COURSE
GUIDE

CHM 291 PRACTICAL MANUAL
PRACTICAL INORGANIC CHEMISTRY III

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Introduction to Chm291 Practical Sessions

CHM291 titled "Practical Inorganic Chemistry III is a practical-based course that will expose you to major reactions of the transition elements. You will be participating in nine (9) practical sessions. However, some of the experiments are in parts. For instance, under the practical session featuring the chemistry of Chromium, the experiment is in three parts. You are required to participate in all the experiments. You will enjoy the practical sessions. Endeavor to read through your course material prior to attending and participating in all the practical sessions. Always follow all Laboratory rules for your own good and that of your laboratory colleagues.

Laboratory Safety Regulations

The following practices will be followed:

1. Report any accident to the teacher immediately, no matter how minor, including reporting any burn, scratch, cut, or corrosive liquid on skin or clothing.
2. Prepare for each laboratory activity by reading all instructions. Follow all directions implicitly and intelligently. Make note of any modification in procedure given by the instructor.
3. Any science project or individually planned experiment must be approved by the teacher.
4. Use only those materials and equipment authorized by the instructor.
5. Inform the teacher immediately of any equipment not working properly.
6. Clean up any nonhazardous spill on the floor or work space immediately.
7. Wear appropriate eye protection, as directed by the instructor, whenever you are working in the laboratory. Safety goggles must be worn during hazardous activities involving caustic/corrosive chemicals, heating of liquids, and other activities that may injure the eyes.
8. Splashes and fumes from hazardous chemicals present a special danger to wearers of contact lenses. Therefore, students should preferably wear regular glasses inside splash-proof goggles during all lab activities where exposure to chemicals or chemical fumes is possible.
9. Students with open skin wounds on hands must wear gloves or be excused from the laboratory activity.
10. Never carry hot equipment or dangerous chemicals through a group of students.
11. Check labels and equipment instructions carefully. Be sure correct items are used in the proper manner.

12. Never taste anything or touch chemicals with the hands, unless specifically instructed to do so.
13. Test for odor of chemicals only by waving your hand above the container and sniffing cautiously from a distance.
14. Eating or drinking in the laboratory or from laboratory equipment is not permitted.
15. Use a mechanical pipette filler (never the mouth) when measuring or transferring small quantities of liquid with a pipette.
16. When heating material in a test tube, do not look into the tube or point it in the direction of any person during the process.
17. Never pour reagents back into bottles, exchange stoppers of bottles, or lay stoppers on the table.
18. When diluting acids, always pour acids into water, never the reverse. Combine the liquids slowly while stirring to distribute heat buildup throughout the mixture.
19. Keep hands away from face, eyes, and clothes while using solutions, specimens, equipment, or materials in the laboratory. Wash hands as necessary and wash thoroughly at the conclusion of the laboratory period.

MAIN COURSE

CONTENT

Experiment 1 Preparation of Chloropentaamine Cobalt (III) Chloride, $[Co(NH_3)_5Cl]Cl_2$

Experiment 1 Precipitation Reactions

Title: Study of Precipitation Reactions in Aqueous Solutions
Aim: To identify ions present in various aqueous solutions via chemical reaction known as ionic precipitation

Introduction

Precipitation is a form of chemical reaction involving ions in solution. The ions in solution are totally mobile and independent of each other. Precipitation is based on ionic bonding, that is the attraction between positively and negatively charged ions which leads to the formation of three-dimensional ionic lattice or network.

Precipitation involves the formation of an insoluble solid called precipitates when two ionic solutions react. For instance, an aqueous solution of Copper (II) chloride having Cu^{2+} and Cl^- ions upon reacting with an aqueous solution of NaOH having Na^+ and OH^- ions, a blue precipitate is formed. Since Copper (II) hydroxide is insoluble in water just like most hydroxides, we could deduce the identity of the blue precipitate to be Copper (II) hydroxide. (Note: Copper salts gives a blue precipitate). You may wonder why Copper (II) hydroxide and not NaCl is formed. Well based on Solubility rules (See **page 26** of CHM291 Course Material for **Solubility Rules**), sodium compounds and most chlorides are soluble in water, hence the selective precipitation of $Cu(OH)_{2}$.. In the reaction vessel (test tube), Na^+ and Cl^- ions are present but not involved in the reaction. They are called "Spectator ions".

In writing an ionic equation, spectator ions are excluded because such equations involve the interaction of ions.

For example;
$Cu^{2+(}{}_{aq)} + 2OH^-{}_{(aq)} \rightarrow Cu(OH)_2(s)$ ---------------- Equation (1) This is an ionic equation

The full equation for the reaction is shown in Equation (2) below
$CuCl_2(aq) + 2NaOH(aq) \rightarrow Cu(OH)_2(s) + 2NaCl(aq)$ --------------- Equation (2)

Reagents: Copper (II) chloride, Iron (II) sulphate, Iron (III) nitrate, Iron (III) oxide, Magnesium carbonate, Potassium carbonate, 0.4M Sodium hydroxide.

