Al-Mustnsiriyah University

College of Science

Department of Chemistry

1.500

Inorganic Chemistry

Practical II

The First Course

By

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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
MnO_4^{-1}	Permanganate
IO_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
$Cr_2O_7^{-2}$	Dichromate
HCO_3^{-1}	Hydrogen Carbonate
$C_2O_4^{-2}$	Oxalate
SO_4^{-2}	Sulfate
SO_3^{-2}	Sulfite
$S_2O_3^{-2}$	Thiosulfate
$S_4O_6^{-2}$	Persulfate
N^{-3}	Nitride
PO_4^{-3}	Phosphate
NH_4^{+1}	Ammonium
NH_2^{-1}	Amide

Contents

Laboratory Reports:

Each report should include the following:

1. The title (Name of the Experiment).
2. Your name, student number.
3. Data.
4. The theory of the 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] $3S^1$
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] $7S^1$

Questions:
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?

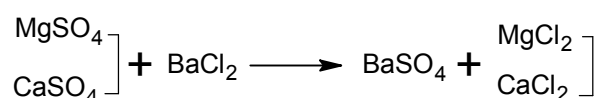# Experiment No. (1)
Purification of Sodium Chloride NaCl

sodium (I) chloride

$$2Na - 2e^- \rightarrow 2Na^+$$
$$\underline{Cl_2 + 2e \rightarrow 2Cl^-}$$
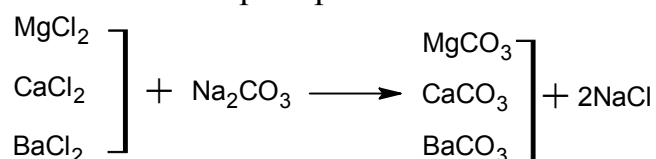$$Na + Cl_2 \rightarrow 2NaCl$$
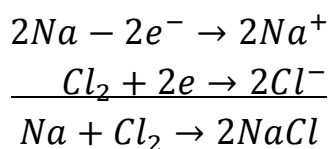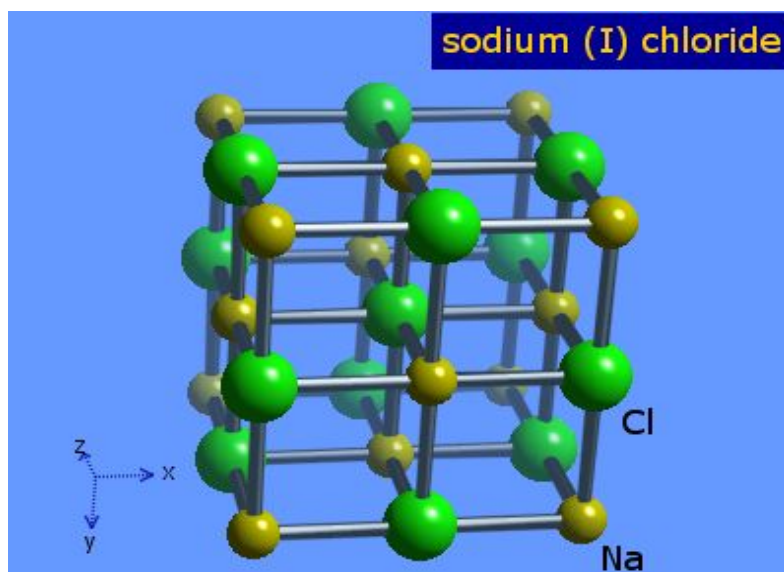
The Reagents Required:
1. Table salt (1g)
2. $BaCl_2$ solution 2%
3. Na_2CO_3 solution 5%
4. HCl solution (0.2M)

Procedure
1. Dissolve (1g) of table salt in (10) ml of 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.

$$\left.\begin{array}{c} MgSO_4 \\ CaSO_4 \end{array}\right] + BaCl_2 \longrightarrow BaSO_4 + \left[\begin{array}{c} MgCl_2 \\ CaCl_2 \end{array}\right.$$

