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Inorganic Chemistry

Practical I

The first Course

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2022 - 2023

مع تحيات مكتب البيت الهندسي للجامعة المستنصرية للطباعة والاستنساخ والهدايا / مجاور الباب الرئيسي للجامعة المستنصرية 07703890040

Periodic Table

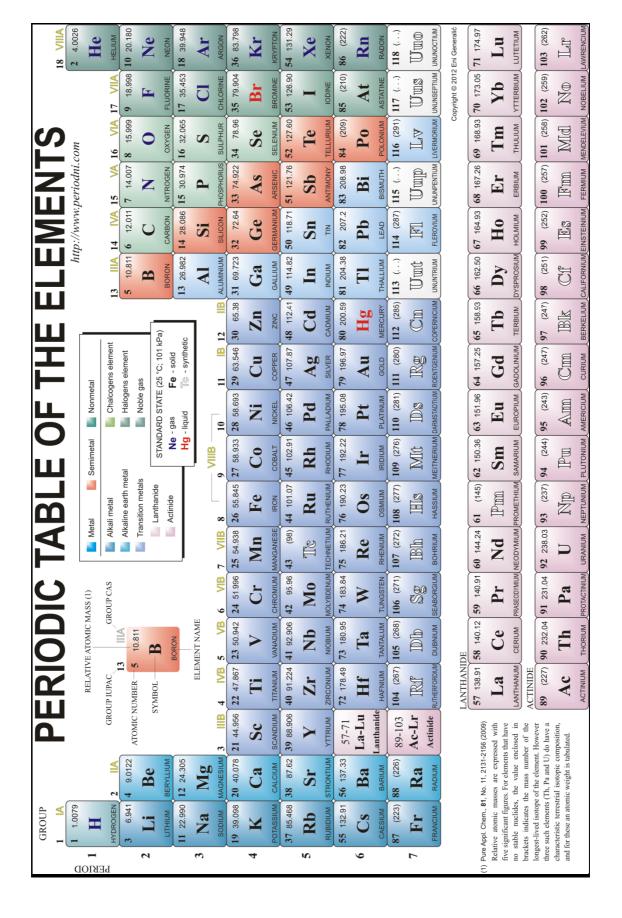


Table Illustrates Negative Anions

Anion	Name of Anion
Br ⁻¹	Bromide
Cl ⁻¹	Chloride
F ⁻¹	Fluoride
I ⁻¹	Iodide
Clo ₃ -1	Chlorate
Clo ₂ ⁻¹	Chlorite
Clo ⁻¹	Hypochlorite
CN ⁻¹	Cyanide
SCN ⁻¹	Thiocyanate
$C_2H_3O_2^{-1}$	Acetate
HSO ₃ -1	Hydrogen Sulfite
HSO ₄ -1	Hydrogen Sulfate
OH ⁻¹	Hydroxide
H ⁻¹	Hydride
NO ₃ -1	Nitrate
NO ₂ -1	Nitrite
$\mathrm{MnO_4}^{-1}$	Permanganate
10_{3}^{-1}	Iodate
IO ₄ -1	Periodate
CNO ⁻¹	Cyanate
O^{-2}	Oxide
S ⁻²	Sulfide
P ⁻³	Phosphsid
CO ₃ -2	Carbonate
CrO ₂ -1	Chromite
CrO ₄ -2	Chromate
$\operatorname{Cr}_2\operatorname{O}_7^{-2}$	Dichromate
HCO ₃ -1	Hydrogen Carbonate
$C_2O_4^{-2}$ SO_4^{-2} SO_3^{-2}	Oxalate
SO ₄ -2	Sulfate
SO ₃ -2	Sulfite
$S_2O_3^{-2}$	Thiosulfate
$S_4O_6^{-2}$ N^{-3}	Persulfate
N ⁻³	Nitride
PO ₄ -3	Phosphate
NH ₄ ⁺¹	Ammonium
NH ₂ -1	Amide

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Laboratory Reports:

Each report should include the following:

- 1. The title (Name of the Experiment).
- 2. Your name, student participating.
- 3. Data.
- 4. The theoretical part of experiment.
- 5. Experiment procedure: describe the steps taken the synthesis and isolate your products. Sometimes these steps, which you have actually taken, might be different from the laboratory procedures given to you. You have to express everything in your own words.
- 6. Results and calculations.
- 7. Discussion.