Apparatus/Equipment: Spatula, $10cm^3$ or $20cm^3$ test tubes, glass rod, test tube racks, dropping pipette.

Precautionary Measures: Ensure all protective wears (Safety goggles, hand gloves, face masks, laboratory apron or coat) are worn before commencing the experiments.

Procedure

- Use a spatula to transfer a tiny quantity of each of the compound provided to a test tube, enough to cover just the end of the spatula.
- Add about 5 cm ' of de-ionized (or distilled) water and shake the tube, and stir using a glass rod if necessary, to see whether the solid is capable of dissolving in water. If you use too much solid, it may be hard to tell whether any has dissolved.
- Note the colour of any solution formed. You may need to hold a test tube of water alongside for comparison, because some of the solutions may be very pale in colour. If the solid does not dissolve record this fact in your table.

Results

Sample	Appearance	Solubility in water	Colour of solution	Addition of NaOH(aq) Observation Inference	
SALT A					
SALT B					

SALT C					
SALT D					
SALT E					
SALT F					

Questions

1. Were any of the metal compounds you used coloured? If so, identify their positions in the periodic table. **2marks**
2. Did the formula for any of the compounds include the formula for water? If so, what does this tell you about the solid? **2marks**
3. Did the compounds obey the solubility rules? **2marks**
4. Construct ionic equation for each of the reactions with sodium hydroxide. **7marks**

ANSWERS

Experiment 2 Investigation of Transitional Properties of D-Block Elements

Title: Experiments on the transitional Properties of D-block elements

Aim: To examine some of the solution chemistry of the transition elements

Introduction

The d- and f-block elements are generally referred to as the transition elements. They are located between the s and p-block elements and their properties reflect transition from electropositive nature of the s-block to electronegative nature of the p-block elements. The d-block elements are characterized by the gradual filling of the penultimate (n-1) d orbitals of the main shell of atom. The valence shell configuration of the d-block elements can be represented by (n-1) d1-10ns1-2npo. One or two electrons are found in ns-state, while the other valence electrons are in (n-1) d state. Cu, Ag and Au (the coinage metals) are considered transition elements because in their commonly occurring oxidation states $Cu2^{2}$+(3d9), $Ag2^{2}$+ (4d9, $Au3^{2}$+ (5d8), their d-orbitals are partly filled. Although Zn, Cd and Hg do not possess partly filled (n-1) d orbitals both in their elementary state [(n-1) $d^{10}ns^{2}$] and their +2 oxidation states [(n-1) d^{10}], their chemical behaviour is on the whole similar to that of transition elements, hence they are appropriately considered as transition metals.

Precautionary measures: Most of the reagents to be used in the following experiments (Experiment 2A and Experiment 2B) are either eye/skin irritants, carcinogenic or hazardous. Therefore, ensure to put on all laboratory protective wears before commencing your laboratory work.

Experiment 2A

Title: Investigation of transition metals in their lowest oxidation states

Aim: To examine evidence of the formation of complexes and change in oxidation states by the transition elements

Reagents: Hydrogen sulphide, 0.1 M solutions of Titanium (III) sulphate, vanadium (III)alum, chromium (III)chloride, manganese (II) chloride, iron (II) sulphate, iron (III) chloride, cobalt and nickel chlorides, copper (II) sulphate, silver nitrate, zinc and cadmium chlorides, mercurous nitrate and mercury (II) chloride and ammonium thiocyanate.

2.0M solutions of sodium hydroxide, ammonia, hydrochloric acid and sodium hypochlorite, and 1.5M solution of sodium carbonate.

Apparatus: Standard flasks, Analytical balance, spatula, weighing bottles, stirring rod, test tubes, test tube racks, reagent bottles, glass funnels, beakers, water bath

Procedure

a. Add a few drops of the test solutions to respective test tubes
b. Add 2.0 M sodium hydroxide dropwise until an excess is present.
c. Add 2.0 M ammonia dropwise until an excess is present.
d. Add 1.5 M sodium carbonate.
e. Add 0.1M ammonium thiocyanate until an excess is present.
f. Acidify with 2M hydrochloric acid and pass hydrogen sulphide.
g. If there is no reaction in (f), add 2.0 M ammonia until alkaline.
h. Add 2.0 M sodium hypochlorite solution and warm gently.

NOTE: Each test solution shall be examined via its reaction with each of the solutions slated in b-h. Look for the precipitation of insoluble compounds and for colour changes. Try to work out what has happened; for example, if a precipitate dissolves in an excess of a reagent it is likely that a complex has been formed. Write your results and observations in the table below.

RESULTS

	NaOH	NH_3	Na_2CO_3	NH_4SCN	$HCl + H_2S$	NaOCl + Heat
Ti (III) solution						
V (III) solution						

Cr (III) solution						
Mn (II) solution						

Fe (II) solution						
Fe (III) solution						

Co (II) solution						
Ni (II) solution						

Cu (II) solution						
Zn (II) solution						

Note: column 2: NaOH, column 3: NH_3, column 4: Na_2CO_3, column 5: NH_4SCN, column 6: HCl + H_2S, and column 7: NaOCl + heat.