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

$$\left.\begin{array}{c} MgCl_2 \\ CaCl_2 \\ BaCl_2 \end{array}\right] + Na_2CO_3 \longrightarrow \left[\begin{array}{c} MgCO_3 \\ CaCO_3 \\ BaCO_3 \end{array}\right. + 2NaCl$$

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

$$Na_2CO_3 + HCl \longrightarrow 2NaCl + CO_2\uparrow + H_2O\uparrow$$
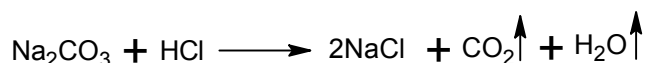
6. Evaporate the solution in a pre-weighted beaker using a mild heat source till pure crystals of NaCl appears.

7. Weight the yield then calculate the percentage of pure salt.

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

Question:
1. Why $BaCl_2$ and Na_2CO_3 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)

Aluminum (III) Potassium Sulfate Ddodecahydrate

Potassium alum, potash alum, tawas, or potassium aluminum sulfate is a chemical compound: the potassium double sulfate of aluminum. Its chemical formula is $KAl(SO_4)_2$, and it is commonly found in its dodecahydrate form as $KAl(SO_4)_2.12(H_2O)$. 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.

Octahedral Potassium Alum Crystal with Unequal Distribution of the 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 $92^{o}C$

Types of Alum
- Potassium aluminum sulfate ($KAl(SO_4)_2 \cdot 12H_2O$
- Sodium aluminum sulfate ($NaAl(SO_4)_2 \cdot 12H_2O$,
- Ammonium aluminum sulfate ($NH_4Al(SO_4)_2 \cdot 12H_2O$
- Chromium potassium sulfate ($KCr(SO_4)_2 \cdot 12H_2O$-
- Aluminum fluorosulfate ($FAl(SO_4)_2.12H_2O$.

The Reagents Required:

- Potassium sulfate K_2SO_4 (0.25 g).
- Distilled water.
- Aluminum sulfate $Al_2(SO_4).18H_2O$ (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 $Al_2(SO_4).18H_2O$ 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 $KAl(SO_4)_2.12H_2O$ (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 $KAl(SO_4)_2.12H_2O$:

In this experiment you will prepare and characterize Alum (potassium aluminum sulfate dodecahydrate, $KAl(SO_4)_2.12 \ H_2O$. 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:

$$2Al(S) + 2KOH_{(aq)} + 6H_2O(I) \rightarrow 2KAl(OH)_{4(aq)} + 3H_2(g)$$
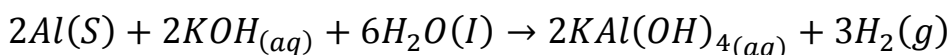
The second step in the procedure is to convert the $KAl(OH)_4$ to Alum by adding sulfuric acid (H_2SO_4) in an acid-base reaction. Under the experimental conditions, the Alum has a limited solubility in water, so it precipitates from the solution. The balanced chemical reaction that occurs in this step is

$$KAl(OH)_{4(aq)} + 2H_2SO_{4(aq)} + 8H_2O \ (I) \rightarrow KAl(SO_4)_2.12H_2O_{(S)}$$
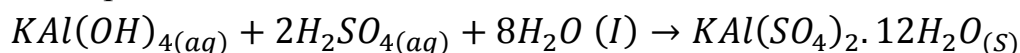
The overall balanced chemical reaction for the conversion of aluminum to Alum, shown below, can be obtained by adding together the balanced chemical equation og each step.

$$2Al_{(S)} + 2KOH_{(aq)} + 22H_2O_{(I)} + 4H_2SO_{4(aq)}$$
$$\rightarrow 2KAl(SO_4)_2.12H_2O_{(S)} + 3H_{2(g)}$$
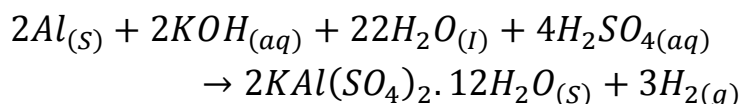
The second and third weeks of this exercise will be devoted to characterize the Alum. Alum is an ionic compound, hence its melting and boiling points are likely to be too high to be conveniently measured. Also, most spectroscopic methods would not yield useful information. Therefore, we will rely on 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. (3)