The First Group IA (1) Elements (Alkali Metals)

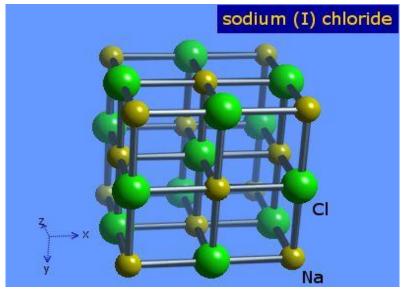
The Elements	Symbol	Electronic Structure
Lithium	₃ Li	$_{3}[He] 2S^{1}$
Sodium	₁₁ Na	$_{10}[Ne] 3S^1$
Potassium	₁₉ K	$_{13}[Ar] 4S^{1}$
Rubidium	₃₇ Rb	₃₆ [Kr] 5S ¹
Cesium	₅₅ Cs	₅₄ [Xe] 6S ¹
Francium	₈₇ Fr	$_{86}[Rn] 7S^1$

- 1. Why the alkali metals are soft and volatile?
- 2. Why they are highly electropositive?
- 3. Write down the electronic structure of Francium?
- 4. Why the first ionization energies of the group (IA) (1) are low atoms?
- 5. Why the chemical reactivity of the metals increases from Li to Cs?
- 6. How the charge-radius ratio of Li⁺ differs from those of the other group IA (1) ions?

Experiment No. (1)

Purification of Sodium ChlorideNaCl





$$2Na - 2e^{-} \rightarrow 2Na^{+}$$

$$\underline{Cl_{2} + 2e \rightarrow 2Cl^{-}}$$

$$2Na + Cl_{2} \rightarrow 2NaCl$$

The Reagents Required:

- 1. Crude salt (1g)
- 2. BaCl₂ solution 2%
- 3. Na₂CO₃ solution 5%
- 4. HCl solution (0.2M)

Procedure

- 1. Dissolve (1g) of Crude salt in (10) ml of Distilled water in a beaker.
- 2. Filter the solution and put the filtrate in a small beaker, then add drops of Barium chloride solution (2%) to the solution, observe the appearance of precipitate.
- 3. Filter the solution and put the filtrate in another beaker, repeat this step till no precipitate is formed.

$$\begin{bmatrix}
MgSO_4 \\
CaSO_4
\end{bmatrix}$$
 + BaCl₂ \longrightarrow BaSO₄ + $\begin{bmatrix}MgCl_2 \\
CaCl_2\end{bmatrix}$

4. Add sodium carbonate solution (5%) to the filtrate, observe the precipitation formation if precipitate formed then filter the solution.

$$\begin{bmatrix}
MgCl_2 \\
CaCl_2 \\
BaCl_2
\end{bmatrix} + Na_2CO_3 \longrightarrow CaCO_3 \\
BaCO_3
\end{bmatrix} + 2NaCl$$

5. Add few drops of diluted HCl (0.2M) to the filtrate to neutralize it using pH paper as indicator.

$$Na_2CO_3 + HCI \longrightarrow 2NaCI + CO_2 + H_2O$$

- 6. Evaporate the solution in a pre-weighted beaker using a mild heat source till pure crystals of NaCl appears.
- 7. Weight the yield then calculate the percentage of pure salt.

