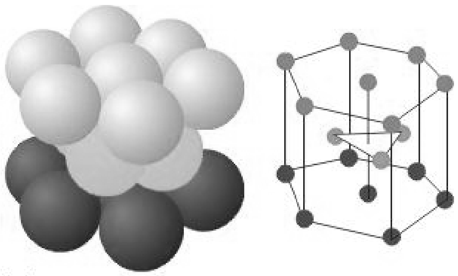
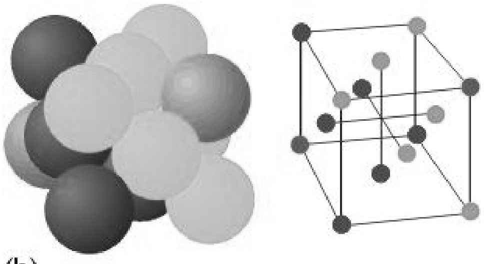
Laboratory manual
Of
PRACTICAL INORGANIC II CHEMISTRY



(a)

HCP



(b)

CCP

By:

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FACULTY OF MATHEMATICS AND NATURAL SCIENCES
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2011

PREFACE

The manual of Inorganic II laboratory work is a laboratory guideline to the students of Chemistry Department, Mathematics and Natural Sciences Faculty, Yogyakarta State University, in Inorganic II course. According to the material of Inorganic II course, the laboratory activities are directed to understand the model of ionic compound close packing, reduction-oxidation reaction of several metals, improve the double salt preparation and re-crystallization skills as the basic for advance laboratory activities.

Because of the restrictiveness of equipment and chemicals, some of practical subject can not be done. Of course, this laboratory manual does not support optimally to the Inorganic II course.

The English edition of Inorganic II laboratory work manual is the same in contents with Indonesian edition. Revision done by simplifies the language and the direction also separates the manual and the worksheet.

It is hoped, this manual may be a guideline to improve student laboratory skill especially in inorganic laboratory work.

Yogyakarta, August 2011

M. Pranjoto Utomo

RULES OF INORGANIC II LABORATORY WORK

A. Laboratory work presence

1. Every student must come on time and fill the presence book.
2. Students, who come over than 15 minutes from the laboratory work schedule, may follow the laboratory work if only permitted by the lecturer.
3. All students must participate in every laboratory work activity.
4. The permitted practical tags are twice.

B. Carrying Out of Laboratory Work

1. Laboratory work of Inorganic II is group laboratory activity. Each group consists of 4 students. Each group member has individual responsibility to the result of laboratory work (report).
2. Every student has to wear white-laboratory coat, bring a napkin and dropper pipette.

C. Laboratory Work Report

1. Every student must compose individual report on the student worksheet.
2. Student worksheet must be collected to assistant or lecturer at the same day with laboratory work day.

LABORATORY PRECAUTIONS

A. Inside the Laboratory

1. Do not eat, drink beverages or chew gum in the laboratory. Do not use laboratory glassware as containers for food or beverages
2. Wear safety goggles and aprons
3. Always keep the working area clean and orderly
4. Know the locations and operating procedures of all safety equipment.
5. Notify the instructor immediately of any unsafe condition you observe

B. Handling Chemicals

1. All chemicals in the laboratory are to be considered dangerous. Do not touch, taste or smell any chemical unless specifically instructed to do so
2. Check the label on chemical bottles twice before removing any of the contents.
3. Never return unused chemicals to their original containers.
4. Acid must be handled with extreme care. ALWAYS ADD ACID SLOWLY TO WATER.
5. Handle flammable hazardous liquids over a pan to contain spills. Never dispense flammable liquids anywhere near an open flame or source of heat.

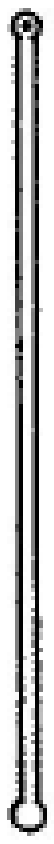
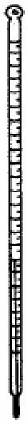
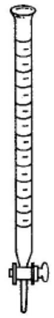
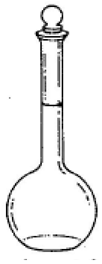
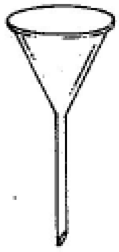
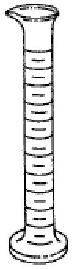
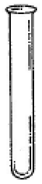
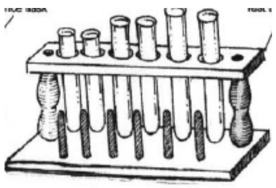
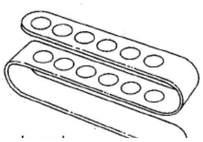
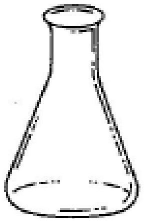
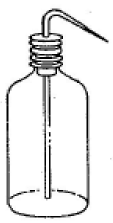
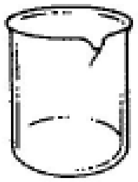
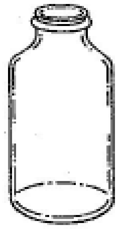
C. Handling glassware and Equipment

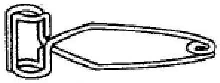
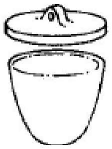
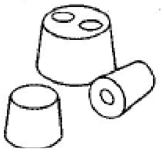
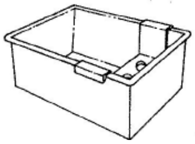
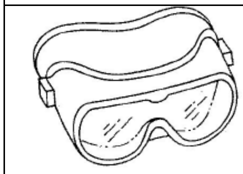
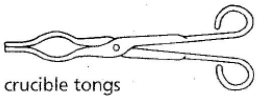
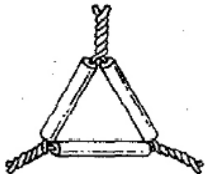
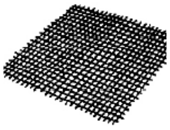
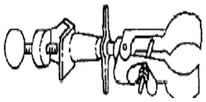
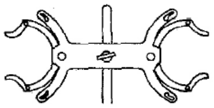
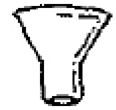
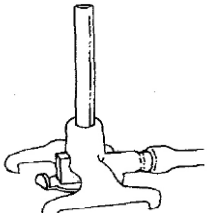
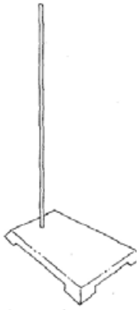
1. Always lubricate glassware (tubing, thistle tubes, thermometers, etc.) before attempting to insert it in a stopper.
2. When removing an electrical plug from its socket, grasp the plug, not the electrical cord. Keep your hands dry when working with electricity.
3. Do not immerse hot glassware in cold water, it may shatter.
4. Report damage electrical equipment immediately.

D. Heating Substances

1. TURN OFF THE GAS AT GAS OUTLET VALVE after using.
2. Never leave a lit burner unattended. Never leave anything that is being heated or is visibly reacting unattended.
3. Use tongs or heat-protective gloves when holding or touching heated apparatus.

LABORATORY EQUIPMENTS

 Gas collecting tube	 Measuring pipette	 Stirring rod	 Thermometer
 Glass-stoppered burette	 Volumetric flask	 funnel	 Graduated cylinder
 Test tube	 Test tube rack	 Spot plate	 s-shaped test tube rack
 Forceps	 Dropper pipette	 spatula	 Triangular file
 Erlenmeyer flask	 Plastic wash bottle	 Beaker	 Gas-collecting bottle

 Test tube brush	 Pinch clamp	 Test tube holder	 Watch glass
 Evaporating dish	 Crucible and cover	 Rubber stoppers	 Pneumatic trough
 Safety goggles	 Crucible tongs	 Clay triangle	 Wire gauze
 Utility clamp	 Iron ring	 Burette clamp	 Wing tip
 Burner	 Ring stand		

CONTENTS

Page

PREFACE i

RULES OF INORGANIC II LABORATORY WORK ii

LABORATORY PRECAUTIONS iii

LABORATORY EQUIPMENTS iv

CONTENTS v

EXPERIMENT 1. CLOSE PACKING GEOMETRY IN SOLID 1

EXPERIMENT 2. QUALITATIVE INORGANIC REACTION …… 10

EXPERIMENT 3. REDUCTION-OXIDATION REACTION ……… 12

EXPERIMENT 4. REDUCTION-OXIDATION REACTION, THE INFLUENCES OF ACID AND BASE TO METALS …………… 15

EXPERIMENT 5. ELECTROCHEMISTRY CELL AND ELECTRODE POTENTIAL 18

EXPERIMENT 6. CORROSION OF METAL (1) ………………... 20

EXPERIMENT 7. CORROSION OF METAL (2) ………………… 23

EXPERIMENT 8. CORROSION OF METAL (3) ………………… 24

EXPERIMENT 9. PREPARATION OF POTASSIUM-CHROMIUM ALUM, $KCr(SO_4)_2 \cdot 12H_2O$ ……………………………………....... 26

EXPERIMENT10. PREPARATION OF POTASSIUM-ALUMINUM ALUM, $KAl(SO_4)_2 \cdot 12H_2O$ …………………………… 28

EXPERIMENT 11. PREPARATION OF COORDINATION COMPOUND, $[Ni(NH_3)_6]I_2$ …………………………………………….. 30

EXPERIMENT 12. PURIFICATION OF KITCHEN SALT BY RE-CRYSTALLIZATION METHOD ………………………………….. 32

REFERENCES 34

EXPERIMENT 1

CLOSE PACKING GEOMETRY IN SOLID

Purpose

The main purpose of this activity is to study close packing geometry i.e. to illustrate the structure of ionic solid.

Introduction

Ionic solid can be viewed as a close packing of metal atoms. The arrangement of atoms, molecules, or ions in a regularly and repeatedly pattern is known as a lattice space. The arrangement's nature is determined by three factors:

1) relative shape and size of atom, molecule or ion
2) nature and relative strength of chemical bonding
3) thermal energy of the system

Two applied models of the arrangement are **hole filling model** (balls represent atoms or ions that are packed close one to others) and **ball and stick expanding model**, where the balls are separated by the stick connectors. In ball and stick crystal structure model, stick connectors represent covalent bonds (such as bonds in diamond) or ionic bonds (such as bonds in NaCl). On the other hand, stick connector illustrate crystal lattice of a solid.

In this activity, you have to arrange the balls, observe and fill the observation data on your work sheet to understand close packing geometry in solid.

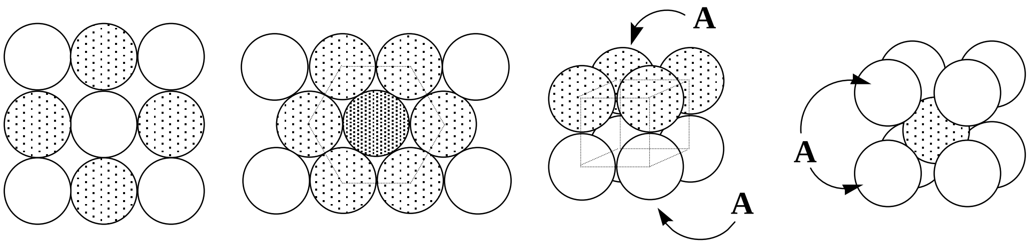
Materials

- ping-pong balls (at least contain of two colors)
- marbles
- buckshot
- electrical glue gun

Ball Packing Efficiency

Use amount of ping-pong and other smaller balls to arrange various arrangement patterns and to complete the following tasks.

1. Arrange the balls in side-by-side arrangement (Figure 1a) and in closest arrangement (Figure 1b).



(a) (b) (c) (d)

Figure 1.1 Packing models of (a) side-by-side layer, (b) hexagon layer, (c) simple cubic layer, (d) body centered cubic

Determine the maximum amount of balls that can touch another one ball (center- ball) in the same layer for each arrangement.

Answer: ………………….. balls for (a) and …………………….. balls for (b)

Determine which is the closer packing, (a) or (b).

Answer: ……………………………………………………………………………...

In this case, geometry structure in (a) is not a close packing, whereas (b) is a close packing. The packing (b) often called as hexagon layer (pay attention to the hexagon illustrated by dots)

2. If we add the second, third layer, and so on, to the arrangement model (a) so that the balls of second and third layer lie above the first layer, we get the packing pattern of layer A, A, A.