Quantitative Determination of Water of Hydrate:
Reagents Required:
1. $KAl(SO_4)_2.12H_2O$
2. Crucible
3. Crucible tongs
4. Bunsen burnet

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 about (1.0) of your Alum and then heat the crucible 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:
$w_1 = Mass\ of\ Alum$
$w_2 = Mass\ of\ Anhydrous\ Alum$
$w_2 - w_1 = w_3\ (The\ Mass\ of\ Water)$
$\%H_2O = \dfrac{w_3}{w_1} \times 100$

$$Water\ Molecules\ Performuls\ Unit\ of\ Alum = \dfrac{w_2}{M.Wt\ of\ Unhydrous\ Alum}$$
$$= \dfrac{w_1}{M.Wt\ of\ Unhydrous\ Alum + 18x}$$

$x = Water\ Molecules$

Questions:
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. (4)

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 solution is dark violet and turns green when it is heated above 50°C. In addition to the dodecahydrate, the hexahydrate $KCr(SO_4)_2.6H_2O$, dihydrate $KCr(SO_4)_2.2H_2O$, and the monohydrate $KCr(SO_4)_2.H_2O$ are known.

Chrome alum

Application:

Chromium potassium sulfate dodecahydrate is used in catalyst, mordant, ceramics, tannage, developer and dyes
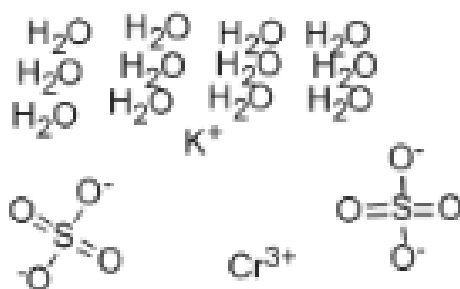
The Reagent Required:
1. Potassium dichromate $K_2Cr_2O_7$ (1.75 g).
2. Sulfonic acid conc. H_2SO_4 (1.5 ml).
3. Absolute ethanol CH_3CH_2OH (3.5 ml).
4. Distilled water.

Procedure:

1) Dissolve (1.75 g) of potassium dichromate $K_2Cr_2O_7$ 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^oC) 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.
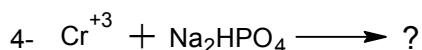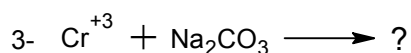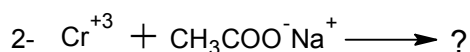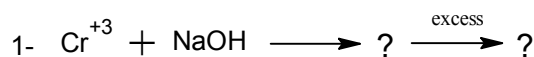
Questions:

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. (5)

Detection of Cr^{3+}, and Al^{3+} Ions:

Take (1 ml) of each solution $KAl(SO_4)_2.12H_2O$ and $KCr(SO_4)_2.12H_2O$ for each detection in a test tube by using several indicators as below:

$$KAl(SO_4)_2.12H_2O \xrightarrow{H_2O} [K(H_2O)_6]^+ + [Al(H_2O)_6]^{3+} + 2SO_4^=$$

$$KCr(SO_4)_2.12H_2O \xrightarrow{H_2O} [K(H_2O)_6]^+ + [Cr(H_2O)_6]^{3+} + 2SO_4^=$$

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

2- $Cr^{+3} + CH_3COO^-Na^+ \longrightarrow ?$

3- $Cr^{+3} + Na_2CO_3 \longrightarrow ?$
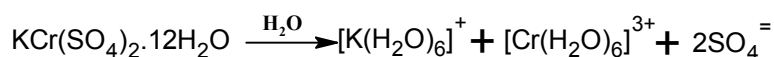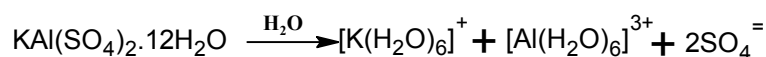
4- $Cr^{+3} + Na_2HPO_4 \longrightarrow ?$

1- Al^{+3} + NaOH $\longrightarrow$? $\xrightarrow{\text{excess}}$?