Percentage of Pure NaCl =
$$\frac{Mass\ of\ pure\ NaCl}{(1)g} \times 100$$

- 1. Why BaCl₂ and Na₂CO₃ are used? Explain with chemical equation.
- 2. Potassium carbonate cannot be used instead of sodium carbonate? Why? Explain with chemical equation.
- 3. Explain why the percentage of pure NaCl increases in the last step, with equation.
- 4. Give the properties of NaCl.
- 5. Write the equations of this reaction.

Experiment NO. (2)

Preparation and Standardization of Sodium Hydroxide

Sodium hydroxide, also known as lye and caustic soda is an inorganic compound with the formula NaOH. It is a white solid ionic compound consisting of sodium cations Na+ and hydroxide anions OH-.

Sodium hydroxide is a highly caustic base and alkali that decomposes proteins at ordinary ambient temperatures and may cause severe chemical burns. It is highly soluble in water, and readily absorbs moisture and carbon dioxide from the air. It forms a series of hydrates NaOH·nH2O. The monohydrate NaOH·H2O crystallizes from water solutions between 12.3 and 61.8 °C. The commercially available "sodium hydroxide" is often this monohydrate, and published data may refer to it instead of the anhydrous compound.

As one of the simplest hydroxides, sodium hydroxide is frequently used alongside neutral water and acidic hydrochloric acid to demonstrate the pH scale to chemistry students.

Sodium hydroxide is used in many industries: in the manufacture of pulp and paper, textiles, drinking water, soaps and detergents, and as a drain cleaner. Worldwide production in 2004 was approximately 60 million tons, while demand was 51 million tons.



The Required Reagents

25ml of Sodium Carbonate (0.5M).

0.72g of calcium hydroxide.

Distilled water.

Phenolphthalein as indicator 2-3 drops.

Hydrochloric acid (HCl 0.1M).

Procedure

- 1- Add 0.72g of Calcium Hydroxide to 25ml of Sodium Carbonate (0.5M) in a small beaker
- 2- Mark the level of mixture in the beaker then heat it for 10 minute
- 3- Add 25ml of Distilled water to the mixture
- 4- Turn the mixture to graduated cylinder (100ml) and record the height of precipitate column in different times from the time of transferring mixture to the cylinder (1,2,3,4,6,8) minute .leave the mixture to the next laboratory
- 5- Take 10ml of filtrate solution by using pipette to the conical flask then add 2-3 drops of Phenolphthalein as indicator and then titrate against the standard solution Hydrochloric acid (HCl 0.1M).
- 6- Record volume downward from the acid at the end point. and re-experiment to get two readings close

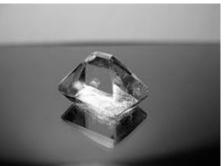
- 1- Write the balance equation of this reaction?
- 2- Draw agraph between height of precipitate column against times?
- 3- Give the reason why the mixture leaves to the next laboratory with cover it?
- 4- Prepare 0.1M standard solution Hydrochloric acid?
- 5- Calculate the concentration of sodium hydroxide?
- 6- Give the percentage of yield?
- 7- Prepare 0.5M of Sodium Carbonate?

Experiment No. (3)

Aluminum (III) Potassium Sulfate Dodecahydrate

Potassium alum, potash alum, tawas, or potassium aluminum sulfate is a chemical compound: the potassium double sulfate of aluminum. Its chemical formula is KAl(SO₄)₂, and it is commonly found in its dodecahydrate form as KAl(SO₄)₂.12(H₂O). Alum is the common name of this chemical compound, given the nomenclature of potassium aluminum sulfate dodecahydrate. It is commonly used in water purification, leather tanning, dyeing, fireproof textiles, and baking powder. It also has cosmetic uses as a deodorant, as an aftershave treatment and as a styptic for minor bleeding from shaving.