   Determine the amount of balls that touch every another ball in layer A, A, A.

   Answer: ……………… balls

   The numeric is called as **coordination number** (amount of balls, atoms, ions that touch one center-ball, atom or ion). To get this geometry structure, can be represented by two layers: A, A, where each layer contains four balls. What geometry is resulted? (See Figure 1c)

   Answer: ……………………………………………………………………………...

3. If the hole between the two A, A layers filled by one more ball (in the same size) so that the filled-ball touch all other balls, the two layer will expand. The resulted-geometry from those expanding is called .... (Figure 1d) which has coordination number : ....

Determine which is the closer packing, (c) or (d).

Answer: …………………………………………………………………………...

The two resulted-geometry structures from the expanding of (a) model are not a close packing or closest packing, because these arrangements still can be changed to be a closer one.

Hexagon and cubic close packing

Two possibilities arrangement of closest packing of same size balls are **hexagon closest packing (hcp)** and **cubic closest packing (ccp**). The last arrangement also called as face centered **cubic closest packing (fcc)**. Both arrangements use the hexagon layer (Figure 1b) and represent the most effective way to arrange balls to fill the hole/space optimally. To simplify, one layer represented by 3-balls or 7-balls pattern.

To observe hexagon closest packing (hcp), firstly, put the 3-balls-triangular pattern (layer A) on the desk. Secondly, put the 7-balls–hexagon pattern (layer B) above it so that the balls fit into the hole of layer A. Finally put another 3-balls-triangular pattern so that the fit into fill the hole of layer B and lie above of those layer A (Figure 2a). The expanding of this pattern produces the hexagon closest packing geometry (hcp). The hexagon closest packing follows the pattern of A, B, ..., ..., ..., ...

Note: the first layer always called as layer A. The second layer is called layer B if the balls fit into the hole or do not lie above to those in layer A. The third layer is called layer C if the balls fit into the hole to those in layer B and do not live above to those in layer A.

Determine the maximum balls that touch the center-ball in the hcp pattern.

Answer: ……………… balls, consists of ……………….. balls in the same layer and ……………………….. balls in above and under those layer. This numeric called as coordination number.

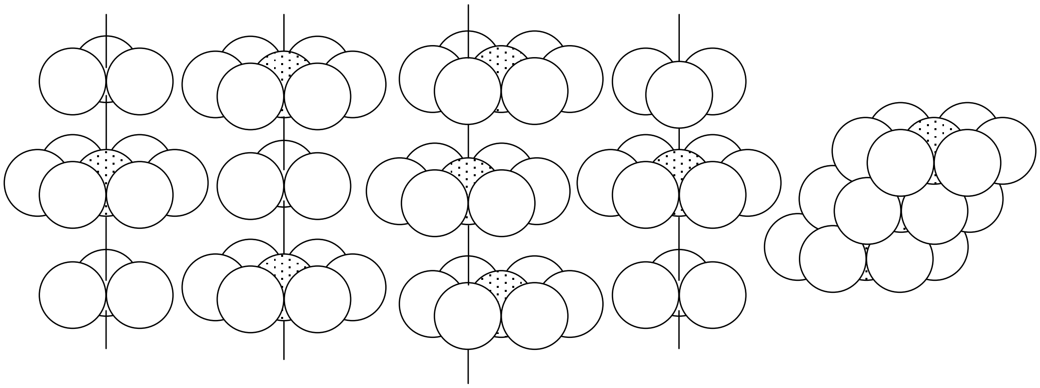
Analogically, arrange another hcp pattern by using 7-balls–hexagon, 3-balls-triangular and 7-balls-hexagon layers. Firstly, put the7-balls-hexagon layer on the desk (layer A). Secondly, put the 3-balls-triangular layer (layer B) above it so that the balls fit into the hole of layer A. Finally put another 7-balls–hexagon layer so that the balls fit into the holes of layer B and lie above of layer A's balls (Figure 2b).

Determine the coordination number of this pattern.

Answer: ………………

Is the coordination number of this pattern still the same with the previous one?

Answer: (Yes / No)*



(a) (b) (c) (d) (e)

Figure 1.2. Various possibilities of closest packing

Repeat the packing models of hcp, at least, consists of three layers, where each layer consists of 7-balls-hexagon layer. Put one ball as center-ball, and then count other balls that touch it (Figure 2c). Determine the coordination number of this pattern.

Answer: ………………

Hold this hcp packing pattern and direct it to the light source. Pay attention to the route of the light on the hcp packing. What do you get?

Answer: …………………………………………………………………………………….

Alternatively, put the hcp packing on the desk and put the wire through the hcp packing via the holes among the balls. Pay attention to the route of the wire in the hole of hcp packing. What do you get?

Answer: …………………………………………………………………………………….

To observe the cubic closest packing (ccp) or face centered cubic (fcc), arrange the hcp packing (Figure 2d). Hold the 3-balls-triangular layer on the top and turn it 60^o clockwise. Now, the balls in the 3-balls-triangular layer on the top do not lie above to those in the first layer (the 3-balls-triangular layer at the bottom), but fit into the holes. The expanding of this pattern yield to the face centered cubic closest packing or face centered cubic (fcc). The fcc follows the pattern of A, B, ..., ..., ..., ...

Determine the maximum balls that touch the centered-ball in the fcc pattern.

Answer: ……………… balls, consists of ……………….. balls in the same layer and ……………………….. balls in above and under those layer. This numeric called as coordination number.

Analogically, arrange the fcc pattern with three layers of 7-balls–hexagon layer (Figure 2e).

Determine the coordination number of this pattern.

Answer: ………………

Is the coordination number of this pattern still the same with the previous one?

Answer: (Yes / No)*

Hold this fcc packing pattern and direct it to the light source. Pay attention to the route of the light on the fcc packing. What do you get?

Answer: …………………………………………………………………………….

Alternatively, put the fcc packing on the desk and put the wire through the fcc packing via the holes among the balls. Pay attention to the route of the wire in the hole of fcc packing. What do you get?

Answer: …………………………………………………………………………….

The terminology of closest packing of face centered cubic (fcc) in the previous activity is often confusing, because the fcc geometry is invisible. To overcome that problem, see Figure 3. The structure is represented by 14 balls consisted of 8 balls at the corner of cubic and 6 balls at the center of cubic face. Investigate the 14 balls carefully.

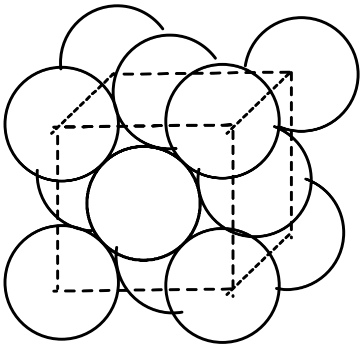


Figure 1.3. Face center cubic

Determine the amount of balls that touch center-ball on each cubic face.

Answer: ………………….. balls

For each cubic face, is it a closest packing layer?

Answer: …………………..

In order to get closest packing layer, from what direction (axis or diagonal) of the structure must be viewed.

Answer: ……………………………………………………………………………..

(Shade the balls in the same layer and label it with layer A, B or C for Figure 3).

Tetrahedron Geometry (in Cubic)

Place 3-triangular-balls pattern on the desk and put a ping-pong ball on its hole. The resulted hole from the compilation of four balls called as **tetrahedral hole**, the four balls occupy the corner of tetrahedron structure.

Put a smaller ball in the hole so that touch all of four other balls. Shake the pattern. Pay attention to the sound. No sound means the smaller ball fit the hole precisely. If the smaller ball does not fit the hole precisely, it will make sound. Therefore, you have to change the smaller ball with another ball. Then measure the diameter of each ball, big and small balls and then determine the ratio. (Note: r_+ represents the cation radius and r_- represents the anion radius. Remember: cation radius is always shorter than anion radius)

Diameter of small ball = ..., so the radius (r_+) = ...

Diameter of ping-pong ball = ..., so the radius (r_-) = ...

Radius ratio $\frac{r_+}{r_-}$ = ………………….., or $\frac{r_-}{r_+}$ = ...

The truth of resulted-ratio could be tested mathematically according to Figure 1.4a (structure of body centered cubic with side length a)

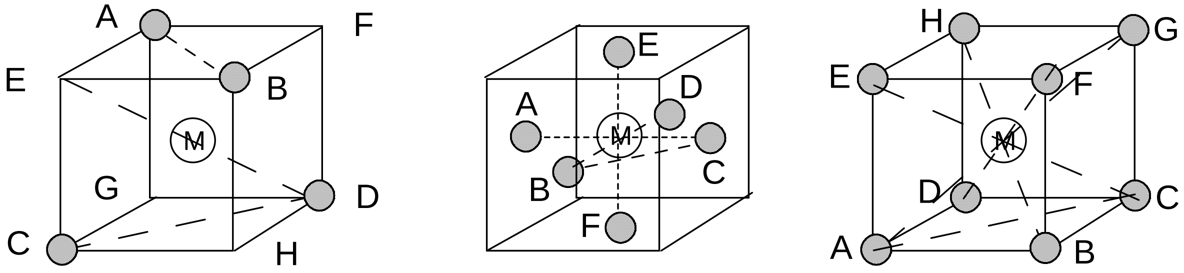


Figure 1.4. Tetrahedron (a), octahedron (b), and body centered cubic (c) geometries in cubic perspective

Imagine, ball M that fit the tetrahedron hole touch 4 ball of ABCD. Determine the radius ratio of ping-pong ball (A=B=C=D) with hole ball (M).

Hint: √3 = 1.732 and √2 = 1.414

i) State the ball radius in the term of r_+ or r_- and the length in a.

ii) State the face diagonal, CD, and body diagonal, ED, (and then MD) in the term of r_+ or r_- and a.

iii) Obtain the value of r_+ and r_- in the term of a, then calculate its ratio

Answer: ……………………………………………………………………………………..

……………………………………………………………………………………..

Therefore the radius ratio:

$$\frac{r_+}{r_-} = \text{..................}, \text{ OR } \quad \frac{r_-}{r_+} = \text{................}$$

(Compare with your calculation)

Octahedron Geometry (in Face Centered Cubic)

Place 3-triangular-balls pattern on a desk, and put the second 3-triangular-balls pattern on it (the second pattern precisely lies above on the first one). Turn the second pattern 60^o clockwise. The resulted-hole from the pattern of those six balls is called **octahedral hole** (the center of each ball that build the hole, occupies the corner of octahedron geometry).

Put a smaller ball into the hole so that touch all of 6 other balls. Shake the pattern. No sound means the smaller ball fit the hole precisely. If the smaller ball does not fit the hole precisely, it will make sound. Therefore, you have to change the smaller ball with another ball. Then measure the diameter of each ball, big and small balls and then determine the ratio. (Note: r_+ represents the cation radius and r_- represents the anion radius. Cation radius shorter than anion radius)

Diameter of small ball = ..., so the radius (r_+) = ...

Diameter of ping-pong ball = ..., so the radius (r_-) = ...

Radius ratio $\frac{r_+}{r_-}$ = ………………….., or $\frac{r_-}{r_+}$ = ...

The truth of resulted-ratio could be tested mathematically according to Figure 4b (structure of body centered cubic with side length *a*). The six corner of geometry octahedron represent the center of face centered of cubic.

Imagine, ball M that fit the octahedron hole touch 6 ball of ABCDEF. Determine the radius ratio of ping-pong ball to hole-ball (ball that fit the hole).

Hint: $\sqrt{2} = 1.414$

i) State the ball radius in the term of r_+ or r_- and the length in a.

ii) Determine the length of BM (= MC) and BC in the term of r_+ or r_- and a.

iii) Obtain the value of r_+ and r_- in the term of a, then calculate its ratio

Answer: ………………………………………………………………………………..

………………………………………………………………………………..

Therefore the radius ratio:

$\frac{r_+}{r_-}$ = ..., OR $\frac{r_-}{r_+}$ = ...

(Compare with your calculation)

Which hole is bigger, tetrahedral or octahedral hole?