QUESTIONS

Work out the oxidation states of the transition elements in the following ions and compounds:

(a) MnO_4^-
(b) MnO_2
(c) $Cr_2O_7^{2-}$
(d) K_2CrO_4
(e) Cr_2O_3
(f) $KCrO_3Cl$
9marks

ANSWERS

Experiment 2b

Title: Solution chemistry of transition elements

Aim: To determine the solution chemistry of transition elements in relation to change in oxidation states

Reagents: 0.2M solutions each of Potassium chromate, potassium manganite (VII), ammonium vanadate, Copper (II) nitrate, Iron (III) nitrate, iron (II) sulphate and potassium iodide.

0.5M Cobalt nitrate, 0.1M Silver nitrate, 0.1M Potassium thiocyanate, 1.0M Hydrochloric acid, 1.0M Sosium hydroxide, 3.0M Ammonia solutions, zinc powder, starch solution.

Apparatus: Standard flasks, Analytical balance, spatula, weighing bottles, stirring rod, test tubes, test tube racks, reagent bottles, glass funnels, beakers,

Precautions: Handle all chemicals with care as some are flammable (Zinc), skin/eye/respiratory irritants.

Procedure

Two drops each of the transition elements solutions (test solutions) are to be used respectively.

1. To vanadium solution, add one drop of dilute HCl and a small piece of zinc.
2. To chromium solution, add one drop of silver nitrate solution.
3. To manganese solution, add one drop of iron (II) solution.
4. To iron (II) solution, add one drop of potassium iodide solution. After one minute, add one drop of starch test solution.
5. To cobalt solution, add one drop of ammonia solution.
6. To copper solution, add one drop of ammonia solution.
7. To Zinc, add two drops of sodium hydroxide.

Record your observations from each of the tests and your inference/ comment on your observations in the table below.

RESULTS

Solution of metal ions	Test	Observation	Explanation/Inference
V			
Cr			
Mn			

Fe			
Co			
Cu			
Zn			

Question
Which of the elements among the ones that you have tested does not behave as a transition element? Give concise reason for your answer. **5marks**

Answer

Experiment 3 Chemistry of vanadium

Title: Illustrating the oxidation states of vanadium

Aim: To illustrate the presence of several different oxidation states for vanadium, and to show how it is possible to change from one oxidation state to another.

Introduction

Vanadium as a transition element exhibits variable oxidation state of +5, +4, +3, +2, +1, 0 and (-1). On heating, vanadium combines directly with non-metals to form their corresponding compounds. Vanadium in +5 oxidation state is oxidizing and stable compounds are formed with strongly electronegative elements like oxygen and fluorine. Thus, the chemistry of V5+ is dominated by the formation of pentafluoride, oxohalides and penta-oxide: $VF5_5$, $VOF3_3$, $V2O5_5$. Zinc is used to reduce a yellow solution of ammonium vanadate(V) to a mauve solution containing vanadium(II) ions. The intermediate oxidation states of vanadium (IV) (blue) and vanadium(III) (green) are also seen. This is to demonstrate that different oxidation states of transition metal ions often have different colours and that electrode potentials can be used to help predict the course of redox reactions (via the 'anticlockwise rule' or otherwise).

Reagents: 11.7g of Ammonium vanadate (V), zinc powder (15g), acidified potassium permanganate (100cm^3 of approximately 0.25 mol dm^{-3} potassium permanganate in 1 mol dm^{-3} sulphuric acid; that is 4g potassium manganate (VII) in 100 cm^3 of 1.0 M sulphuric acid.

Make up a 0.1 mol dm$^{–3}$ solution of ammonium metavanadate by dissolving 11.7 g of solid in 900 cm^3 of 1 mol dm$^{–3}$ sulphuric acid and making up to 1 dm^3 with deionised water. This yellow solution is usually represented as containing $VO2^{2+}$(aq) ions (dioxovanadium(V) ions) in which vanadium has an oxidation number of +5.

Apparatus: One 1 dm3 conical flask, Filter funnel, boiling tube, Dropping pipette, Four petri dishes (optional), Access to an overhead projector (optional), Test-tubes and rack (optional), white tile.

Precautionary measures: Wear gloves and safety googles. Do not breath in Zinc powder. Wear mask when measuring Zinc powder.

Procedure

1. Place 500 cm^3 of the ammonium metavanadate solution in a 1 dm^3 conical flask and add about 15 g of powdered zinc.

2. Observe if there is effervescence and take note of the changes in the colour of the solution immediately, after a few seconds, after about fifteen minutes and thereafter. The changes in colour indicates a change in oxidation state of Vanadium. (***See the table on page 29 of CHM291 course material for the colour of different species of vanadium ions in solution***).
3. When the reaction has reached the mauve stage, filter off a little of the solution into a boiling tube and add acidified potassium permanganate solution dropwise. Notice the changes in colour as vanadium is re-oxidised. Take care with the final few drops of acidified $KMnO_4$ to avoid masking the final colour of the vanadium ion in solution with the purple of permanganate ions.
4. Record your observations and inference on the table below.