Potassium alum crystallizes in regular octahedra with flattened corners, and is very soluble in water. The solution reddens litmus and is an astringent. When heated to nearly a red heat it gives a porous, friable mass, which is known as "burnt alum". It fuses at 92°C (198°F) in its own water of crystallization. "Neutral alum" is obtained by the addition of as much sodium carbonate to a solution of alum as will begin to cause the separation of alumina.



Potassium Alum Crystal with Unequal Distribution of the Octahedral Face Area

PROPERTIES AND USES

- It is white crystalline solid.
- It is soluble in water.
- ◆ It is used for the purification of water.
- ◆ It is also used in leather industry and in paper industry.
- It is used in fire extinguisher.
- ♦ Melting point is 92°C

Types of Alum

- Potassium aluminum sulfate KAl(SO₄)₂·12H₂O
- Sodium aluminum sulfate NaAl(SO₄)₂·12H₂O,

- Ammonium aluminum sulfate NH₄Al(SO₄)₂·12H₂O
- Chromium potassium sulfate KCr(SO₄)₂·12H₂O
- Ammonium Ferric Sulfate NH₄Fe(SO₄)₂.12H₂O

The Reagents Required:

- Potassium sulfate K₂SO₄ (0.25 g).
- Distilled water.
- Aluminum sulfate $Al_2(SO_4)_3.18H_2O$ (0.9 g).
- Filter paper.

Procedure:

- 1) Dissolve (0.25 g) of potassium sulfate K_2SO_4 in (5 ml) distilled water.
- 2) Dissolve (0.9 g) of Aluminum sulfate hydrate Al₂(SO₄)₃.18H₂O in (10 ml) distilled water.
- 3) Mix the two solutions in one flask or until the next laboratory period.
- 4) Remove the flask overnight to get crystal formation throughout the liquid.
- 5) Filter the crystal and calculate the percentage yield of Alum.

% Yield =
$$\frac{Mass\ of\ Alum\ Obtained\ (g)}{Mass\ of\ theoretical\ Obtanable\ (g)} \times 100$$

Questions:

- 1. Give another method to prepare potassium Alum.
- 2. Calculate the mass of KAl(SO₄)₂.12H₂O (theoretical mass).
- 3. Calculate the percentage yield of Alum.
- 4. Draw the structure of potassium Alum.
- 5. Give another structure of Alum.

Another Method to Prepare KAl(SO₄)₂.12H₂O:

In this experiment you will prepare and characterize Alum (potassium aluminum sulfate dodecahydrate, KAl(SO₄)₂.12 H₂O. The first step in this synthesis, which you will perform during Week 1, is to react metallic aluminum with a concentrated solution of potassium hydroxide (KOH) to form the potassium salt of the tetrahydroxoaluminate complex ion, [Al(OH)₄]⁻. The balanced chemical equation for this oxidation-reduction reaction is:

$$2Al(S) + 2KOH_{(aq)} + 6H_2O(I) \rightarrow 2KAl(OH)_{4(qq)} + 3H_2(g)$$

The second step in the procedure is to convert the $KAl(OH)_4$ to Alum by adding sulfuric acid (H_2SO_4) in an acid-base reaction. Under the

experimental conditions, the Alum has a limited solubility in water, so it precipitates from the solution. The balanced chemical reaction that occurs in this step is

$$KAl(OH)_{4(aq)} + 2H_2SO_{4(aq)} + 8H_2O(I) \rightarrow KAl(SO_4)_2.12H_2O_{(S)}$$

The overall balanced chemical reaction for the conversion of aluminum to Alum, shown below, can be obtained by adding together the balanced chemical equation og each step.

$$2Al_{(S)} + 2KOH_{(aq)} + 22H_2O_{(I)} + 4H_2SO_{4(aq)}$$

 $\rightarrow 2KAl(SO_4)_2.12H_2O_{(S)} + 3H_{2(q)}$

The second and third weeks of this exercise will be devoted to characterize the Alum. Alum is an ionic compound, hence its melting and boiling points are likely to be too high to be conveniently measured. Also, most spectroscopic methods would not yield useful information. Therefore, we will rely on chemical means to show that we did, in fact, form Alum in our reaction. This procedure duplicates how chemists characterized chemical reactions until the late 20th century, and in some cases chemical means of characterization are still the only methods available.