Answer: ………………………………………………………………………………..

Body Centered Cubic Geometry

Place the four-ball pattern in side-by-side pattern on the desk. Put the second four-ball pattern on the first one (the second pattern precisely lie above on the first one) to get simple cubic geometry. The packing of those eight balls results geometry of **body centered cubic**.

Put a smaller ball into the hole so that touch all of eight other balls. Shake the pattern. Pay attention to the sound. No sound means the smaller ball fit the hole precisely. If the smaller ball does not fit the hole precisely, it will make sound. Therefore, you have to change the smaller ball with another ball. Then measure the diameter of each ball, big and small balls and then determine the ratio. (Note: r_+ represents the cation radius and r_- represents the anion radius. Remember: cation radius shorter than anion radius)

Diameter of small ball = ..., so the radius (r_+) = ...

Diameter of ping-pong ball = ..., so the radius (r_-) = ...

Radius ratio $\frac{r_+}{r_-}$ = ………………….., or $\frac{r_-}{r_+}$ = ...

The truth of resulted-ratio could be tested mathematically according to Figure 4c (structure of body centered cubic with side length *a*). The six corner of geometry octahedron represent the center of face centered of cubic.

Imagine, ball M that fit the octahedron hole touch 8 ball of ABCD-EFGH. Determine the radius ratio of ping-pong ball to hole-ball (ball that fit the hole).

Hint: $\sqrt{3} = 1.732$

i) State the ball radius in the term of r_+ or r_- and the length in a.

ii) Determine the length of DH and HM (= DM) in the term of r_+ or r_- and a.

iii) Obtain the value of r_+ and r_- in the term of a, then calculate its ratio

Answer: ……………………………………………………………………………..

……………………………………………………………………………..

Therefore the radius ratio:

$\frac{r_+}{r_-}$ = ..., OR $\frac{r_-}{r_+}$ = ...

(Compare with your calculation)

Which one is the bigger hole, tetrahedral, octahedral or simple cubic?

Answer: ……………………………………………………………………………..

Identification of Tetrahedral and Octahedral Hole

There are two types of hole in closest packing, **tetrahedral and octahedral hole.** The amount of tetrahedral hole is twice as octahedral hole. Remember the second layer balls just fit a half of the first layer holes. The bottom part of first layer also gives the same amount of holes. The first layer holes occupied by second layer balls yield to tetrahedral holes, whereas unoccupied holes yield to octahedral holes.

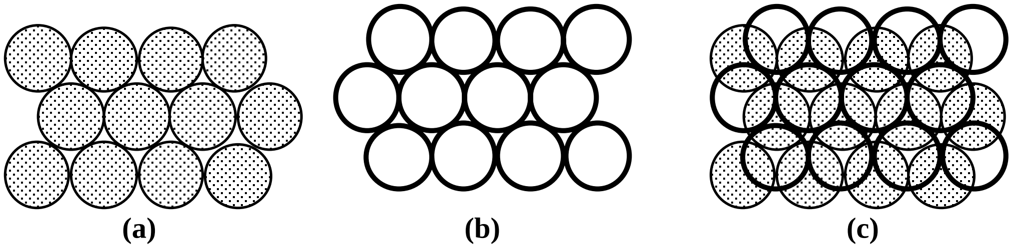


Figure 1.5 Models of hexagon layer packing (a,b), and layer A, B closest packing (c)

Arrange a 12-balls-hexagon layer, (Figure 5a) and there are 12 holes. Arrange another 12-balls-hexagon layer in opposite direction (Figure 5b). Put layer (b) on layer (a) according to A, B pattern. Balls of layer (b) fit the holes of layer (a). Identify or mark the type and the amount of formed holes between two layers (for example, red for tetrahedral holes and blue for octahedral holes). Identify the tetrahedral and octahedral holes by directing the two layers (Figure 5c) to light source. Light can penetrate TETRAHEDRAL/OCTAHEDRAL*) holes, but cannot penetrate TETRAHEDRAL/OCTAHEDRAL*) holes. How many tetrahedral and octahedral holes are there?

Answer: ……………………………………………………………………………..

Therefore, between two hexagons closest packing, the amount of tetrahedral hole is twice as octahedral hole. Ionic solid, anions (bigger size) often adopt closest packing, *fcc* or *hcp*, and cations fit those holes completely or partially depend on the stoichiometry of the species.

EXPERIMENT 2

INORGANIC QUALITATIVE REACTION

Purpose

To study the reaction of metal ions with hydroxide ion and ammonia

Introduction

Metal cations react characteristically with base in the term of form and nature of product solubility, especially in water. Adding of (strong) base excessively often give more influences, according to the characteristic of cation in the term of amphoterism. Besides, transition cations often form complex compounds with ammonia. By this, identification of cation with strong base (NaOH) and weak base (NH_3) is an interesting activity in inorganic qualitative reaction.

Materials

- Semi micro test tube
- 2 M NaOH solution
- Dropper bottle
- 2M NH_3 / NH_4Cl solution
- Centrifuge
-
- 0.5 M NaOH solution
- 2 M NH_3 solution
- Mg^{2+}, Ba^{2+}, Al^{3+}, Cr^{2+} 0.1 M solution
- Fe^{3+}, Mn^{2+}, Pb^{2+}, Cu^{2+}, Ni^{2+}, Ag^{2+}, Zn^{2+}
- Dropper pipette

Procedure

1. Put nitrate cations (as mentioned above), 0.5M NaOH solution, 2 M NH_3 solution and NaOH 0.5 M solution in labeled-dropper bottle. Those solutions used as mother liquid.
2. Add drop to drop (about 5 drops) 0.5 NaOH M solution into 0.1M $Mg(NO_3)_2$ solution. Volume of 0.5 M NaOH solution must not exceed of 1 mL.
3. Divide the resulted precipitation into 2 parts, and put each to semi micro test tube. Centrifuge for 1 minute. Take the supernatant with dropper pipette
   a. In tube 1, add 2 M NaOH solution (volume must not exceed of 1 mL) into the resulted precipitate.

   b. In tube 2, add 2 M NH_3 (solution volume must not exceed of 1 mL) into the resulted precipitate.

4. Repeat step 2 to 3 for the 0.1 M solution of Ba^{2+}, Al^{3+}, Cr^{3+}, Fe^{3+}, Mn^{2+}, Pb^{2+}, Cu^{2+}, Ni^{2+}, Ag^{+} dan Zn^{2+}. Record the observation in the table on the worksheet
5. Identify which cations that form precipitation on the adding of NaOH
6. Identify which cations that form precipitation on the adding of NaOH
   a. Dissolves in the adding of excess NaOH
   b. Dissolves in the adding of excess ammonia
7. a. Add 0.1 M $Al(NO_3)_3$ solution slowly into 1 mL of 2 M NaOH. Record your observation.
   b. Add 0.1 M $Fe(NO_3)_3$ solution slowly into 1 mL of 2 M NaOH. Record your observation.
   c. Repeat the activities of (a) and (b) in opposite steps of reactant adding. Record your observation and explain.

EXPERIMENT 3

OXIDATION-REDUCTION REACTION (1)

Purpose

To study oxidation-reduction reaction on several compounds

Introduction

Oxidation is the releasing of electron and reduction is the capturing of electron. Oxidation and reduction reaction always a pair reaction, that transfer electron occurred. Oxidizing agent is a species that cause other species oxidized and it self reduced. Reducing agent is a species that cause other species reduced and it self oxidized.

In this experiment, several general oxidation-reduction reactions studied.

Table 3.1 Several Oxidizing and Reducing Agents

Oxidized form	Reduced form	Differentiation test or reaction character
MnO_4^- purple	Mn^{2+} uncolored	The color change, from purple to uncolored
$Cr_2O_7^{2-}$ orange	Cr^{3+} green	The color change, from orange to green
I_2 brown	I^- uncolored	The color change, from brown to uncolored Indicator sensitivity: starch solution change to blue in the presence of I_2 . If the blue color is not sharp, add 5 drops of $CHCl_3$, blue color will form on chloroform layer at the bottom of tube.
Fe^{3+} brown	Fe^{2+} green	The color changing of the two ions is difficult to be observed Test 1: add 1 drop 0.1 M KSCN solution to make red blood color of $Fe(SCN)^{2+}$ for Fe^{3+} ion Test 2: Add 4 drops 2 M solution of NaOH. The precipitate of $Fe(OH)_2$ is green, and $Fe(OH)_3$ is brown.
Sn^{4+}	Sn^{2+}	No color changing Test: add 1 drop of 0.25 M $HgCl_2$ solution. White to purple precipitate of Hg_2Cl_2 and Hg formed in the presence of Sn^{2+} ion.
SO_4^{2-}	SO_3^{2-}	No color changing Test: add 2 drops of 0.1 M $BaCl_2$ solution and several drops of 2M HCl solution. White precipitate of $BaSO_4$ formed, whereas $BaSO_3$*) dissolves in the addition of HCl.

*) Generally, sulfite (SO_3^{2-}) ion is contaminated with sulfate because sulfite easily oxidized by dissolved oxygen.

Several compounds oxidized and reduced on a reaction. The aims of this activity are to study oxidation-reduction reaction on several compounds and to test (special test) on it as seen on Table 2.1.

Each activity is conducted by using two reaction tubes. First tube for “test tube”, labeled as T and second one for “blank solution tube”, labeled as B. Blank solution is a solution contains all reagents (solvents) except compound that will be tested or studied. Use water or aquadest to replace the tested compound (in same volume).

Example:

Contents of tube T	Contents of tube B
5 drops of 0.1 M Fe^{2+} solution	5 drops of 0.1 M Fe^{2+} solution
2 drops of 2.5 M H_2SO_4 solution	2 drops of .5 M H_2SO_4 solution
5 drops of 3% H_2O_2 solution	5 drops of aquadest

The purpose of the blank solution making is to know the condition before and after reaction. Do the test to both tube T and tube B in order to know every change in the reaction clearly. In some cases, two blank solutions are needed. First blank solution for solvent one, and second solution for the other.

Materials

- Test tube
- Semi micro test tube
- Dropper pipette
- Test tube shelf
- $H_2C_2O_4$ (0.1 M)
- $Fe(NH_4)_2(SO_4)_2$ (0.1 M) (must be in fresh condition)
- H_2O_2 (3%)
- ($SnCl_2$) (0.1 M)
- HCl (5 M)
- MnO_4 (0.02 M)
- $K_2Cr_2O_7$ (0.02 M)
- H_2SO_4 (5 M),
- KSCN (0.05M)
- KI (0.1 M)

Procedure

Do the experiments according to the procedure on Table 2.2 in worksheet and refer to Table 2.1.

1. Preparation of fresh solution of Fe^{2+}

   a. Pour 2 gram of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ crystal into beaker glass 250 mL.
   b. Add 5 mL of 5 M H_2SO_4 solution and 50 mL of aquadest to beaker glass in point a.
   c. Mix the mixture until all crystals dissolved (heat the mixture if necessary).
2. For each reaction in Table 2.2, use 5 drops reagent (reactant) for tube T and tube B, as shown in Table 2.2. Prepare blank solution for each system and check it with the assistant before doing the experiment.
3. If there is no reaction at room temperature, heat the solution by placing the test tube in hot water.
4. If it necessary, appropriate tests must be conducted to both tube T and B.

Notes:

a. Conduct the redox tests to both tube T and B (see Table 2.1).
b. Use fresh solution of $Fe(NH_4)_2(SO_4)_2$ to get Fe^{2+} ion.
c. The KI solution must be uncolored/in fresh condition, (replace the KI solution if the color is yellow).

EXPERIMENT 4
OXIDATION-REDUCTION REACTION (2)
THE INFLUENCES OF ACID AND BASE TO METALS

Purpose

To study the influences of acid and base to metals

Introduction

- **Acid**

Acid is species that can donate proton (proton donor). Strong acid donate its entire proton. Mineral acids such as HCl, HNO_3 dan H_3PO_4 are strong acids. Acid can act as oxidizing agent. H^+ is oxidizing agent (and reduced to H_2). Table 4.1 shows the influences of several acids to metals.