RESULTS

TEST	**OBSERVATION**	**INFERENCE**
NH_4VO_3 + Zinc powder		
Immediately as reaction proceeds		
After few seconds		
At about 15 minutes (more or less than 15minutes)		
At about 16minutes or more		
Vanadium solution + acidified $KMnO_4$ dropwise		

Questions

1. Write equations for the reactions involved in the change of each oxidation states of Vanadium from the highest to the lowest. ***12 marks.***
2. State the role of $KMnO_4$ and Zinc in the reactions carried out. ***2marks***

ANSWERS

Experiment 4 Chemistry of Chromium

Title: Experiment on the Chemistry of Chromium

Aim: To illustrate the oxidation and reduction reactions of Chromium

Introduction

Chromium is a transition metal having variable oxidation states of +6, +3 and +2. Cr dissolves in non-oxidizing mineral acids like HCl and sulphuric acid with the evolution of H_2 and formation of chromous compounds. Cr has the ability to transit from one oxidation state to another, either from +6 to +3 to +2 or +3 to +6 under certain reaction conditions.

NOTE: Endeavor to read through page 45 to 47 of your CHM291 Course material prior to this laboratory experiment.

This experiment is in three parts: A, B and C.

Reagents

Part A: Chromium (III) chloride hexahydrate, KOH, 2M H_2O_2, glacial acetic acid, NaOH.

Part B: potassium dichromate, sulphuric acid, zinc dust

Part C: 5% hydrogen peroxide, 0.2M potassium dichromate

Apparatus: 250ml beaker, stirring rod, temperature-controlled heating device, evaporating basin or dish, refrigerator or ice bath, desiccator, conical flask, stopper with Bunsen valve, analytical balance, large (7cm) buchner funnel.

Precautionary Measures: Chromates and dichromates are carcinogens. Handle with utmost care.

Part A Procedure: Change of Oxidation state from +3 to +6

- Dissolve 14 g (0. 05 mole) of chromium (III) chloride hexahydrate in 40 ml of water in a 250 ml beaker.
- Add slowly, with stirring, a solution of 17 g (0'25 mole) of potassium hydroxide in 40 ml of water. Note your observations.
- Now warm the mixture gently, do not boil, and add slowly with stirring, 60 cm^3 of ‘20 volume' (2M) hydrogen peroxide.

- When all the hydrogen peroxide has been added, bring the solution to the boil for a few minutes to destroy any excess of oxidizing agent, then filter hot through a large (7 cm) Buchner funnel.
- Transfer the solution of potassium chromate to an evaporating basin and boil away half the liquid.
- Add to the hot solution 5 ml (0.09mole) of glacial acetic acid, and cool the solution in a refrigerator or ice bath.
- Filter off the orange crystals formed, wash with a small volume of iced water and dry in a desiccator containing sodium hydroxide.
- Record the weight of the orange crystals after drying.

QUESTIONS

1. What did you notice in the first reaction mixture and what is the identity of the compound formed in the first reaction mixture? 6mks

2. What do you think the orange crystals could be? Support your answer with a chemical equation illustrating the reaction. 6mks

3. Haven weighed the crystals, calculate the amount in mole of the crystals obtained supposing the Molar mass of the compound is 294.185g/mol. 5mks

RESULTS/CALCULATIONS

Part B Procedure: Change of oxidation state from +6 to +3 to +2

- Dissolve 3g (0'01 mole) of potassium dichromate in 10 ml of water in a conical flask.
- Add 10 ml of 2M sulphuric acid to the dichromate solution and 3 g (an excess) of zinc and fit the conical flask with a stopper carrying a Bunsen valve. Note and record your observations in a tabular form (test, observation, inference).
- Expose the mixture to air and note your observations too.

Questions

1. Why expose the reaction mixture to air? 2mks
2. What is the essence of the addition of sulphuric acid to the dichromate solution? 2mks
3. In the above experiment, what role does zinc dust play? 2mks

RESULTS

8marks for correct presentation of tests, observation and inference made.