Experiment No. (4)

Quantitative Determination of Water in aqueous salt: Reagents Required:

- 1. $KAl(SO_4)_2.12H_2O$
- 2. Crucible
- 3. Crucible tongs
- 4. Bunsen burner

Procedure:

- 1) Weight your Alum sample before starting this section.
- 2) Weight the clean and dry crucible and record this mass in your notebook.
- 3) Put the weight of your Alum in crucible and then heat it by Bunsen burner on the porcelain triangle supported by a ring and ring stand for few minutes until Alum properties change.
- 4) Cool the crucible to room temperature and then weight your crucible with anhydrous Alum, record the second mass in your notebook.

Calculations:

$$\begin{aligned} w_1 &= \textit{Mass of Alum} \\ w_2 &= \textit{Mass of Anhydrous Alum} \\ w_2 - w_1 &= w_3(\textit{The Mass of Water}) \\ \% H_2 O &= \frac{w_3}{w_1} \times 100 \\ \textit{Water Molecules Performuls} \\ \textit{Unit of Alum} &= \frac{w_2}{\textit{M.Wt of Unhydrous Alum}} \\ &= \frac{w_1}{\textit{M.Wt of Unhydrous Alum}} \end{aligned}$$

x = Water Molecules

- 1. Why we heated the crucible before using it?
- 2. What are the factors on which the number of water in hydrous Alum relays on?
- 3. What is the anhydrous Alum basic?
- 4. Give examples of hydrous salt?

Experiment No. (5)

Chromium (III) Potassium Sulfate Dodecahydrate

Chrome alum or chromium(III) potassium sulfate is the potassium double sulfate of chromium. Its chemical formula is $KCr(SO_4)_2$ and it is commonly found in its dodecahydrate form as $KCr(SO_4)_2.12(H_2O)$. It is used in leather tanning.

Chromium alum crystallizes in regular octahedra with flattened corners and it is very soluble in water. The solution reddens litmus and is an astringent. Its aqueous solutionis dark violet and turns green when it is dodecahydrate, above 50°C. addition the heated In to hexahydrateKCr(SO₄)₂.6H₂O, dihydrateKCr(SO₄)₂.2H₂O, and the monohydrate KCr(SO₄)₂.H₂O are known.



Chrome alum

Application:

Chromium potassium sulfate dodecahydrate is used in catalyst, mordant, ceramics, tannage, developer and dyes

The Reagent Required:

- 1. Potassium dichromate $K_2Cr_2O_7$ (1.75 g).
- 2. Sulfonic acid conc. H₂SO₄ (1.5 ml).
- 3. Absolute ethanol CH₃CH₂OH (3.5 ml).
- 4. Distilled water.

Procedure:

- 1) Dissolve (1.75 g) of potassium dichromate K₂Cr₂O₇ in (8 ml) of distilled water in a beaker and warm it with swirling until all solid compound is dissolved.
- 2) Add (1.5 ml) of sulfonic acid H_2SO_4 drop wise and carefully swirl the flask with stirring rod.
- 3) Add (3.5 ml) of absolute ethanol drop wise, during this addition a dark green solution will be formed.
- 4) Heat the solution in a water bath at (70°C) for 30 minutes.
- 5) Prepare an ice bath and put the solution, then you will notice formation of violate crystals.
- 6) Separate the crystals, then dry them, leave the solution until next week if the crystallization has not been performed.

- 1. Write the equation of this reaction.
- 2. Give the properties of chromium Alum.
- 3. What is the basic idea of this experiment? Explain.
- 4. What is the color of the yield? Why?