- **Metal**

Metal tends to form cation (positive ion) whether in solution or compound. Solid metal reacts with acid to produce cation and release electron(s).

$$M\,(s) \rightarrow M^{n+}\,(aq) + n\,e$$

The released electron is captured by oxidizing agent (H^+, NO_3^-, SO_4^{2-}) and gas released. The series of metals listed in Table 4.1 known as **activity series**. Left (above) series elements will reduce right (below) elements. Therefore, potassium (K) is the strongest reducer that can replace all right (below) metals in **activity series**, according to the reaction:

$$n\,K\,(s) + M^{n+}\,(aq) \rightarrow n\,K^{+}\,(aq) + M\,(s)$$

Vice versa, all left (above) metal of hydrogen, can replace acid (for example replace with H^+) and all right (below) metals of hydrogen will react with oxidizing acids.

- **Alkali**

Alkali refers to strong base with the formula of $M(OH)_n$, where M is alkali metals (such as Na, K) or alkaline earth metals (such as Ca, Mg) and the value of n is 1 (for alkali) or 2 (for alkaline earth). Several metals react with alkali solution. The alkali reaction shows the “semi metal” nature of the elements. Semi metal nature is a combination of metal and non-metal nature. In some cases, metal oxide found react with acid and base. Those metal oxides called as amphoteric oxides. Elements that have amphoteric oxides are also able to react with alkali and acid to produce H_2 gas.

Zinc also reacts with acid and base in the same way, but slow and relatively difficult to observe the occurrence of H_2 gas. To prove that zinc has already dissolved, add sulfide ion to form white precipitate of zinc sulfide.

Table 4.1 The Influence of Acids to Metals

Metals	Acid replacement		Oxidizing acids		
	HCl dilute / concentrated (up to 10 M)	H_2SO_4 dilute	H_2SO_4 concentrated (± 18 M)	HNO_3 dilute	HNO_3 concentrated (± 15 M)
K	Dissolve to form chloride with lower oxidation state and hydrogen.	Dissolve to form sulfate with lower oxidation state and hydrogen.	Dissolve to form sulfate with higher oxidation state and sulfur dioxide (SO_2)	Dissolve to form nitrate with lower oxidation state and nitrogen(II) oxide (NO) 1) 2) 3)	Dissolve to form nitrate with higher oxidation state and nitrogen(IV) oxide (NO_2) 2)
Na					
Ba					
Sr					
Ca					
Mg1)					
Al2)					
Zn					
Cd					
Fe					
Co					
Ni					
Sn					
Pb		No influence			
H					
Cu3)	No influence				
Hg					
Ag					
Pt			No influence	No influence	No influence
Au					

Notes:

1) With HNO_3 solution dilute (< 1M), Mg produces H_2)
2) HNO_3 react very slowly with Al in cold condition.
3) Co(II) nitrate formed with the addition of HNO_3, whereas Co(I) nitrate does not formed.

Materials

- Test tube
- Test tube rack
- Dropper pipette
- Fe, Zn, Cu, Al, Pb metals
- Iron nail
- Na_2S solution
- NaOH (2 M)
- HNO_3 (5 M)
- HCl (5 M)

Procedure

1. Prepare small pieces of Zn, Fe, Cu, Al and Pb metals. Clean those metals by using steel fiber (sandpaper) and place the samples to test tube rack separately.
2. Add 3 mL of 5 M HCl solution to test tube and record the resulted-observation in Table 4.2 in worksheet. Write the reaction equation.
3. If the reaction does not occur, heat the test tube gently and record the resulted-observation.
4. Repeat steps 2 to 3 for other metals.
5. Replace HCl 5 M with 5 M HNO_3 solution, and repeat step 1 to 4. Record the resulted-observation in Table 4.2. Write the reaction equation.
6. Replace 5 M HCl with 5 M NaOH solution, and repeat step 1 to 4. If there is no resulted-observation after heating, pour the solution to another test tube. Record the resulted-observation in Table 4.3. Write the reaction equation.
7. Add 2 mL of Na_2S solution into the test tube. Record the resulted-observation in Table 4.3. Write the reaction equation.

Attention.

1. Acid and alkali are corrosive substances. Use goggles during the experiment.
2. If the solution spilled out to clothes or skin, wash it with water immediately.
3. Poisonous gas may be resulted during the experiment. Do the experiment separately and use reagents in small amount to avoid or to minimize the produced-poisonous gas. If excess reagents are used, move rack and test tube to the fume hood.
4. Clean the residue with flowing water. Take the metal residue from washing vessel and throw to rubbish bin.
5. Sulfide solution is dangerous and poisonous compound. Store the solution in the fume hood. Throw residual solution into the washing vessel in fume hood.

EXPERIMENT 5

ELECTROCHEMISTRY CELL AND ELECTRODE POTENTIAL

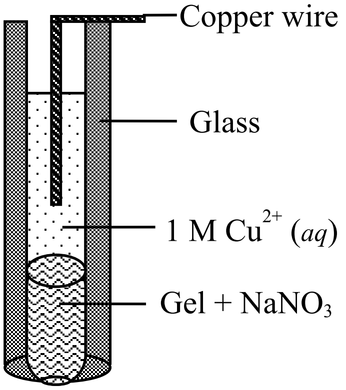
Purpose

To study electrode potential of several metals in electrochemistry cell

Introduction

Electrode potential of a metal illustrates the reduction-oxidation tendency of particular metal relatively to standard electrode, usually H_2 system (100 kPa)│H^+ (1 M) where the value of $E^0 = 0.00$ V. The measurement of electrode potential carried out simpler by using standard electrode Cu^{2+} (1 M) │ Cu. Of course, converting relative to hydrogen electrode, the value of standard electrode of Cu^{2+} │ Cu ($E^0 = 0.34$ V) must be subtracted.

Standard Electrode of Cu^{2+} (1M) | Cu and salt bridge



Standard electrode of Cu^{2+} | Cu

Standard electrode contains narrow glass tube and hollowed-bottom. The mixture contains of gel, $NaNO_3$ and cotton filled at the bottom of tube. The bottom hollow plugged with cotton in order to restrain gel position. Add 1 M $CuSO_4$ solution above gel and immerse copper wire as terminal. This standard electrode circuit called half cell circuit and salt bridge all at once. If this standard electrode circuit immersed in another half-cell cation standard solution, such as Zn^{2+} | Zn and both terminal (Cu and Zn) are connected to Voltmeter, electromotive force cell value is obtained. Gel electrolyte is used to keep charge balance during redox process occurred and gel for prevent the mixing of ions from the two half-cell areas.

Materials

- Standard electrode of Cu^{2+} | Cu
- Half cell system of Fe^{2+} | Fe, Mg^{2+} | Mg, Zn^{2+} | Zn, Sn^{2+} | Sn, Pb^{2+} | Pb, Al^{3+} | Al
- Voltmeter

Procedure

1. Immerse the standard electrode into the solution of half-cell system.
2. Connect each terminal with voltmeter wire; turn the voltmeter button to DC position, read, and record the value of emf.
3. Lift the standard electrode; wash it with flowing water on glass part, use again for other half-cell systems.

EXPERIMENT 6

CORROSION OF METALS (1)

Purpose

To study the nature of corrosion of several metals in gel medium

Introduction

Spontaneously redox reaction in electrochemistry cell is the sum of two half reaction cell with positive value of total electromotive force cell, emf. The level of corrosion of metal is studied by comparing oxidation level relative to O_2 in water. In base condition, reduction of oxygen in water yield to OH^- ion, which forms pink-red color with phenolphthalein (pp) indicator. Iron oxidized to Fe^{2+} which form blue color with ferricyanide ion. If such redox reactions take place in gel medium, the resulted-color localized in oxidation or reduction area. Due to the slow spreading of ions, it is possible to identify anode and cathode side. The active site of iron stick (such as iron nail), found at the end of nail. The electrons flow trough the stick and then captured by oxygen. Therefore, oxidation occurred at the end of nail, and reduction at the center.

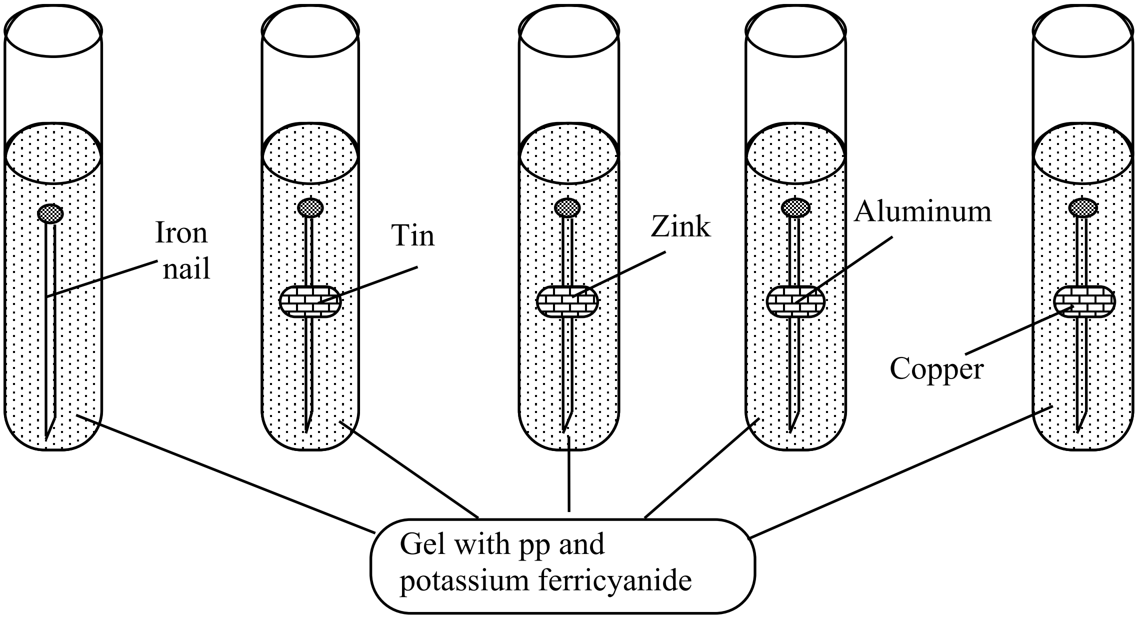


Figure 6.1 Experiment design

Materials

- Test tube
- Beaker glass 250 mL
- Bunsen burner, wire gauze
- Iron nail
- Aluminum sheet
- Copper sheet
- $K_3[Fe(CN)_6]$ solution *)
- Gel
- Phenolphthalein (pp)
- Zink sheet
- Tin sheet

***) Do not use $K_4[Fe(CN)_6]$**

Procedure

A. Seaweed gel making

1. Boil 80 mL of aquadest in beaker glass 250 mL.
2. Pour 0.5 g seaweed into aquadest and stir it until the gel dissolved.
3. Add 5 g of NaCl into the solution and stir continuously
4. Add 2 mL of phenolphthalein (pp) indicator and 1 mL of 0.1 M $K_3[Fe(CN)_6]$ solution. Stir until homogeny and stop the heating. Cool down the gel. The color of the mixture must be yellow, not green, blue or uncolored.

B. Cleaning of iron nail

5. Submerge five iron nails into 15 mL of 2 M H_2SO_4 solution in the test tube for five minutes.
6. Boil 50 mL of water in beaker glass 250 mL, clean the acid from the nail carefully (on step 5), rinse nails with water and then put the nails gently into boiling water. Move the nails into test tube with clean pliers.

C. Working with cleaned nails

7. Label test tubes 1 to 5. Place a cleaned nail into test tube 1. Attention: for test tube 2 – 5, nails must be precisely fit the hole of metals (see Figure 6.1)
8. Make a hole on copper sheet, zinc, tin and aluminum sheet with a nail. Put a cleaned nail through those holes. Ensure, there is a good contact between the two metals (alternative way: wrap the nail with the sheet).

9. Place those pairs of metals in the test tube 2 – 5. Pour gel indicator (that has made) gently into test tube 1 – 5. Attention: there must no bubble.
10. Place the test tubes on a shelf tube. After a while, observe the color changing around gel. According to the observation, color the area around nail and metal sheet on Figure 6.1

Note:

If the color changing unobserved, move the test tubes to beaker glass and observe it next day.