Part C Procedure: Reduction of dichromate (vi) ions to chromate (iii) ions by Hydrogen peroxide

Add a volume of 5% hydrogen peroxide to aqueous solution of potassium dichromate (0.2M). Note what happens in the reaction mixture and as the reaction continues. Present your observations in this format:

Test	Observation	Inference
	2mks	2mks

QUESTIONS

1. State the oxidation numbers of chromium in $Cr_2O_7^{2-}$ and CrO_4^{2-}. 4mks

2. Classify the following changes as oxidation reduction and/or acid-base giving a reason in each case.

i. $Cr_2O_7^{2-} \rightarrow CrO_4^{2-}$

ii. $Cr_2O_7^{2-} \rightarrow Cr^{3+}$

Experiment 5 Chemistry of Manganese

Title: Investigation of uncommon oxidation states of manganese

Aim: To make samples of some of the less common oxidation states of manganese

Introduction

Manganese is more electropositive than its neighbours in the transition series. It is outstandingly reactive. It liberates hydrogen from water and dissolves readily in dilute mineral acids to form Mn (II) salts. In +2 oxidation state, manganese has a d^5 configuration. This symmetrical d^5 configuration confers extra stability on Mn^{2+} compounds, consequently Mn (II) ions shows high resistance to oxidation and reduction. Manganese (II) forms series of salts with all common anions, most of which are soluble in water. For example manganese (II)sulphate $MnSO_4.7H_2O$ contains the aqua complex ion $[Mn(H_2O)_6]^{2+}$.

Mn (III) has a d^4 configuration and is strongly oxidizing in aqueous solution. There is a high tendency for Mn (III) compounds in solution to disproportionate into Mn^{4+} as in MnO_2 and $Mn^{2+}{}_{(aq)}$. Stable compounds are formed mainly by O-donor ($C_2O_4{}^{2-}$, $SO_4{}^{2-}$) and strong field ligands like CN^-. Mn(III) compounds are obtained either by oxidation of Mn(II) salts or by reduction of $KMnO_4$.

Reagents: Potassium manganate(VII) (permanganate) solution,(0.01 M $KMnO_4$), sulphuric acid, dilute, 1 M H_2SO_4, sodium hydroxide solution (2 M NaOH), manganese(IV) oxide (manganese dioxide, MnO_2) manganese(II) sulphate-4-water ($MnSO_4 \cdot 4H_2O$) sulphuric acid, concentrated. H_2SO_4.

Apparatus: test tubes and test tube rack, spatula, stirring rod, filter paper, funnel, safety spectacles.

Precautionary Measures: Do not point the mouth of a test tube towards your neighbour in the laboratory or yourself. Acids and alkalis are corrosive substances so handle with utmost care.

Procedure:

This experiment involves three methods.

Method A

1. Put about 5 cm^3 of potassium manganate(VII) solution in each of three test-tubes.
2. To one of the three tubes add about 3 cm^3 dilute sulphuric acid and to another add about
3 cm^3 sodium hydroxide solution.
4 To each of the three tubes add a little solid manganese(IV) oxide and stir for about a minute.
5 Filter enough of each mixture into a clean tube to see the colour of the filtrate clearly. Use a fresh filter paper for each mixture.
6 One
7 of the tubes should now have in it a clear green solution of Mn[VI). Add to this a little dilute sulphuric acid.

Method B

a. Dissolve about 0.5 g manganese(II) sulphate in about 2 cm^3 of dilute sulphuric acid in a test-tube.
b. Carefully add about 10 drops concentrated sulphuric acid and cool the tube under a running tap.
c. Add a few drops of potassium manganate[VII) solution to obtain a deep red solution of Mn(III).
d. Dilute the red solution with about five times its volume of water, wait a few moments, and note any colour change.

Method C

i. In each of two test-tubes dissolve a little manganese (II) sulphate in water and add an equal volume of sodium hydroxide solution to obtain a precipitate of manganese (II) hydroxide.
ii. To one of the two tubes add a little manganese (IV) oxide and stir.
iii. Let both tubes stand for a few minutes and note any changes.

Note your observations and give answers to these questions on the Result/Observation section

Questions/Results and Observations

Method A

1. Explain why only one of the three mixtures reacted to give green Mn(VI).
2. What happened when acid was added to Mn (VI)? Explain.

Method B

1. Explain what happened when the Mn(III) solution was diluted.
2. Can you see any sign of Mn(III) in the tubes?
3. What is different about the conditions of this experiment (Method C) compared with the one in method B,which makes its success less likely? What happens in the test-tube which had no manganese(IV) added? Suggest an explanation.

Method C

1. Can you see any sign of Mn(III) in the tubes?
2. What is different about the conditions of this experiment (Method C) compared with the one in Method B which makes its success less likely?
3. What happens in the test-tube which had no manganese (IV) added? Suggest an explanation.
4. $KMnO_4$ is widely used in the laboratory as strong oxidants. The MnO_4^- can be reduced at different pH to Mn(II), Mn(IV) or Mn(VI). With chemical equations only, show the different stages of reduction specifying the pH medium and the electrode potentials.

Experiment 6 Redox Reactions of Iron

Title: Analysis of iron tablets and double salts

Aims: To determine the mass of iron in a given substance (Iron tablet) and to establish the molar ratio of Fe^{2+} ions to Cu^{2+} ions in a supposed double salt solution.