2- Al^{+3} + $CH_3COO^-Na^+$ $\longrightarrow$?

3- Al^{+3} + Na_2CO_3 $\longrightarrow$?

4- Al^{+3} + Na_2HPO_4 $\longrightarrow$?

The Second Group IIA (2) (Alkaline Earth)

The Elements	Symbol	Electronic Structure
Beryllium	$_4Be$	$_2[He]\ 2S^2$
Magnesium	$_{12}Mg$	$_{10}[Ne]\ 3S^2$
Calcium	$_{20}Ca$	$_{18}[Ar]\ 4S^2$
Strontium	$_{38}Sr$	$^{36}[Kr]\ 5S^2$
Barium	$_{56}Ba$	$_{54}[Xe]\ 6S^2$
Radium	$_{88}Ra$	$_{86}[Rn]\ 7S^2$

Questions:
-   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. (6)
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.
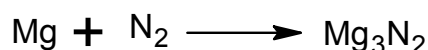
The Reaction is:

$$Magnesium + Oxygen \rightarrow Mangnesium\ Oxide$$

$$2Mg + O_2 \longrightarrow 2MgO$$
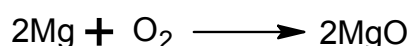
3. Because the air also contains a great deal of nitrogen gas, put several drops of water in the crucible, careful heating with water to form magnesium nitride and ammonia.

$$Magnesium + Nitrogen \rightarrow Mangnesium\ Nitride$$

$$Mg + N_2 \longrightarrow Mg_3N_2$$

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

$$Mg_3N_2 + 3H_2O \longrightarrow 3MgO + 2NH_3$$
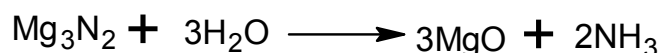
Calculations:
We will use measured mass data to determine the empirical formula of magnesium oxide. This will give us an appreciation of the atomic make up of this compound.

1) $Mass\ Oxygen\ Consumed =$
   $Mass\ of\ Magnesium\ Oxide - Mass\ of\ Magnesium$

2) $Mole\ Mg\ Atoms = \dfrac{Mass\ of\ Mg}{Their\ Atomic\ Mass}$

3) $Mole\ O\ Atoms = \dfrac{Mass\ of\ O}{Their\ Atomic\ Mass}$

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

 $\qquad\qquad$ O=16 g/mole
4. Write the equation of the reaction.

Experiment No. (7)

Preparation of Barium Chloride Di-Hydrate $BaCl_2.2H_2O$

Barium chloride is an inorganic compound whose formula is $BaCl_2$. 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):

$$BaSO_{4(s)} + 4\ C_{(s)} \rightarrow BaS_{(s)} + 4\ CO_{(g)}$$
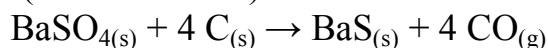
The first step requires high temperatures.

$$BaS + CaCl_2 \rightarrow BaCl_2 + CaS$$
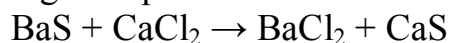
The second step requires fusion of the reactants. $BaCl_2$ can then be leached out from the mixture with water. From water solutions of barium chloride, the dihydrate can be crystallized as white crystals: $BaCl_2 \cdot 2H_2O$.

