
Volume of Hg^{2+}/mL	a_1-a_2	B_1-b_2
Moles of Cl^-*		
Weight of Cl^-		
% Cl^- in $[Ni(NH_3)_6]Cl_2$		

***** moles of Cl^- = 2 moles of Hg^{2+} = 2 $M_{(Hg2+)}\ V_{(Hg2+)}$ / 1000

4. QUESTIONS

1. Write an equation for the formation of $[Ni(NH_3)_6]Cl_2$ and calculate its percentage yield in your experiment.
2. Assuming that the complex consists of Ni^{2+}, NH_3 and Cl^- only, verify that its empirical formula is $[Ni(NH_3)_6]Cl_2$ from your analytical results. What other information do you need to enable you to write its molecular formula?

Experiment 6 Inorganic Chemistry I

CHEMICAL BONDING AND MOLECULAR POLARITY

1. INTRODUCTION

Electronegativity is a measure or the relative attraction an atom has for the shared electrons in a bond. The higher the electronegativity value for an element is, the greater the electron attracting ability of the atom for the shared electrons. The difference in the electronegativity values of the atoms in a bond is the key to predicting the polarity of that bond. **Polarity** is a measure or the inequality in the sharing of bonding electrons.

When two identical atoms (atoms of equal electronegativity) share one or more pairs of electrons, each atom exerts the same attraction for the electrons, which results in the electrons being shared equally. This type of bond is called a nonpolar covalent bond. A **nonpolar covalent** bond is one in which the sharing of bonding electrons is equal.

When two atoms involved in a covalent bond are not identical (atoms of different electronegativities) the atom that has higher electronegativity attracts the electrons more strongly than the other atom; this results in unequal sharing of electrons. This type of bond is called a polar covalent bond. A **polar covalent** bond is one in which the sharing of bonding electrons is unequal. It follows that most chemical bonds are neither 100% covalent (equal sharing) nor 100% ionic (no sharing); instead, they fall somewhere in between (unequal sharing).

Bond character based on electronegativity differences
It is possible to predict whether a given bond will be non-polar, polar covalent, or ionic based on the electronegativity difference, since the greater the difference, the more polar the bond.

Electronegativity difference, ΔχP	**Bond**
$\Delta\chi < 0.4$	covalent
$0.4 < \Delta\chi < 1.7$	polar covalent
$\Delta\chi > 1.7$	ionic

When there are three or more atoms bonded together, it is possible to have a nonpolar molecule even though there are polar bonds present. When a molecule contains more than two atoms, we must consider its geometry to decide if the bond is polar. Consider, as a simple example, a molecule AB2. Suppose the central atom A is more electronegative than B. Two geometries are possible, bent and linear. In predicting the polarity of molecules, the following generalizations might prove useful:

1. Molecules containing identical atoms are always nonpolar.
2. Molecules containing unlike atoms are:

    i. Nonpolar, if the arrangement of the atoms is symmetrical.
    ii. Polar, if the arrangement of the atoms is nonsymmetrical.

2. LEARNING OUTCOME

1. Able to identify the bond type, polarity and shapes of chemical molecules.
2. Able to estimate the electronegativity difference and the percentage of ionic character of chemical molecules.

3. METHODOLOGY

1. Assemble the first set of model of given molecules.

a. Use the following colors to represent the atoms:

Hydrogen..... Yellow

Chlorine ..... Green

Oxygen ..... Red

Nitrogen..... Black

Bromine ..... Purple

Carbon..... Black

Sulfur ..... Red

Iodine..... Orange

b. Use a set of spring connectors for multiple bonds.

c. Draw the Lewis Electron Dot formula for each formula.

*Use dots to represent both shared and unshared valence electrons.

d. Evaluate the bond type, note the molecular shape and predict if the molecule is polar or not.

2. Assemble the next set of model molecules. Evaluate the bond types, note the molecular shape and predict if the molecule is polar is not. If the molecule contains more than one type of bond (three different atoms), each bond should be evaluated individually in order to predict if the molecule is polar or not.