Experiment No. (6)

Detection of Cr³⁺, and Al³⁺ Ions:

Take (1 ml) of each solution KAl(SO₄)₂.12H₂O and KCr(SO₄)₂.12H₂O for each detection in a test tube by using several indicators as below:

$$KA(SO_{4})_{2}.12H_{2}O \xrightarrow{H_{2}O} [K(H_{2}O)_{6}]^{+} + [A(H_{2}O)_{6}]^{3+} + 2SO_{4}^{=}$$

$$KCr(SO_{4})_{2}.12H_{2}O \xrightarrow{H_{2}O} [K(H_{2}O)_{6}]^{+} + [Cr(H_{2}O)_{6}]^{3+} + 2SO_{4}^{=}$$

$$1 - Cr^{+3} + NaOH \xrightarrow{} ? \xrightarrow{excess} ?$$

$$2 - Cr^{+3} + CH_{3}COO^{-}Na^{+} \xrightarrow{} ?$$

$$3 - Cr^{+3} + Na_{2}CO_{3} \xrightarrow{} ?$$

$$4 - Cr^{+3} + NaOH \xrightarrow{} ? \xrightarrow{excess} ?$$

$$1 - A^{+3} + NaOH \xrightarrow{} ? \xrightarrow{excess} ?$$

$$2 - A^{+3} + CH_{3}COO^{-}Na^{+} \xrightarrow{} ?$$

$$3 - A^{+3} + Na_{2}CO_{3} \xrightarrow{} ?$$

$$4 - A^{+3} + Na_{2}HPO_{4} \xrightarrow{} ?$$

The Second Group IIA (2) (Alkaline Earth)

The Elements	Symbol	Electronic Structure		
Beryllium	₄ Be	$_2$ [He] $2S^2$		
Magnesium	₁₂ Mg	₁₀ [Ne] 3S ²		
Calcium	₂₀ Ca	$_{18}[Ar] 4S^2$		
Strontium	₃₈ Sr	36 [Kr] $5S^2$		
Barium	₅₆ Ba	$\int_{54} [Xe] 6S^2$		
Radium	₈₈ Ra	86[Rn] 7S ²		

- Name the important minerals of the group IIA (2) elements and write the electronic structure of the elements.
- Why these metals have higher melting points than the Alkali metals?

- Why beryllium tends to form covalent compound?
- How the solubility varies in group IIA (2)?
- Do alkaline earth cautions form many complexes? Which cation tends most to do so and what are the best complexing agent?

Experiment No. (7)

Determination of Magnesium Oxide Formula

Magnesium is a moderately reactive alkaline earth metal. (The alkaline earth's represents the second group IIA of the periodic table of elements. They are referred to as alkaline because their oxides are basic in water. The term "earth" was used by early alchemists to describe non-metals, the oxides in this case that did not melt in their furnaces. At room temperature, only Magnesium reacts very slowly with oxygen and can be kept for long periods of time without appreciable oxide build up. At elevated temperatures, however, Magnesium will ignite in an excess of oxygen gas, burning with an intensely white flame and producing Magnesium oxide. Because of the brightness of its flame, Magnesium is used in flares and in photographic flashbulbs.

Procedure:

- 1. Heat (0.1 g) of Magnesium using Bunsen burner in a closed crucible for 5 minutes.
- 2. The Magnesium will undergo with more controlled oxidation, gradually turning from shiny metal to grayish-white powder oxide.

The Reaction is:

$$Magnesium + Oxygen \rightarrow Mangnesium Oxide$$

$$2Mg + O_2 \longrightarrow 2MgO$$

3. Because the air also contains a great deal of nitrogen gas, put several drops of water in the crucible, careful heating with water to form magnesium nitride and ammonia.