INTRODUCTION

Iron, an essential mineral viable for many functions in the human body undergoes redox reactions. Iron has two oxidation states; +3 and +2. Its redox reaction can be facilitated by the action of an oxidizing agent like Potassium permanganate (Potassium manganate (VII)) which oxidizes Fe^{2+} to Fe^{3+} in an acidified solution. Manganate ions (MnO_4^-) in solution have an intense purple colour whereas Mn^{2+} ions are almost colourless. So, in titrating Fe^{2+} solutions with $KMnO_4$, the colour of the manganate fades off and the endpoint detected by the appearance of a permanganate purple colour. However, if the titrant is the unknown iron (ii) solution, the endpoint of the titration is given by the disappearance of the purple manganate (VII) ion colour. This titration, a redox titration which involves the transfer of electrons between two reacting species (in this case, MnO_4^- and Fe^{2+}) in solution forms the basis for the analytical estimation of iron in any substance. It has been established stoichiometrically that 1 mole of $MnO_4^-{}_{(aq)}$ reacts with 5 moles of $Fe^{2+}{}_{(aq)}$ as shown in the equation below:

$$\mathbf{MnO_4^-\,(aq) + 8H^+\,(aq) + 5Fe^{2+}\,(aq) \rightarrow Mn^{2+}\,(aq) + 4H_2O(l) + 5Fe^{3+}\,(aq)}$$

Part A Reagents: Ferrous sulphate tablets, 1M H_2SO_4, 0.0050M $KMnO_4$ solution

Part B Reagents: standardized 0.02M potassium permanganate, standardized 0.1M sodium thiosulphate, 0.5M potassium iodide, Starch indicator.

Apparatus: Mortar and pestle, digital analytical balance, 100ml volumetric flask with stopper/cork, funnel, conical flask, burette, pipette, Titration apparatus.

Precautionary measures: Acids are corrosive. Ensure all protective gears are worn before commencement of practical sessions.

Part A experimental procedure:

1. Weigh accurately two 'ferrous sulphate' tablets.
2. Grind up the tablets with a little I M sulphuric acid, using a pestle and mortar.
3. Through a funnel, transfer the resulting paste into a 100 cm^3 volumetric flask. Use further small volumes of 1M sulphuric acid to rinse the groundup tablets into the flask. During this process, you must take great care to ensure that all the particles of tablet get into the flask.
4. Add sufficient 1M sulphuric acid to make up the solution to exactly 100 cm'. Stopper the flask and shake it to make sure that all the contents are thoroughly mixed. They will not all be in solution although the Fe^{2+} ions which were present in the tablets will be dissolved.
5. Titrate 10.0 cm^3 portions of the solution with 0.0050 M potassium manganate (VII). The end-point is marked by the first permanent purple colour.
6. Prepare a titration table.

QUESTIONS

Using the data from this experiment, calculate the following:

1. The number of moles of $KMnO_4$ used.
2. The number of moles of iron (II) ion used in the titration.
3. The mass of iron per tablet

Results/Calculations

Part B Experimental Procedure (Analysis of Double Salt)

Prepare the suspected double salt as follows:

1. Add 50 g of hydrated copper (II) sulphate to 100 cm^3 of 2M sulphuric acid and heat to 75 ^oC.
2. Then add 45 g of hydrated iron (II) sulphate and, when dissolved, decant from any residue and chill the solution to 0°.
3. Collect the crystals at the pump, and dry at room temperature.
4. Prepare a standard solution of the salt (roughly 40 g/L) by accurate weighing.
5. Determine the iron (II) by titrating aliquots after acidification with 2M sulphuric acid, with the 0.02 M potassium permanganate.
6. Determine the copper (II) by titrating aliquots after treatment with excess 0.2M potassium iodide, with the 0.1 M sodium thiosulphate.
7. Prepare your titration table for titrations; step 6 and step 7.

QUESTION

Use the equations of the reactions to calculate the molar ratio of iron (II) ion to copper (II) ion:

$5Fe^{2+} + MnO_4^- + 8H^+ \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$

$2Cu^{2+} + 4\ I^- \rightarrow 2Cul + I_2$

$I^{2+}\ 2S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2I^-$

Results/Calculations

Experiment 7 Preparation of a Coordination Compound

Title: Preparation of potassium tris-oxalatoferrate(III)trihydrate ($K_3[Fe(C_2O_4)_3].3H_2O$

Aim: To synthesize a coordination compound

Introduction

A complex or coordination compound therefore consists of a central metal atom surrounded by a set of electron pair donors called ligands. One of the characteristic properties of transition elements is their ability to form complex compounds. This ability is attributed to the presence of vacant (n-1)d orbitals and existence of variable oxidation states. The formation of complex compounds is through a donor-acceptor interaction involving a central metal atom/ion with vacant orbitals and a molecule or ion possessing a lone-pair of electrons. The metal atom/ion with vacant orbitals acts as the acceptor atom, while the molecule or ion with lonepair(s) of electrons serves as the donor. The bond resulting from this type of interaction is called coordinate covalent bond and the compound formed is known as coordination (or complex) compound. Coordination compounds result when a Lewis acid and a Lewis base react together to form a Lewis acid-base adduct (or coordination complex).