EXPERIMENT 7

CORROSION OF METALS (2)

Purpose

To study the corrosion character of metals (iron and copper)

Introduction

The amount of electron transferred during corrosion process measured by using multimeter. The function of electrodes (anode species and cathode species) confirmed by knowing the direction of electron flowing or potential gap. Sodium chloride acts as electrolyte to keep ions mobility.

Materials

- Iron sheet 8 cm x 2 cm
- Copper sheet 8 cm x 2 cm
- Sandpaper
- Multimeter or milliammeter
- 0.1 M $K_3[Fe(CN)_6]$ solution
- Phenolphthalein (pp)
- 3% NaCl solution
- Acetone

Procedure

1. Clean the iron and copper sheets with sandpaper and acetone soaked-cotton to clean the fat.
2. Mix the solutions of 40 mL of 3 % NaCl solution and 20 mL of 0.1 M $K_3[Fe(CN)_6]$ solution in beaker glass 250 mL to make feroxyl indicator. Add phenolphthalein indicator gently into the mixture and stir it. In this experiment, feroxyl indicator produces blue color with Fe^{2+} ion and pp produce pink color with OH^- ion.
3. Place an iron sheet and a cooper sheet into white paper based-beaker glass 250 mL. By using alligator clips, connect the two metals with milliammeter. Pour the feroxyl solution into the beaker glass until the electrode ends immersed. (Note: keep the alligator clip dry and two metals do not connect directly).

4. Observe the electricity current indicator on milliammeter to investigate the amount electrons flow through the two metals and the color changing. When the color changed, observe the indicator on milliammeter.
5. Record the result of the observation on worksheet paper and compare with Experiment 5 that has done.

EXPERIMENT 8

CORROSION OF METALS (3)

Purpose

To study the corrosion characters of metals (iron, magnesium and copper)

Introduction

The protection of corrosion of metals on machineries is an important effort. Beside painting and platting, there is a method to prevent corrosion based on the characteristic of metals. In many cases, metal is becoming less reactive due to the protection of strong oxide layer from more reactive metal. By this, metal is protected from the corrosion process by sacrificed-electrode. The method is known as sacrificial anode.

Sacrificial anode means that anode is sacrificed to protect the anode from further corrosion. The process based on the chemistry nature of the metals. The more oxidized metals will protect the lower oxidized metals from the corrosion.

Materials

- Iron sheet 8 cm x 2 cm
- Copper sheet 8 cm x 2 cm
- Magnesium ribbon
- Multimeter or milliammeter
- 3% NaCl solution
- Acetone
- Sandpaper
-

Procedure

1. Clean the iron and copper sheets with sandpaper and acetone soaked-cotton to clean the fat.
2. Prepare 50 mL of 3% NaCl solution in beaker glass 250 mL.
3. Immerse an iron sheet and a copper sheet into the beaker glass and then use alligator clips to connect the two metals to milliammeter. (Note: keep the alligator clips dry and two metals do not connect directly).
4. Observe the electricity current indicator on milliammeter to investigate the amount electrons flow through the two metals.

5. Change copper electrode with magnesium ribbon and then observe the electricity current indicator on milliammeter to investigate the amount electrons flow through the two metals.
6. Record the result of the observation on worksheet paper and compare the results with the rate of iron metals corrosion.

EXPERIMENT 9

PREPARATION OF POTASSIUM-CHROMIUM ALUM, $KCr(SO_4)_2 \cdot 12H_2O$

Purpose

To study the preparation of potassium-chromium alum

Introduction

Alum is a double salt of $M^I M^{II}(SO_4)_2 \cdot nH_2O$, where M^I is alkali metas (Na, K); M^{II} is metals with oxidation state +3, such as Al, Cr and Fe. Alum of K/Na – Al – sulfate and K/Na – Cr – sulfate are good example of alum, which its crystallization is easy to be studied.

Materials

- Beaker glass
- Stirring rod
- Evaporating dish
- Filter paper
- Hirsch funnel
- Watch glass
- Water bath
- H_2SO_4 (5 M)
- Ethanol
- Sodium dichromate
- Hydrogen peroxide 3%
- HNO_3(2 M)
- NaOH (5 M)

Procedure

1. Pour 25 mL of 5 M H_2SO_4 solution into a beaker glass and then add 4 g potassium dichromate. Stir the mixture and heat in a water bath to dissolve dichromate.
2. Cool the solution in ice bath for about 10 minutes and then add 4 mL of ethanol drop to drop into the mixture. Add ethanol carefully, because the reaction releases heat. Observe the changing occurred and record on worksheet paper.
3. Cover the beaker glass with watch glass and observe the changing occurred next day.
4. Collect the crystals formed in Hirsch funnel and move the residue from beaker glass by adding 5 mL of 60 % ethanol solution. If it necessary, repeat the procedure until no more residue left in Hirsch funnel. Let crystals dry at room temperature (called as air-drying) until next day.

5. Weigh the crystal mass and calculate the yield percentage of potassium-chromium alum based on the amount of dichromate used.
6. Test of the chromium ion presence in alum.

   Add drop to drop of 5 M NaOH to sample (0.05 g alum in 2 mL of water) until no more changing. (Every one drop of NaOH, shake and observe carefully before next adding). Then add 1 mL of 3% H_2O_2 solution and heat the mixture until the color change. The yellow color indicates the presence of chromate ion (CrO_4^{2-}). Record the observation on worksheet paper and write the balanced ionic reaction of the oxidation of Cr^{3+}(aq) by H_2O_2 in base condition.

   Note: the test for Cr^{3+} must be undertaken in base condition.
7. Test the presence of sulfate ion in alum

   In a test tube, dissolve 0.05 g chromium alum in 5 mL of water. Add few drops of 0.1 M $Ba(NO_3)_2$ solution and 2 M HNO_3 solution. Record the observation on worksheet paper and write the balanced ionic reaction of the test.

EXPERIMENT 10

PREPARATION OF POTASSIUM-ALUMINUM ALUM, $KAl(SO_4)_2\cdot 12H_2O$

Purpose

To study the preparation of potassium-aluminum alum

Materials

- Beaker glass 100 mL
- Stirring rod
- Filter paper
- Hirsch funnel
- Graduated cylinder 10 mL
- Water bath
- Ethanol 60%
- Aluminum (soft drink cane)
- KOH (2 M)
- H_2SO_4 (9 - 10 M)
- Watch glass
- Glass wool

Procedure

1. Weigh 0.2 g of small pieces of aluminum on watch glass.
2. Pour 10 mL of 0.2 M KOH solution into beaker glass.
3. Warm the solution on water bath and then add a piece of aluminum (do it in fume hood).

   Note: **Do not warm the solution too hot and remove beaker glass from fume hood until all pieces of aluminum added to the solution. The reaction of aluminum and KOH releases hydrogen gas**.
4. As the reaction occurred, move out the beaker glass from water bath immediately. Move it back into water bath as the reaction slowed down (no more bubbles produced) and add other pieces of aluminum.
5. If all pieces of aluminum have been reacted, filter the mixture with a funnel that has plugged with glass wool. (Ask the assistant how to do it).
6. Add 20 mL of 9-10 M H_2SO_4 into filtrate solution carefully and check it with litmus paper. Ensure that the solution is acid.

   Note: **Concentrated (10 M) sulfuric acid irritates and burns the skin. If it happened, wash with flowing water and take medical care**.

7. Cover the beaker glass with watch glass and let it for about 24 hours. After 24 hours, the crystal of potassium aluminum alum, $KAl(SO_4)_2 \cdot 12H_2O$, formed. (The growing of crystal may be speed up by scraping the stirring rod to inner part of solution while cooling or add 2-3 mL of ethanol).
8. Collect the formed-crystal on Hirsch funnel and move the residue from beaker glass by adding 5 mL of 60% ethanol. If it necessary, repeat the procedure until no more residue left in Hirsch funnel.
9. Let the crystal dry until next day.
10. Weigh the crystal mass and calculate the yield percentage based on the amount of aluminum used.
11. Do the re-crystallization to the impure yield with water as solvent.
12. For students who have synthesized both alum (chromium and aluminum), both alums have the same structure, therefore possible to grow mixing crystals. Hang up a small part of aluminum alum with yarn and immerse it into saturated solution of chromium alum. By this, the alternate layers formed, uncolored of aluminum alum and purple to reddish of chromium alum.

Re-crystallization technique

The purpose of re-crystallization is to purify resulted-solid. The resulted-solid dissolved in minimum amount of solvent in an erlenmeyer flask or beaker glass. The solid must has high solubility in hot solvent, but low in cold one. If undissolved-impurities solid found in hot solution, filter it with funnel and filter paper in hot condition to avoid early crystallization. If crystal found on filter paper, wash it with hot solvent. If the filtrate is too dilute, concentrate it by heating. Pure crystals grow during the cooling process. The growing of crystal may be speed up by scraping the stirring rod to inner part of glass. The pure crystal filtered and washed with solvent in minimum amount.

EXPERIMENT 11

PREPARATION OF COORDINATION COMPOUND, $[Ni(NH_3)_6]I_2$

Purpose

To study the preparation of coordination compound of $[Ni(NH_3)_6]I_2$

Introduction

Complex (coordination) compound is characteristic compound of transition metals that correspond to the existence of d orbital. The existence of d orbital cause transition metals not only have various oxidation states but also the capability to interact coordinately with other atom donor. Complex compound of $[Ni(NH_3)_6]I_2$ is an example of Ni^{2+} compound with coordination number 6 where its crystallization is relatively easy to be studied. The success of the compound preparation is easily tested qualitatively to Ni^{2+}.

Materials

- Beaker glass 100 mL
- Stirring rod
- Filter paper
- Hirsch funnel
- Graduated cylinder 10 mL
- Labeled-test tube
- Ammonia 1 M
- Ethanol
- Nickel chloride hexahydrate
- Potassium iodide
- Starch indicator
- H_2O_2 (3%)

Procedure

1. Dissolve 1 g of nickel chloride hexahydrate into 5 mL water a beaker glass.
2. Place that beaker glass in the fume hood and add 10 mL of concentrated (15M) NH_3 solution.
3. Add 2.6 g of potassium iodide to the mixture. Let the mixture for several minutes.
4. Collect the formed-crystal on Hirsch funnel, wash it twice with 2 mL of ethanol solution 1:1 and then add 2 mL of ethanol solution.
5. Dry the crystals in windy air for several minutes.
6. Move the dried-crystals to filter paper. Ask the assistant how to move crystals from Hirsch funnel to filter paper.

7. Move out the exceeding solvent by press the crystals between two filter papers.
8. Move the resulted crystal to the weighed and labeled-tube. Weigh the tube mass with the contents. Calculate mass percentage of the product based on the amount of nickel chloride hexahydrate.
9. Test the existence of nickel ion in the compound.

   Dissolve a small amount of sample (about 0.001 g of compound in 0.5 mL of water), add 5 M NH_3 solution, and then add 5 drops of dimethyl glioxym solution. Red strawberry solid produced if there is Ni^{2+} ion.
10. Test the existence of iodide ion in the compound.

    Dissolve a small amount of compound (about 0.001 g of compound in 0.5 mL of water), acidify with 2 drops of 5 M sulfuric acid solution and then add 3% H_2O_2 solution.

EXPERIMENT 12

PURIFICATION OF KITCHEN SALT BY RE-CRYSTALLIZATION METHOD

Purpose

To study the crystallization method on the purification of kitchen salt by evaporation and precipitation

Introduction

Resulting purity high-level compound is an important thing in chemistry. The usual method on solid purification is re-crystallization (the forming of repeating crystal). Re-crystallization based on the difference of solubility capacity of solid and impurities in particular solvent. If it possible, use alternate solvent that only dissolve the impurities. Such purification is widely used in industrial and laboratory to improve the quality of particular substance.

Requisites of a solvent in re-crystallization process are:

1. Give significant solubility differences between purified-substance and impurities.
2. The solubility of substance in solvent is a temperature function. The solubility usually decreases with the decreasing of temperature.
3. Easily separate from the crystals.
4. Do not leave the impurities in the purified crystals.
5. Do not react with purified substance.