 $Magnesium + Nitrogen \rightarrow Mangnesium Nitride$

$$Mg + N_2 \longrightarrow Mg_3N_2$$

4. By heating the product, the magnesium oxide will form.

$$Mg_3N_2 + 3H_2O \longrightarrow 3MgO + 2NH_3$$

Calculations:

We will use measured mass data to determine the empirical formula of magnesium oxide. This will give us an appreciation of the atomic make up of this compound.

- Mass Oxygen Consumed =
 Mass of Magnesium Oxide Mass of Magnesium
- 2) Mole Mg Atoms = $\frac{Mass\ of\ Mg}{Their\ Atomic\ Mass}$
- 3) $Mole\ O\ Atoms = \frac{Mass\ of\ O}{Their\ Atomic\ Mass}$

By dividing each result to less one we can form appropriate mole ratios.

Questions:

- 1. In our synthesis of magnesium oxide, why we do not count the mass of ammonia when considering the mass of final product? And how?
- 2. Give the reason why we add several drops of water to the crucible then heat it?
- 3. You have (59.95 g) of titanium and (40.05 g) of oxygen, give the mole ratio of each element and then give the formula of titanium oxide.

The atom mass of Ti=47.87 g/mole

4. Write the equation of the reaction.

Experiment No. (8)

Preparation of Barium Chloride Di-Hydrate BaCl₂.2H₂O

Barium chloride is an inorganic compoundwhose formulais BaCl₂. It is one of the most common water-soluble salts of barium. Like other barium salts, it is toxic and imparts a yellow-green coloration to a flame. It is also hygroscopic.

Preparation:

Barium chloride can be prepared from barium hydroxide or barium carbonate, with barium carbonate being found naturally as the mineral witherite. These basic salts react with hydrochloric acid to give hydrated barium chloride. On the industrial scale, it is prepared via two-step process from barite (barium sulfate):

$$BaSO_{4(s)} + 4 C_{(s)} \rightarrow BaS_{(s)} + 4 CO_{(g)}$$

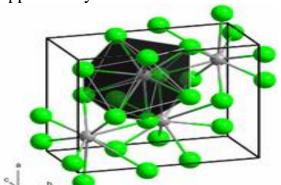
The first step requires high temperatures.

$$BaS + CaCl_2 \rightarrow BaCl_2 + CaS$$

The second step requires fusion of the reactants. BaCl₂ can then be leached out from the mixture with water. From water solutions of barium chloride, the dihydrate can be crystallized as white crystals: BaCl₂·2H₂O.

Uses:

As an inexpensive, soluble salt of barium, barium chloride finds wide application in the laboratory. It is commonly used as a test for sulfate ion. In industry, barium chloride is mainly used in the purification of brine solution in caustic chlorine plants and also in the manufacture of heat treatment salts, case hardening of steel, in the manufacture of pigments, and in the manufacture of other barium salts. BaCl₂ is also used in fireworks to give a bright green color. However, its toxicity limits its applicability.



The Required Reagents:

- 1. 5 ml of diluted HCl.
- 2. 2 ml of conc. HCl.
- 3. BaCO₃ (1 g).
- 4. Ice bath.

Procedure:

- 1) Dissolve (1 g) of BaCO₃ in 5 ml of diluted HCl with stirring.
- 2) Add (2 ml) of conc. HCl.
- 3) Boil the mixture.
- 4) Cool down the flask after filtering it in ice bath.
- 5) Collect the crystals by filtering then dry it at room temperature.
- 6) Weight the precipitate.

- 1. Write the equation of this reaction.
- 2. Why conc., and diluted HCl are used in this experiment?
- 3. Calculate the percentage of the yield.
- 4. Calculate the number of water molecules per formula.