Reagents and Apparatus: 250 mL beaker, top-loading balance, iron(III) nitrate nonahydrate, $Fe(NO_3)_3 \cdot 9H_2O$, Bunsen burner. deionized water, potassium hydrogen oxalate monohydrate, $KHC_2O_4 \cdot H_2O$, potassium hydroxide, KOH.

Precautionary measures: Handle all chemicals with care.

Procedure:

- Place a clean, dry 250 mL beaker on the top-loading balance, record its mass and weigh by difference about 8.1 g (record actual mass used) of iron(III) nitrate nonahydrate, $Fe(NO_3)_3 \cdot 9H_2O$, into it.
- Add 20 mL of deionized water and a few boiling granules to the beaker and heat it gently using a Bunsen burner.
- Do not allow the solution to boil. Weigh by difference about 7.7 g (record actual mass used) of potassium hydrogen oxalate monohydrate, $KHC_2O_4 \cdot H_2O$ into a clean, dry weighing vial and add it, with stirring, to the beaker when all the $Fe(NO_3)_3 \cdot 9H_2O$ has dissolved.
- Heat the mixture gently until all the solid dissolves, then, stirring carefully, add 10 mL of 6.0 mol L^{-1} KOH. Stir the mixture until

all of the brown precipitate which forms initially has dissolved. If the brown solid fails to dissolve you have made an error in your weighings and must begin again.

- Filter the hot solution through a glass funnel fitted with a fluted filter paper into a 250 mL conical flask. Slowly, with swirling, add 2 mL of 95% ethanol to the filtrate. Allow the solution to cool without disturbing it for at least 30 minutes to induce crystal growth. If the crystals have not appeared at this time add 3 more mL of 95% ethanol. Shake the flask vigorously and then allow it to stand undisturbed for a further 10 minutes to complete the crystallization.
- When crystallization is complete, collect the product by suction filtration in a Buchner funnel.
- Wash the crystals twice with 10 mL portions of acetone. Break the suction before each addition by pulling the hose off the aspirator or loosening the funnel.
- Warning: acetone and ethanol are flammable solvents. All flames must be extinguished in the lab before removing these solvents from the fumehood.
- Allow the crystals to dry for 5 minutes under suction. Then spread them out on a clean piece of smooth paper to air dry while you prepare the sample vial and label.
- Transfer the crystals to a pre-weighed vial and weigh the vial plus crystals. Record the yield, product formula and your name and bench # on the product label. Immediately wrap the vial in aluminum foil to protect your product from exposure to light and leave the product at your bench to be collected along with your report.

QUESTIONS

1. Calculate molar masses and the number of moles for all reagents and your product.
2. Using the balanced equation for the reaction you carried out, calculate the limiting reagent.
3. Calculate the theoretical yield of your product, $K_3[Fe(C_2O_4)_3]\cdot 3H_2O$.
4. Calculate the percentage yield of the product.

Results/Calculation

Tabulate the results of your experiment using the format below. Show calculations in detail with full identification of the terms, paying attention to units and significant figures.

Measurement	Amount grams in
Mass of $KHC_2O_4 \cdot H_2O$ + Vial + Lid = Mass of emptied vial + Lid = Mass of $KHC_2O_4 \cdot H_2O$ =	
Mass of $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ + vial + Lid = Mass of vial + lid = Mass of $K_3[Fe(C_2O_4)_3] \cdot 3H_2O(s)$ =	

EXPERIMENT 8 Colours Associated With Ligand Changes In Some Coordination Compounds Of Copper(II)

Title: Identification of colours associated with ligand changes in some coordination compounds of Copper(II).

Aim: To identify the colours associated with ligand changes in some coordination compounds of Copper(II).

Introduction

Many transition metal ions are coloured because the energies required for the electronic transitions within their partially-filled d-subshells lie in the visible region of the electromagnetic spectrum. That is, visible light passing through their crystals or solutions is sufficiently energetic to raise a d-electron from the ground state to a higher energy level within the dsubshell. Light passing through the crystal or solution will have certain wavelengths absorbed. The colour you observe will be composed of the remaining visible wavelengths. For example, octahedral copper(II) complexes such as $[Cu(H_2O)_6]^{2+}$ are typically light blue. This means that the blue and possibly some adjacent green and violet wavelengths pass through the crystal while the lower energy red and yellow wavelengths are absorbed. The wavelength of light absorbed by the crystal and hence its colour will vary both with co-ordination number and ligand type, resulting in a change in colour of the solution when a new ligand is added. See pages 69-71 of CHM291 course material for more details.

Reagents and Apparatus: Ammonia solution (6M), HCl (6M), Hexaaquacopper(II) solution, test tubes.

Precaution: Endeavor to wear all laboratory protective gears. Some of the reagents you are to work with are corrosive.