Kitchen salt contains sodium chloride as major component, and Ca^{2+}, Mg^{2+}, Al^{3+}, Fe^{3+}, SO_4^{2-}, $I^-$and Br^- as impurities. Those impurities easily dissolved in water. Re-crystallization method with water as a solvent is general method to get high-level sodium chloride from kitchen salt. Particular ions needed to eliminate the existence of impurity ions. These ions will bind the impurity ions to form low-level solubility compound in water. By this, the purified and impurities substances easily separated.

Materials

- Burner
- Beaker glass
- Kitchen salt and CaO crystal
- Dilute $Ba(OH)_2$ or $BaCl_2$ (0,5 M) solution

- Graduated cylinder
- Funnel
- Gas adapter
- Filter paper and litmus paper
- $(NH_4)_2CO_3$ solution (6 gram in 200 mL)
- Dilute HCl solution (0,1M),
- Concentrated H_2SO_4
-

Procedure

1. Into a beaker glass, dissolve about 16 g kitchen salt in 50 mL water. Boil and stir the mixture. Divide the solution into 2 parts in equal amount and called as solution A and B.
2. Crystallization of solution A
   a. Add about 0.2 g of CaO into solution A
   b. Add $Ba(OH)_2$ solution drop to drop until no more precipitate formed at the last drop.
   c. Add $(NH_4)_2CO_3$ solution drop to drop and stir continuously.
   d. Filter the mixture into cleaned and weighed beaker glass. Neutralize filtrate by adding of dilute HCl solution drop to drop. (Test the neutrality of the solution with litmus paper in every drop).
   e. Evaporate the solution until relatively dry.
   f. Weigh the resulted NaCl (which is brighter and whiter than original kitchen salt) and calculate the percentage.
3. Crystallization of solution B
   a. Saturate the solution B by HCl gas adding. Hydrochloric acid gas obtained from the reaction of kitchen salt and concentrated sulfuric acid. (Do the reaction in fume hood). The flowing of HCl stopped when no more NaCl crystal growing in the solution.
   b. Separate the crystal by filtering, dry it and then weigh the product and compare to method 1 above.

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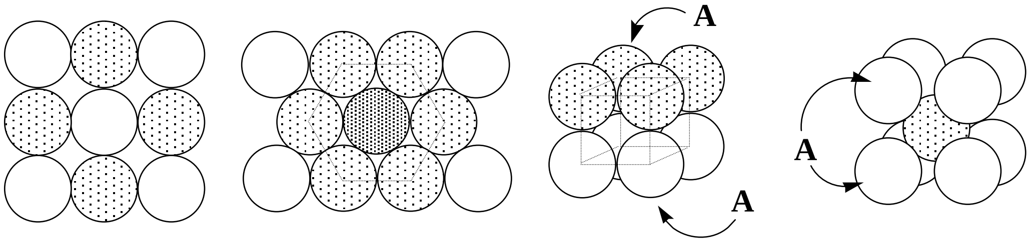
WORKSHEET-1 **Day/Date of Practicum: ......……………….**

CLOSE PACKING GEOMETRY

Ball packing efficiency



(a) **(b)** **(c)** **(d)**

Figure 1

Arrange the balls side-by-side in a plane (Figure 1a)

Arrange the balls as closest as possible (Figure 1b)

How many balls that touch center-ball in the same layer for each packing?

Answer: ………………………… balls for (a) and …………………….. balls for (b)

Which is a closer packing, (a) or (b)?

Answer: …………………………

Geometry structure of (a) is not a closest packing, whereas (b) is a closest packing. The arrangement of layer (b) is known as *hexagon*.

If we add the second, third layer, etc, to the arrangement model (a) so that the balls of second and third layer lie above the first layer, we get layer A, A, A (Figure 1c). How many balls that touch every other ball in layer A, A, A?

Answer: ……………… balls

Place one ball into the hole between two layers A, A so that the hole-ball touch all balls. Layer A, A will expand. The resulted geometry of expanding layer A, A is called ……………………………. (Figure 1d), which has coordination number …………….

Which is the closer packing, (c) or (d)?

Answer: ………………

Hexagon and cubic close packing

Two possibilities arrangement of closest packing of same size balls are **hexagon closest packing (hcp)** and **cubic closest packing (ccp)**. The last arrangement also called as face centered **cubic closest packing (fcc)**. The two arrangements use the hexagon layer (Figure 1b) and represent the most effective way to arrange balls or to fill the hole/space.

Closest packing of *hcp*

Place 7-balls-hexagon pattern (layer 1) on the desk.

Place 3-balls-triangular pattern (layer 2) fit the hole of layer A.

Place another 7-balls-hexagon pattern (layer 3) fit the hole of layer B and thus lies above layer A.

Repeating of these pattern continuously will produce A, B, …., …., …., …. pattern.

Alternatively: arrange 3-balls-triangular pattern (layer 1) - 7-balls-hexagon pattern (layer 2) - 3-balls-triangular pattern (layer 3).

Determine the amount of balls that touch centered-ball in the *hcp* geometry.

Answer: ………………… balls, consisted of …………….. balls in same layer and ……….. balls in layer above and under. (The numeric known as coordination number)

Arrange 3 patterns of 7-balls-hexagon pattern in the same way.

Is the coordination number still the same?

Answer: Yes / No, …………….. balls.

Direct this pattern to light source. What do you get?

Answer: ………………………………………………………………………………..
………………………………………………………………………………...

Put the wire through the hole of hcp packing. What do you get?

Answer: ……………………………………………………………………………....
.........………………………………………………………………………....

Closes packing of ccp or fcc.

1. Arrange the pattern like hcp pattern (3-balls – 7-balls – 3-balls or A – B – A layer)
2. Turn the triangle pattern on top 60^0, so that layer 3 does not lie above layer 1 (layer 3 fit the hole of layer 1).
3. Repeat step 1 and 2 to obtain the pattern of A, B, ..., ….., ….., ….., ….., …., (etc).

4. Determine the amount of balls touch centered-ball in the *ccp* or *fcc* geometry

   Answer: ………………… balls, consisted of …………….. balls in same layer and ……….. balls in layer above and bellow. (The numeric is known as coordination number).
5. Repeat step 1 to 3 with 3 patterns of 7-balls.
6. Is the coordination number still the same?

   Answer: Yes / No, …………….. balls.
7. Direct this pattern to light source. What do you get?

   Answer: ………………………………………………………………………………..
   ………………………………………………………………………………...
8. Put the wire through the hole of hcp packing. What do you get?

   Answer: : ……………………………………………………………………….....
   ....…………………………………………………………………………....
9. Arrange the patterns of 5-balls – 4-balls – 5-balls (14 balls). Determine the amount of balls that touch center-ball on every cubic face.

   Answer: ……………… balls.
10. For every cubic face, is it a closest packing layer?

    Answer : …………………..
11. Is every face of cubic a closest packing layer?

    Answer: ……………………………………………………………………….....
12. In order to get closest packing layer, from what direction (axis or diagonal) the structure must be viewed?

    Answer: ……………………………………………………………………….....
13. Shade the balls in the same layer and label it with layer A, B or C for Figure 1.2

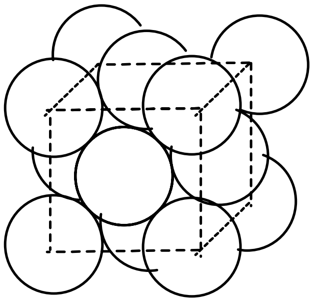


Figure 1.2

Tetrahedron geometry (in cubic)

Place the 3-balls-triangular pattern on the desk.

Fit one ball in the triangular hole (called as **tetrahedron hole**)

Put a smaller ball and place in the tetrahedron hole. Ensure that the smaller ball touch four balls in tetrahedron geometry precisely (signed by no sound when shook).

Measure the diameter or radius of bigger ball (ping-pong ball) and state as r_- (anion radius). Measure the diameter or radius of smaller ball and state as r_+ (cation radius).

Note: anion radius is bigger than cation radius

Diameter of small ball = ..., so the radius (r_+) = ...

Diameter of ping-pong ball = ..., so the radius (r_-) = ...

Radius ratio $\frac{r_+}{r_-}$ = …………………….., or $\frac{r_-}{r_+}$ = ...

The truth of resulted-ratio could be tested mathematically according to Figure 1.3a (structure of body centered cubic with side length a). Determine the ratio of ping-pong ball (A=B=C=D) to hole-ball (M)

Hint: ($\sqrt{3} = 1.732$, $\sqrt{2} = 1.414$)

iv) State the ball radius in the term of r_+ or r_- and the length in a.

v) State the face diagonal, CD, and body diagonal, ED, (and then MD) in the term of r_+ or r_- and a.

vi) Obtain the value of r_+ and r_- in the term of a, then calculate its ratio

Calculation:

Face diagonal CD : ……………. = …………………. (Ball C touch ball D)

Body diagonal ED : ……………. = …………………. (Balls E-M-D touch each other)

r_+ = ……………….... and r_- = ………………………….

Therefore, the radius ratio:

$\frac{r_+}{r_-}$ = ..., OR $\frac{r_-}{r_+}$ = ...

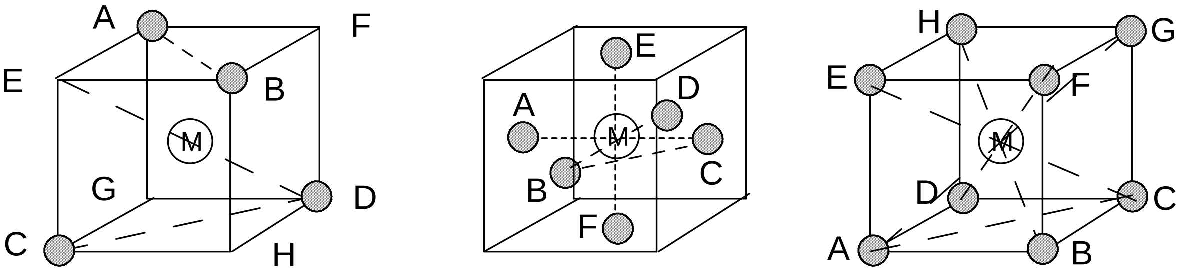


Figure 1.3 Tetrahedron (a), octahedron (b), and body centered cubic (c) geometries in cubic perspective

Octahedron geometry (in face centered cubic)

Place 3-balls-triangular pattern (layer 1) on the desk.

Place 3-balls-triangular pattern (layer 2) so that balls of layer 2 lies above layer 1.

Turn 60^0 clockwise for layer 2 (**octahedron hole**).

Put a smaller ball and place in the octahedron hole. Ensure that the smaller ball touch six balls in octahedron geometry precisely.

Measure the diameter or radius of bigger ball (ping-pong ball) and state as r_- (anion radius). Measure the diameter or radius of smaller ball and state as r_+ (cation radius).

Note: anion radius is bigger than cation radius

Diameter of small ball = ..., so the radius (r_+) = ...

Diameter of ping-pong ball = ..., so the radius (r_-) = ...

Radius ratio $\frac{r_+}{r_-}$ = …………………….., or $\frac{r_-}{r_+}$ = ...

The truth of resulted-ratio could be tested mathematically according to Figure 1.3b (structure of face centered cubic with side length a). Determine the ratio of ping-pong ball (ABCDEF) to hole-ball (M)

Hint: ($\sqrt{2} = 1.414$)

i) State the ball radius in the term of r_+ or r_- and the length in a.

ii) State BM (=CD) and BC in the term of r_+ or r_- and a.

iii) Obtain the value of r_+ and r_- in the term of a, then calculate its ratio

Calculation:

BM (= MC) : ……………. = …………………(Balls BM and MC touch each other)

BC : ……………. = ……………….. (Balls B and C touch each other)

r_+ = ……………….... and r_- = ………………………….

Therefore, the radius ratio:

$\frac{r_+}{r_-}$ = ..., OR $\frac{r_-}{r_+}$ = ...

Which is the bigger hole, tetrahedron or octahedron hole?