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. $BaCl_2$ is also used in fireworks to give a bright green color. However, its toxicity limits its applicability.
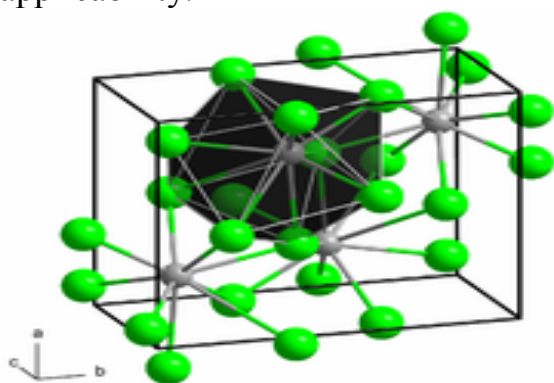
The Required Reagents:
1. 5 ml of diluted HCl.
2. 2 ml of conc. HCl.
3. $BaCO_3$ (1 g).
4. Ice bath.

Procedure:
1) Dissolve (1 g) of $BaCO_3$ in 5 ml of diluted HCl with stirring.
2) Add (2 ml) of conc. HCl.
3) Boil the mixture and then filter it.
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.

Questions:
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. (8)

Preparation of Calcium Peroxide CaO$_2$

Calcium peroxide or Calcium dioxide (CaO$_2$) is a solid peroxide with a white or yellowish color. For all practical purposes calcium peroxide is insoluble in water but it can dissolve in acid to form hydrogen peroxide. When in contact with water it will immediately begin to decompose releasing oxygen.
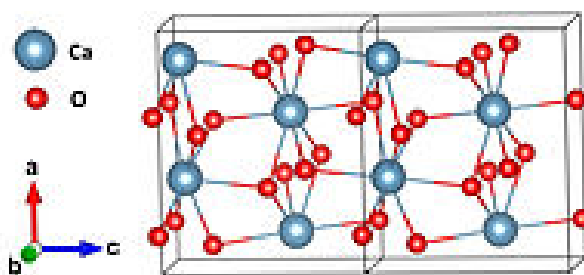
Preparation:
Calcium peroxide is created by the interactions of solutions of calcium salt and sodium peroxide, with subsequent crystallization. The octahydrate is synthesized by the reaction of calcium hydroxide with dilute hydrogen peroxide.

Applications:
Calcium peroxide is manufactured to varying specifications and purity and can be used in different areas of industry and agriculture. In agriculture, it is used as an oxygen fertilizer, and is also used in the presowing treatments of rice seeds. Also, calcium peroxide has uses in the aquaculture industry as it is used to oxygenate and disinfect water, and in the ecological restoration industry as it is used in the treatment of soils. Calcium Peroxide is used in a similar manner of magnesium peroxide for environmental restoration programs. It is used to restore soil and groundwater contaminated with petroleum hydrocarbons by stimulating aerobic microbial degradation of the contaminants in a process known as Enhanced In-Situ Bioremediation.

As a food additive it has the E number E930 and is used as flour bleaching agent and improving agent.

The Required Reagents:
1. Calcium carbonate $CaCO_3$ (0.5 g).
2. Conc. HCl (2 ml).
3. Hydrogen peroxide (4 ml).
4. Conc. Ammonia.
5. Distilled water.

Procedure:
1) Mix in a beaker (2 ml) of distilled water with (2 ml) of conc. HCl, and cover the beaker with a watch glass.
2) Add 0.5 g) of calcium carbonate in a small potions.
3) Boil the mixture to expel the produced carbon dioxide, then add (0.5 g) of calcium carbonate until remains dissolved.
4) Filter the hot solution, then cool the filtrate down, and complete its volume to (2 ml) distilled water.
5) Cool the solution in ice-bath, then add (4 ml) of H_2O_2 60%.
6) Prepare (10 ml) of solution of dilute ammonia by adding (8 ml) of distilled water, and (2 ml) of conc. Ammonia, then add this solution to the solution mixture of calcium chloride-hydrogen peroxide and stir until calcium peroxide is crystalized.
7) Collect the crystals by filteration then wash it with cold water and dry it in room temperature.
8) Calculate the percentage of the yield.

$$Yield \% = \frac{Mass\ of\ CaO_2}{The\ Thiory\ Mass} \times 100$$

Questions:
1. Write the equation of this reaction.
2. Why conc. HCl is used in this experiment?
3. Why ammonia solution is used?
4. Calculate the theory mass of calcium peroxide.