Procedure

- Take 1 cm^3 of the $[Cu(H_2O)_6]^{2+}(aq)$ solution in a large test tube. Add one drop of 6 mol L^{-1} ammonia solution and note any change. Continue addition of the ammonia dropwise noting further changes, until any precipitate that forms finally dissolve. Record your observations.
- To the same solution add 6 mol L^{-1} HCl dropwise, mixing at each addition, until no further changes occur. Record your observations in this format below.

$[Cu(H_2O)_6]^{2+}(aq)$	1 drop $NH_3(aq)$	Excess $NH_3(aq)$	Excess HCl(aq)

QUESTIONS

1. Solutions of copper(II) sulphate and potassium iodide react to form a white precipitate of copper(I) iodide and a brown solution of iodine.

a. Write the ionic equation for this reaction.

b. Explain why this is classed as a redox reaction.

2. Suggest why the copper (I) iodide which forms is white, while copper(II) compounds are coloured.

3. Write net ionic equations to represent the reactions you have observed in this experiment.

RESULTS

EXPERIMENT 9 Preparation of Chloropentaamine Cobalt (III) Chloride, $[Co(NH_3)_5Cl]Cl_2$

Title: Synthesis of Chloropentaamine Cobalt (III) Chloride, $[Co(NH_3)_5Cl]Cl_2$

Aim: To prepare chloropentaaminecobalt(III)chloride, $[Co(NH_3)_5Cl]Cl_2$.

Introduction

Cobalt has two relatively stable oxidation states, the 2+ and 3+ states. The 2+ state is kinetically labile with respect to ligand substitution, which means that ligand exchange for Co(II) is rapid. The 3+ state is inert with respect to ligand substitution. Therefore, complexes of Co(III) are easy to handle and can be dissolved in water without a rapid change in composition. We take advantage of these properties to synthesize Co(III) compounds. A solution of Co(II) is prepared with a high concentration of the ions or molecules that we wish to introduce as ligands. The solution is then treated with an oxidizing agent, such as O_2 or H_2O_2, to convert the Co(II) to Co(III). ***See pages 77-79 of CHM291*** Course material for more on the principles guiding this experiment.

Reagents and Apparatus: cobalt chloride 6-hydrate, ammonium chloride, concentrated ammonia, 30% hydrogen peroxide, concentrated HCl, methanol, Erlenmeyer flask, beaker, heating device, fumehood, ice bath, funnel.

Precaution: Handle all reagents with care.

Procedure:

Synthesis of $[Co(NH_3)_5Cl]Cl_2$

- Accurately weigh 4.0 g of powdered cobalt chloride 6-hydrate in a 125mL Erlenmeyer flask.
- Working in the hood, dissolve 2.0 g of ammonium chloride in 12mL of concentrated ammonia in a beaker. Add the mixture to the flask of $CoCl_2$ and swirl to dissolve. Some brown and/or purple solid will remain in suspension. (see equation 1.)
- Add 4 mL of 30% hydrogen peroxide solution, in 1 mL increments. Swirl the flask between each addition to stir the mixture and allow the effervescence to cease. When all the hydrogen peroxide has been added, allow 5 minutes for the reaction to be completed. (see equation 2.)

- Then slowly, with swirling, add 12 mL of concentrated hydrochloric acid. Considerable heat will be generated by this reaction.
- Heat the mixture (in a hood) at 85°C for 20 minutes, using a 400 mL beaker on a hotplate as a heating bath. Do not allow the temperature to rise above 90°C. (see equations 3., 4.)
- Allow the mixture to cool to near room temperature. Once a considerable amount of solid has formed, the cooling can be speeded by using an ice bath. (see equation 5.)

Isolation of the $[Co(NH_3)_5Cl]Cl_2$

- Assemble the filter flask, trap and filter funnel.
- Pour the solution through the funnel until only a small amount remains. Then swirl the flask to bring the solid into suspension and quickly pour all of the contents of the flask into the funnel. Allow the vacuum to draw the solution away from the precipitate.
- Use 10 mL of ice-cold de-ionized water to transfer any solid remaining in the flask into the funnel.

The washing technique

- Add just enough ice-cold water to the funnel to cover the precipitate, leave to sit for a few seconds to wet the solid thoroughly, and then draw the wash solution through into the filter flask. Washing once with ice-cold water is sufficient.
- Turn off the vacuum, and add 20 mL of ice cold methanol to the product on the filter paper. Let this set for a few seconds, and apply the vacuum. When the methanol has been drawn through the solid product, turn the vacuum off, and rinse again with a 20 mL portion of methanol.
- After this second rinsing with methanol, leave the vacuum on, and draw air through the solid until it is dry.
- Place the solid product in a beaker, and store for a week.
- Accurately weigh the purified $[Co(NH_3)_5Cl]Cl_2$ that has been in your storage drawer for a week. Use the mass obtained to calculate %yield.

Questions

1. Calculate the %yield of your product.
2. Determine the actual mass of cobalt chloride hexahydrate you started with and the mass of the pure product you isolated.
3. Calculate the mass percent Co in your compound. Compare to the expected value by calculating % absolute error.

Results