Answer: ……………………………………………………………………………....

Body centered cubic geometry

Place 4 balls-side by side pattern (layer 1) on the desk.

Place another 4 balls-side by side pattern (layer 2) on layer 1, so that layer 1 lies above layer 2 (body center cubic hole).

Put a smaller ball and place in the octahedron hole. Ensure that the smaller ball touch eight balls in body centered cubic geometry precisely.

Measure the diameter or radius of bigger ball (ping-pong ball) and state as r_- (anion radius). Measure the diameter or radius of smaller ball and state as r_+ (cation radius).

Note: anion radius is bigger than cation radius

Diameter of small ball = ..., so the radius (r_+) = ...

Diameter of ping-pong ball = ..., so the radius (r_-) = ...

Radius ratio $\frac{r_+}{r_-}$ = ………………….., or $\frac{r_-}{r_+}$ = ...

The truth of resulted-ratio could be tested mathematically according to Figure 1.3b (structure of face centered cubic with side length a). Determine the ratio of ping-pong ball (ABCD-EFGH) to hole-ball (M)

Hint: (√3 = 1.732)

i) State the ball radius in the term of r_+ or r_- and the length in *a*.

ii) State BM (=MC) and BC in the term of r_+ or r_- and *a*.

iii) Obtain the value of r_+ and r_- in the term of *a*, then calculate its ratio

Calculation:

BM (= MC) : ……………. = …………………(Balls BM and MC touch each other)

BC : ……………. = ………………….. (Balls BC touch each other)

r_+ = ……………….... and r_- = ………………………….

Therefore, the radius ratio:

$\frac{r_+}{r_-}$ = ..., OR $\frac{r_-}{r_+}$ = ...

Which is the bigger hole, tetrahedron, octahedron or body centered cubic hole?

Answer: ………………………………………………………………………………....

Identification of tetrahedron and octahedron holes

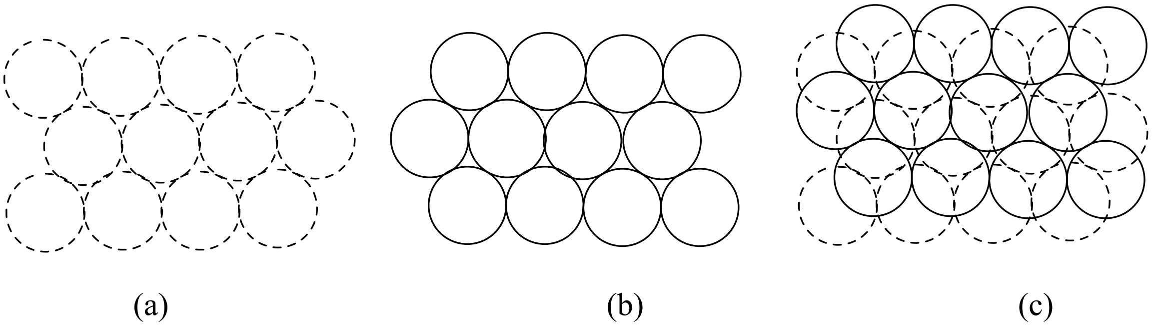

(a) (b) (c)

Figure 1.4 Models of hexagon layer packing (a,b), and layer A, B closest packing (c)

Arrange a 12-balls-hexagon layer (layer 1) as shown in Figure 1.4a. (there are 12 holes)

Arrange another 12-balls-hexagon layer in opposite direction (Figure 1.4b)

Put layer 2 on layer 1 according to AB pattern (balls of layer 2 fit the holes of layer 1). The holes in layer 1 occupied by layer 2 balls called tetrahedron holes, whereas unoccupied called octahedron holes)

Identify and mark the type and the amount of holes.

Direct the two layers (Figure 1.5c) to light source. (Light can penetrate octahedron holes, not tetrahedron holes)

Put the wire through the hole. (Wire may through the octahedron holes, not tetrahedron hole).

How many tetrahedral and octahedral holes are there?

Answer: ……………………………………………………………………………..…..

Therefore, between two hexagon closest packing, there are tetrahedral holes as twice as octahedral holes.

Assistant’s comment :

Assistant Signature: ....

Score: ....

WORKSHEET-2 **Day/Date of Practicum: ....……………….**

INORGANIC QUALITATIVE REACTION

Table 2.1 The observation of metals ion and hydroxide ion reaction in ammonia solution

Metal ion	Observation	Ionic Reaction
Mg^{2+}	Uncolored precipitate formed. The precipitate does not dissolve in excess adding of NaOH and NH_3.	$Mg^{2+}(aq) + 2OH^{-}(aq) \rightarrow Mg(OH)_2(s)$
Ba^{2+}		
Al^{3+}		
Cr^{3+}		
Fe^{3+}		
Mn^{2+}		
Pb^{2+}		
Cu^{2+}		
Ni^{2+}		
Ag^{+}		
Zn^{2+}		

5. Cations that form precipitates on the adding of NaOH are: …..………………………....
………………………………………………………………………………………….

6.a. Cations that form precipitates on the adding of NaOH but dissolved in the excess NaOH are: ……………………………………………………………………..

6b. Cations that form precipitates on the adding of NH_3 but dissolved in the excess NH_3 are: …..………………………………………………………………………..

7a. The observation and equation of ionic reaction in the adding of 0.1 M $Al(NO_3)_3$ drop to drop into 1 mL of 2M NaOH are:

…………………………………………………………………………………………

…………………………………………………………………………………………

…………………………………………………………………………………………

7b. The observation and equation of ionic reaction in the adding of 0.1 M $Fe(NO_3)_3$ drop to drop into 1 mL of 2M NaOH are:

…………………………………………………………………………………………

…………………………………………………………………………………………

…………………………………………………………………………………………

7c. The adding of reactant (between M^{3+} and NaOH) in reversed order are:

Cation	Observation	Reaction
Al^{3+}		
Fe^{3+}		

Which cations result different reaction if the reactant adding in reversed order?

Answer: …....……………………………………………………………………………

………………………………………………………………………………......……

………………………………………………………………………………......……

Assistant's comment :

Assistant Signature: .... Score: ....

WORKSHEET-3 **Day/Date of Practicum: ......…………..….**

OXIDATION-REDUCTION REACTION (1)

Table 3.2

1. Oxidant: permanganate ion

Activity	Test tube T	Test tube B
(a)	5 drops of H_2O_2 (3%) 1 drop of H_2SO_4 (5%) 5 drops of $KMnO_4$ (0.02 M) drop to drop	5 drops of H_2O_2 (3%) 1 drop of H_2SO_4 (5M) 5 drops of aquadest
Observa-tion	Cold : ……………………………… Hot : ……………………………...	Cold : …………………………... Hot : …………………………...
Reaction	Oxidation : ………………………………………………………………. Reduction : …………………………………………………………….. Redox : ……………………………………………………………...	
(b)	5 drops of Sn^{2+} (0.1 M) 1 drop of H_2SO_4 (5M) 5 drops of $KMnO_4$ (0.02M) drop to drop	………………………………… ………………………………… …………………………………
Observa-tion	Cold : ……………………………… Hot : ……………………………...	Cold : …………………………... Hot : …………………………...
	Oxidation : ……………………………………………………………... Reduction : …………………………………………………………….. Redox : ……………………………………………………………...	
(c)	5 drops of $H_2C_2O_4$ 0.1 M 1 drop H_2SO_4 (5M) 5 drops of $KMnO_4$ (0.02M) drop to drop	………………………………… ………………………………… …………………………………
Observa-tion	Cold : ……………………………… Hot : ……………………………...	Cold : …………………………... Hot : …………………………...
Reaction	Oxidation : $C_2O_4^{2-} \rightarrow 2\ CO_2 + 2e$ Reduction : …………………………………………………………….. Redox : ……………………………………………………………...	

2. Oxidant: ion dichromate

Activity	Test tube T	Test tube B
(a)	5 drops of $Fe(NH_4)_2(SO_4)_2$ (0.1M) 1 drop of H_2SO_4 (5M) 5 drops of $K_2Cr_2O_7$ (0.02M) drop to drop	5 drops of $Fe(NH_4)_2(SO_4)_2$ (0.1M) 1 drop of H_2SO_4 (5M) 5 drops of aquadest
Observa-tion	Cold : ...………………………… Hot : ………………………….	Cold : …………………………... Hot : …………………………...
Redox Testt	Test : add KSCN (0.05M) Observation : ……………………. ……………………………………	Test : add KSCN (0.05M) Observation : ……………………… ……………………………………..
Reaction	Oxidation : ……………………………………………………………... Reduction : …………………………………………………………….. Redox : ……………………………………………………………...	
(b)	5 drops KI (0.1M) 1 drop H_2SO_4 (5M) 5 drops of $K_2Cr_2O_7$ (0.02M) drop to drop	………………………………… ………………………………… …………………………………
Observa-tion	Cold : ...………………………… Hot : ………………………….	Cold : …………………………... Hot : …………………………...
Redox Testt	Test : ………………………………. Observation : …………………….. ……………………………………	Test : ……………………………… Observation : ……………………... ……………………………………..
Reaction	Oxidation : ……………………………………………………………... Reduction : …………………………………………………………….. Redox : ……………………………………………………………...	
(c)	5 drops H_2SO_3 (0.1M) 5 drops of $K_2Cr_2O_7$ (0.02M) drop to drop	………………………………… …………………………………
Observa-tion	Cold : ...………………………… Hot : ………………………….	Cold : …………………………... Hot : …………………………...
Redox Testt	Test : ………………………………. Observation : …………………….. ……………………………………	Test : ……………………………… Observation : ……………………... ……………………………………..
Reaction	Oxidation : ……………………………………………………………... Reduction : …………………………………………………………….. Redox : ……………………………………………………………...	

3. Oxidant: Hydrogen peroxide

Activity	Test tube T	Test tube B
(a)	5 drops of $Fe(NH_4)_2(SO_4)_2$ (0.1M) 1 drop of H_2SO_4 (5M) 5 drops of H_2O_2 (3%) drop to drop	…………………………………… …………………………………… 5 drops of H_2O
Observa-tion	Cold : ..………………………… Hot : ………………………….	Cold : …………………………... Hot : …………………………...
Redox Testt	Test : ………………………………. Observation : ………………………. ……………………………………	Test : ………………………………… Observation : ………………………... ………………………………………..
Reaction	Oxidation : ………………………………………………………………... Reduction : …………………………………………………………….. Redox : ………………………………………………………………...	
(b)	5 drops of KI (0.1M) 1 drop of H_2SO_4 (5M) 5 drops of H_2O_2 (3%) drop to drop	…………………………………… …………………………………… ……………………………………
Observa-tion	Cold : ..………………………… Hot : ………………………….	Cold : …………………………... Hot : …………………………...
Redox Testt	Test : ………………………………. Observation : ………………………. ……………………………………	Test : ………………………………… Observation : ………………………... ………………………………………..
Reaction	Oxidation : ………………………………………………………………... Reduction : …………………………………………………………….. Redox : ………………………………………………………………...	

4. Comparison of the HCl dan H_2SO_4 influences on the $KMnO_4$ reaction

Activity	Test tube T	Test tube B
	5 drops of HCl (5M) 1 drop of $KMnO_4$ (0.02)	5 drops of H_2SO_4 (5M) 1 drop of $KMnO_4$ (0.02M)
Observa-tion	Cold : ..…………………………… Hot : ……………………………..	Cold : …………………………... Hot : …………………………...
Reaction	Oxidation : ………………………………………………………………... Reduction : …………………………………………………………….. Redox : ………………………………………………………………...	

Questions:

1. According to the observation no 4 on Table 3.2, explain why sulfuric acid (H_2SO_4) solution is used to acidify the solution of $KMnO_4$ and $K_2Cr_2O_7$, instead of hydrochloric acid (HCl).

   Answer: ………………………………………………………………………………

   …………………………………………………………………………………………

   …………………………………………………………………………………………

   …………………………………………………………………………………………

   …………………………………………………………………………………………

2. In the study of permanganate ion as oxidant, redox test is unnecessary. Why? Explain your argument.

   Answer: ………………………………………………………………………………

   …………………………………………………………………………………………

   …………………………………………………………………………………………

   …………………………………………………………………………………………

   …………………………………………………………………………………………

Assistant's comment :

Assistant Signature: .... Score: ....