Experiment No. (9)

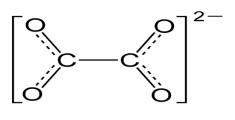
Preparation of Potassium TrisOxalato Aluminate (III) Trihydrate $K_3[Al(C_2O_4)_3].3H_2O$

Aluminium (or **aluminum**; see different endings) is a chemical element in the boron group with symbol **Al** and atomic number 13. It is a silverywhite, soft, nonmagnetic, ductilemetal. Aluminium is the third most abundant element (after oxygen and silicon) in the Earth's crust, and the most abundant metal there. It makes up about 8% by mass of the crust, though it is less common in the mantle below. Aluminium metal is so chemically reactive that native specimens are rare and limited to extreme reducing environments. Instead, it is found combined in over 270 different minerals.^[7] The chief ore of aluminium is bauxite.

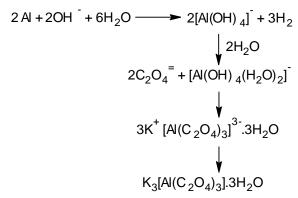
Aluminium is remarkable for the metal's low density and for its ability to resist corrosion due to the phenomenon of passivation. Structural components made from aluminium and its alloys are vital to the aerospace industry and are important in other areas of transportation and structural materials, such as building facades and window frames. [clarification needed] The most useful compounds of aluminium, at least on a weight basis, are the oxides and sulfates. [citation needed]

Oxalateethanedioate) is the dianion with the formula $C_2O_4^{2-}$, also written $(COO)_2^{2-}$. Either name is often used for derivatives, such as salts of oxalic acid, for example sodium oxalate $Na_2C_2O_4$, or dimethyl oxalate $((CH_3)_2C_2O_4)$. Oxalate also forms coordination compounds where it is sometimes abbreviated as **ox**.





The Equation of the Experiment:



The Required Reagents:

- 1. Aluminum shavings 0.05gr.
- 2. Potassium hydroxide 1.75 gr.
- 3. Oxalic acid dehydrates 0.75 gr.
- 4. Ethanol 10 drops.
- 5. Distilled water.

Procedure:

- 1. Dissolve 1.75 gr of potassium hydroxide in 4 ml of distilled water.
- 2. Put aluminum shavings 0.05 gr to the solution when the evolution of the hydrogen gas is stopped; heat the solution to boiling to dissolve aluminum.
- 3. Filter the solution and then add to the filtrate (0.75 gr of oxalic acid dissolve in 2.5 ml of distilled water) drop wise.
- 4. Cool the solution to room temperature by ice-bath, and then add 10 drops of ethanol. Scratch the beaker well and stir well to help crystallization.
- 5. Filter the crystals, wash them with ethanol and dry at room temperature.
- 6. Calculate the percentage of the compounds.

- 1. Why we use the KOH solution?
- 2. Why we add oxalic acid indirectly? Give the reason.
- 3. Why we heat the solution till boiling after adding the aluminum?
- 4. Draw the structure of the complex. Is this complex stable? Why? Give the hybridization, geometry of the complex.
- 5. Give the structure of oxalic $C_2O_4^=$.

Experiment No. (10)

Identification of Oxalate in their Complex as Below:

Take (1 ml) of the complex solution in taste tube for each detection by using several indicators.

1.
$$C_2O_4^{=} + AgNO_3 \longrightarrow ?$$

2.
$$C_2O_4^{=} + BaCl_2$$
 -> ?

3.
$$C_2O_4^{=} + CaCl_2 \longrightarrow ?$$

$$_{4}$$
, $C_{2}O_{4}^{=}$ + KMnO₄ \longrightarrow ? $\stackrel{\triangle}{\longrightarrow}$?

(15) drops of $KMNO_4 + (10)$ drops of Dil. H_2SO_4

Questions:

1. Give equation for:

$$_{a}$$
 $C_{2}O_{4}^{=}$ with BaCl₂

b.
$$C_2O_4^{=}$$
 with $CaCl_2$

- 2. Give the characteristic detection of $C_2O_4^{2-}$.
- 3. Can we use dil. HCl instead of H₂SO₄?
- 4. Give the equation of KMnO₄ treated with water.
- 5. Give the properties of KMnO₄.