3. Take the models apart, and place the balls and the connectors in the kit in the same order you have found them at the beginning.

Results

Formula	Electron-Dot Formula	Bond Type	Shape of Molecule	Kind of Molecule
H_2		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
F_2		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
Br_2		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
I_2		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
N_2		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
Cl_2		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
HCl		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
HBr		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
BrCl		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar

Formula	Electron-Dot Formula	Bond Type	Shape of Molecule	Kind of Molecule
H_2O		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
CO_2		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
H_2S		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
NH_3		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
NCl_3		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
CCl_4		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
CH_3Cl		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
CH_2Cl_2		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
$CHCl_3$		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar
CH_3Br		❑ - Polar ❑ - Nonpolar	❑ - Linear ❑ - Bent ❑ - Pyramidal ❑ - Tetrahedral	❑ - Polar ❑ - Nonpolar

4. QUESTIONS

1. Calculate the electronegativity difference and the percentage of ionic character for each of the bonds listed below.
   a. Use Pauling electronegativity values
   b. Estimate between the given values of percentage ionic character as needed. Linus Pauling proposed an empirical relationship which relates the percent ionic character in a bond to the electronegativity difference.

   Percent ionic character = $(1-e^{-(\Delta x/2)^2})$ x 100

Bond	Electronegativity Difference	Percentage of Ionic Character
H-O		
H-N		
H-Cl		
Br-Cl		
H-S		
H-C		
Cl-Cl		
C-O		
K-Br		
Na-O		

Pauling Electronegativity Values

1 **H** 2.20																
3 **Li** 0.98	4 **Be** 1.57											5 **B** 2.04	6 **C** 2.55	7 **N** 3.04	8 **O** 3.44	9 **F** 3.98
11 **Na** 0.93	12 **Mg** 1.31											13 **Al** 1.61	14 **Si** 1.90	15 **P** 2.19	16 **S** 2.58	17 **Cl** 3.16
19 **K** 0.82	20 **Ca** 1.00	21 **Sc** 1.36	22 **Ti** 1.54	23 **V** 1.63	24 **Cr** 1.66	25 **Mn** 1.55	26 **Fe** 1.83	27 **Co** 1.88	28 **Ni** 1.91	29 **Cu** 1.90	30 **Zn** 1.65	31 **Ga** 1.81	32 **Ge** 2.01	33 **As** 2.18	34 **Se** 2.55	35 **Br** 2.96
37 **Rb** 0.82	38 **Sr** 0.95	39 **Y** 1.22	40 **Zr** 1.33	41 **Nb** 1.6	42 **Mo** 2.16	43 **Tc** 1.9	44 **Ru** 2.2	45 **Rh** 2.28	46 **Pd** 2.20	47 **Ag** 1.93	48 **Cd** 1.69	49 **In** 1.78	50 **Sn** 1.96	51 **Sb** 2.05	52 **Te** 2.1	53 **I** 2.66
55 **Cs** 0.79	56 **Ba** 0.89	57 **La** 1.1	72 **Hf** 1.3	73 **Ta** 1.5	74 **W** 2.36	75 **Re** 1.9	76 **Os** 2.2	77 **Ir** 2.20	78 **Pt** 2.28	79 **Au** 2.54	80 **Hg** 2.00	81 **Ti** 1.62	82 **Pb** 2.33	83 **Bi** 2.02	84 **Po** 2.0	85 **At** 2.2
87 **Fr** 0.7	88 **Ra** 0.9															

2. Both water and carbon dioxide are triatomic molecules. Explain why one of these is polar and the other is nonpolar.

3. Classify each of the following molecules as:

Br_2	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
CCl_4	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
CO_2	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
BaO	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
KCl	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
MgS	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
BeO	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
H_2O	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
AlN	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
LiBr	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
MgO	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule
HI	❑ - Ionic Crystal ❑ - Polar Covalent Molecule ❑ - Nonpolar Covalent Molecule