WORKSHEET-4 **Day/Date of Practicum: ......……………….**

OXIDATION-REDUCTION REACTION (2)
THE INFLUENCES OF ACID AND BASE TO METALS

Table 4.2 The Influence of Acid to Metals

Acid	Metal	Observation		Reaction
		Cold	Hot	
HCl	Zn			
	Fe			
	Cu			
	Al			
	Pb			
HNO_3	Zn			
	Fe			
	Cu			
	Al			
	Pb			

Table 4.3 The Influence of Base to Metals

Metal	Observation and Reaction	
	The influence of alkali	The influence of sulfide
Zn		
Pb		
Fe		
Al		
Cu		

Assistant's comment :

Assistant Signature: .... Score: ....

WORKSHEET-5 **Day/Date of Practicum: ......……………….**

ELECTROCHEMISTRY CELL AND ELECTRODE POTENTIAL

E^0 for $Cu^{2+} | Cu = 0.34$ V

Half-cell system	Is standard electrode of $Cu^{2+} \| Cu$		The measured E_{cell}	E^0 of half-cell	
	+ or -	anode or cathode		Experiment	Literature
$Zn^{2+} \| Zn$					
$Pb^{2+} \| Pb$					
$Sn^{2+} \| Sn$					
$Fe^{2+} \| Fe$					
$Al^{3+} \| Al$					

Write the cell notation to illustrate pair of electrochemistry cell below:

System	Cell notation	E^0_{cell} / V
Fe / Zn		
Fe / Sn		
Fe / Al		
Fe / Cu		

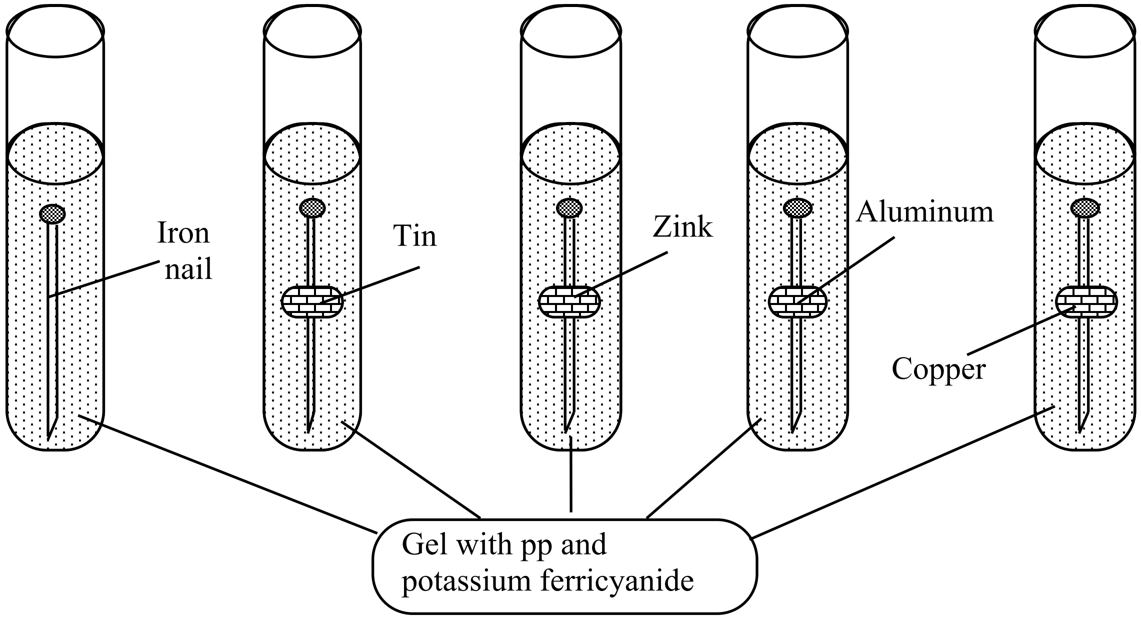
Assistant's comment :

Assistant Signature: .... Score: ....

WORKSHEET-6 **Day/Date of Practicum: ....…………….….**

CORROSION OF METALS (1)

System	Location of red color	Location of blue color	Ionic Reaction	
			Anodic	Cathodic
Fe	Along the nail rod	The ends of nail rod	$Fe(s) \rightarrow Fe^{2+}(aq) + 2e$	$O_2(aq)+2H_2O(\ell)+4e \rightarrow 4OH^-(aq)$
Fe/Sn				
Fe/Zn				
Fe/Al				
Fe/Cu				



Write the line notation of the corrosion for each cell and calculate the E^0. Remember that E^0 refers to cell of 1M M^{n+} solution cell. The value can be used as the reference to determine the possibility of anodic corrosion if two metals connected.

System	Line notation of cell	E^{o} *)
* Fe/Sn		
* Fe/Zn		
* Fe/Al		
* Fe/Cu		

*) Use the reaction data bellow :

$Fe^{2+}(aq) + 2e \rightleftharpoons Fe(s)$	$E^0 = - 0,41$ V
$O_2 + 2 H_2O(\ell) + 4e \rightleftharpoons 4 OH^-$	$E^0 = + 0,40$ V
$Sn^{2+}(aq) + 2 e \rightleftharpoons Sn(s)$	$E^0 = - 0,14$ V
$Zn^{2+}(aq) + 2 e \rightleftharpoons Zn(s)$	$E^0 = - 0,76$ V
$Al^{3+}(aq) + 3 e \rightleftharpoons Zn(s)$	$E^0 = - 1,68$ V
$Cu^{2+}(aq) + 2 e \rightleftharpoons Cu(s)$ E^0	$E^0 = +0,34$ V

Tin cane made from tin platted-iron sheet. How tin prevent the corrosion of iron?

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If the layer scratched, the corrosion rate of tin cane is high, but not for galvanized-iron. Explain for the differences of decomposition rate between the tin cane and galvanized-iron.

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Assistant's comment :

Assistant Signature: .... Score: ....

WORKSHEET-7 **Day/Date of Practicum: ......……………….**

CORROSION OF METALS (2)

The amount of flowing electricity: ………………….. mA

Sketch the experiment design and show the color changing on each electrode.

Write the anodic (oxidation) and cathodic (reduction) reaction.

Cathodic reaction : ……………………………………………………………………….

Anodic reaction: ……………………………………………………………………….

Assistant’s comment :

Assistant Signature: .... Score: ....

WORKSHEET-8 **Day/Date of Practicum: ......……………….**

CORROSION OF METALS (3)

The amount of flowing electricity current (Fe and Cu): ……………..mA
The amount of flowing electricity current (Fe and Mg): ……………..mA

Based on the observation, conclude metals that act as anode (oxidation) and cathode (reduction)

Write the anodic (oxidation) and cathodic (reduction) reaction.
Cathodic reaction : ……………………………………………………………………………….
Anodic reaction: ………………………………………………………………………….

Assistant's comment :

Assistant Signature: .... Score: ....

WORKSHEET-9 **Day/Date of Practicum: ......……………..….**

PREPARATION OF POTASSIUM-CHROMIUM ALUM, $KCr(SO_4)_2·12H_2O$

Observation:

1. The adding of 4 mL to ethanol solution:

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2. The existence of chromium ion test:

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   Write the equation of ionic balance of Cr^{3+} oxidized by H_2O_2 in base condition.

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3. The existence of sulfate ion test:

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   Ionic reaction of the test:

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Assistant's comment :

Assistant Signature: .... Score: ....

WORKSHEET-10 **Day/Date of Practicum: ......……………….**

PREPARATION OF POTASSIUM-ALUMINUM ALUM, $KAl(SO_4)_2{\cdot}12H_2O$

Mass of aluminum = ……………………..

Mass of tube and cover = ……………………..

Mass of tube + cover + potassium alum = ……………………..

Mass of potassium alum = ……………………..

Write the coefficient of ionic balance (use the method of ion-electron half-reaction balance)

1) $Al(s) + OH^-(aq) + H_2O(\ell) \rightarrow H_2(g) + [Al(OH)_4]^-(aq)$

2) $[Al(OH)_4]^-(aq) + H^+(aq) \rightarrow Al^{3+}(aq) + H_2O(\ell)$

3) $K^+(aq) + Al^{3+}(aq) + SO_4^{2-}(aq) + H_2O(\ell) \rightarrow KAl(SO_4)_2.12H_2O$

Theoretical yield = …………….

Percentage yield = …………….

What type reaction shown on reaction 1?

Answer: ……………………………………………………………………………....

What type reaction shown on reaction 2?

Answer: ……………………………………………………………………………....

The beaker glass must be placed in fume hood when the first reaction occurred. Why?

Answer: ……………………………………………………………………………....

On the adding of 10 M sulfuric acid solution, the acidity solution must be checked. Why?

Answer: ……………………………………………………………………………....

Why the percentage of yield less than 100%?

Answer: ……………………………………………………………………………....

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Assistant's comment :

Assistant Signature: .... Score: ....

PRETEST SHEET (done before the experiment)

PREPARATION OF COORDINATION COMPOUND, $[Ni(NH_3)_6]I_2$

Preparation of complex compound of $[Ni(NH_3)_6]I_2$

1. Pour 1.0 g of nickel(II) hexahydrate to 10 mL of 15 M NH_3 solution and 2.6 g of potassium iodide. Calculate the theoretic yield of complex compound of $[Ni(NH_3)_6]I_2$ based on the reaction.

2. Dimethylglioxim often used as testing agent for the existence of nickel(II) ion. Sketch the structure of dimethylglioxim.

3. Red strawberry precipitate formed from the reaction of dimethylglioxim and nickel(II) ion in base condition. Sketch the structure of *bis*-(dimetilglioksimato)nikel(II) ion.

Assistant's comment :

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WORKSHEET-11 **Day/Date of Practicum: ......……………….**

PREPARATION OF COORDINATION COMPOUND, $[Ni(NH_3)_6]I_2$

The observation of the existence of nickel(II) ion:

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Ionic reaction equation:

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The observation of iodide ion existence:

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Ionic reaction equation:

………………………………………………………………………………………

Mass of tube + cover = ……………………..

Mass of tube + cover + complex = ……………………..

Mass of complex = ……………………..

Theoretical yield = ……………………..

Percentage yield = ……………………..

On the iodide ion testing, sulfuric acid solution needs to be added. Why?

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On the nickel(II) ion testing, ammonia solution needs to be added. Why?

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Assistant’s comment :

Assistant Signature: .... Score: ....

WORKSHEET-12 **Day/Date of Practicum: ......……………….**

PURIFICATION OF KITCHEN SALT BY RE-CRYSTALLIZATION METHOD

1. Mass of resulted-pure crystal by evaporation method (Activity 2):
   NaCl = ………………….. gram = ……………………….. % (weight)
2. Mass of resulted-pure crystal by evaporation method (Activity 3):
   NaCl = ………………….. gram = ……………………….. % (weight)
3. Physical appearance (color/powder size/crystal) of resulted-crystal by the two methods:
   ……………………......................................……………………………………...
   ……………………………………………………………………………………
4. The aim of CaO or $Ba(OH)_2$ or $BaCl_2$ adding is: …… ……..…………………….
   ……………………………………………………………………………………
   ……………………………………………………………………………………
5. The aim of $(NH_4)_2CO_3$ adding is : ………………………………………………...
   ……………………………………………………………………………………
   ……………………………………………………………………………………
6. Neutralization by HCl solution suggests that all solutions are basic. The basic property comes from: …………………………………………………………….......................
   ....
7. Neutralization used HCl, instead of other acids. Why? …………………………………
   ……………………………………………………………………………………
8. Hydrochloric acid used to neutralize solution B (Activity 3.1). Why? ………..
   ……………………………………………………………………………………
9. Explain how to test the purity of resulted NaCl concisely.
   ……………………………………………………………………………………
   ……………………………………………………………………………………

Assistant's comment :

Assistant Signature: .... Score: ....