The Third Group III B (B)

Elements	Symbol	Electronic structure
Boron	$_5B$	$_2[H]\ 2s^2\ 2p^1$
Aluminum	$_{13}Al$	$_{10}[Ne]\ 3s^2\ 3p^1$
Gallium	$_{31}Ga$	$_{18}[Ar]\ 3d^{10}\ 4s^2\ 4p^1$
Indium	$_{49}In$	$_{36}[Kr]\ 4d^{10}\ 5s^2\ 5p^1$
Thallium	$_{81}Tl$	$_{54}[Xe]\ 4F^{10}\ 5d^{10}\ 6s^2\ 6p^1$

Questions:

1. The oxidative state of (+1) can be more stable from B→Tl, why?
2. The elements are more ionic from B→Tl, why?
3. The elements are more metallic from B→Tl, why?
4. Give the reason for electronically of these elements subdivided into two classes.
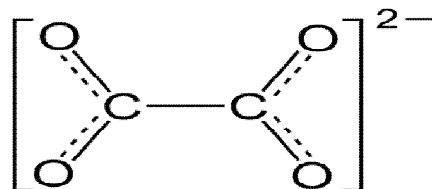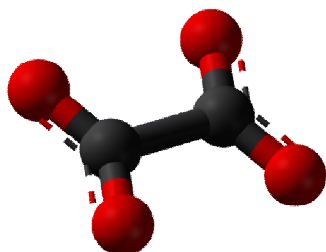5. Write down the electronic structure of Thallium?

Experiment No. (9)

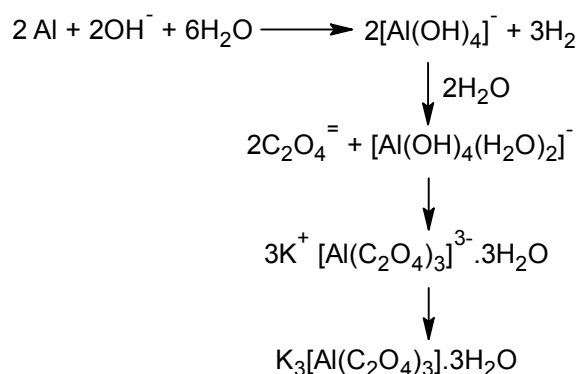## Preparation of Potassium Tris Oxalato Aluminate (III) Trihydrate $K_3[Al(C_2O_4)_3].3H_2O$

Aluminium (or **aluminum**; see different endings) is a chemical element in the boron group with symbol **Al** and atomic number 13. It is a silvery-white, soft, nonmagnetic, ductile metal. 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]

Oxalate ethanedioate) 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:

$$2\,Al + 2OH^- + 6H_2O \longrightarrow 2[Al(OH)_4]^- + 3H_2$$

$$\downarrow 2H_2O$$

$$2C_2O_4^{=} + [Al(OH)_4(H_2O)_2]^-$$

$$\downarrow$$

$$3K^+ [Al(C_2O_4)_3]^{3-}.3H_2O$$

$$\downarrow$$

$$K_3[Al(C_2O_4)_3].3H_2O$$

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.

Questions:
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. $C_2O_4^= + BaCl_2 \longrightarrow$?
3. $C_2O_4^= + CaCl_2 \longrightarrow$?
4. $C_2O_4^= + KMnO_4 \longrightarrow$? $\xrightarrow{\Delta}$?

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

Questions:
1. Give equation for:
   a. $C_2O_4^=$ with $BaCl_2$
   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 H_2SO_4?
4. Give the equation of $KMnO_4$ treated with water.
5. Give the properties of $KMnO_4$.

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