INORGANIC CHEMISTRY

Second stage-Food technology department

2023-2024

MSC.SAWEN M.EZZALDDIN

Experiment:1

Preparation of Potassium dichromate ($K_2Cr_2O_7$)

Potassium dichromate is prepared from chromite ore ($FeCr_2O_4$) ın the following steps

Step (1):
Preparation of sodium chromate
$4FeCr_2O_4 + 16NaOH + 7O_2 \rightarrow 8Na_2CrO_4 + 2Fe_2O_3 + 8H_2O$

Step (2):
Conversion of sodium chromate into sodium dichromate
$2Na_2CrO_4 + conc.H_2SO_4 \rightarrow Na_2Cr_2O_7 + Na_2SO_4 + H_2O$

Step(3):
Conversion of sodium dichromate to potassium dichromate
$Na_2Cr_2O_7 + 2KCl \rightarrow K_2Cr_2O_7 + 2NaCl$

Potassium dichromate being less soluble than sodium chloride is obtained in the form of orange coloured crystals and can be removed by filtration.

The dichromate ($Cr_2O_7^{2-}$) ion exists in equilibrium with chromate (CrO_4^{2-}) ion at pH 4. However, by changing the pH, they can be interconverted.

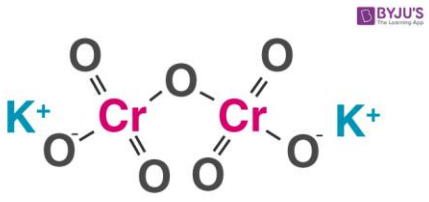
$2CrO_4^{2-} + 2H^+ \longrightarrow Cr_2O_7^{2-} + H_2O$ Dichromate (orange)

$Cr_2O_7^{2-} + 20H^- \longrightarrow 2CrO_4^{2-} + H_2O$ Chromate (yellow)

Yellow chromate changes into orange dichromatc in acid medium and dichromate changes into chromate in basic medium

Structure of Potassium Dichromate Molecules

Potassium dichromate is an ionic compound consisting of two potassium cations and one dichromate anion. The coordination geometries around the chromium atoms are tetrahedral. The structure of a potassium dichromate molecule is illustrated below.



.

Physical Properties:

1. It forms orange-red crystals which melt at 396°C.
2. It is easily soluble in hot water but moderately soluble in cold water.

Chemical Properties

1-Action of heat

Potassium dichromate decomposes on heating to irom potassium chromate, chromic oxide and oxygen.

$$4K_2Cr_2O_7 \longrightarrow 4K_2CrO_4 + 2CrO_3 + 3O_2$$

2- Oxidization:
$K_2Cr_2O_7$ is known as a powerful oxidizing agent.

$$K_2Cr_2O_7 + 7\ H_2SO_4 + 6KI \rightarrow 4\ K_2SO_4 + Cr_2(SO_4)_3 + 3I_2 + 7H_2O$$

3- Chromyl Chloride Test:

When salt containing chloride is treated with $K_2Cr_2O_7$ and con. H_2SO_4 chromyl chloride (reddish brown) vapors are produced.

$$K_2Cr_2O_7 + 4KCl + 6\ H_2SO_4 \rightarrow 2CrO_2Cl_2 + 6KHSO_4 + 3H_2O$$

Chromyl chloride

The reaction is used to detect chloride ions in qualitative analysis.

Uses of potassium dichromate

Sodium and potassium dichromate are uses as :

1. Strong oxidizing agents.
2. Primary standard material in volumetric analysis.
3. It is used to stain certain type of woods to produce deep, rich browns.
4. It is used to clean glassware material.
5. Used in pyrotechnic displays along with iron and tungsten.
6. Medically it can be used externally as an antiseptic and astringent.

Safety hazards

Potassium dichromate is a hexavalent chromium compound, hence it is highly toxic and carcinogenic. Potassium dichromate is very corrosive by nature and it can cause severe irritation in the eye and skin, burning sensation, and even lead to blindness. It is referred to as affecting reproductive health and it acts as a mutagenic agent i.e. it affects the genetic material and harms unborn children.

Proceduer

1- Add 1.8 gm of potassium chloride to 8 ml of D.W then heat the mixture until the salt dissolved.

2- Add 2 gm sodium dichromate to 4 ml D.W then heat the solution to dissolve the salt then add it to the first solution.

3- Stir the solution well the cool with tap water until crystals appear.

4- Collect the precipitate on a filter papar, dry it and finally record yield

Homework:

- Sodium dichromate more soluble than potassium dichromate. Why?
- Dichromate use as primary standard material in titration?
- During the preparation, KCl dissolve in D.W with out heating while sodium dichromate must be heated to dissolve. Explain that.

Experiment:2
Solubility ot KCl at different temperature

Solubility: the maximum amount of a substance that will dissolve in a given amount of solvent at a specified temperature.(the number of grams of the solute required to saturate 100g of the solvent at a particular temperature.)
Solutions can be classified into 3 types on the bases of an of present in the solution.

1- **Unsaturated Solutions**: the solution can still dissolve more solute in solvent.

2- **Saturated Solution**: a solution in which a solvent is not capable of dissolving any more solute at a given temperature.

3- **Supersaturated Solutions**: The solution in which solute is present in an excess amount, and are dissolved in the solvent forcefully by raising the temperature.

These excess solute particles are later found in the form of crystals with the help of the crystallization process.

Temperature plays an important role in solubility because the solubility of a substance is different at different temperatures. The solubility of solids and liquids generally increases with increasing temperature. There are many exceptions to this general rule; for examp\e lithium carbonate decreases in solubility as the solution temperature increases and sodium chloride shows practically no change.

Importance of Solubility Curve

Solubility curve can be used to determine the amount of substance deposited when the solution is cooled. Solubilities of different substances at a particular temperature can be determined. The importance of solubility curve is listed below.

- The solubility of a substance at a particular temperature can be determined.
- The solubility curve helps us to predict which substance will crystallize out first from a solution containing two or more solutes.
- The solubility curve helps us to compare the solubilities of different substances at the same temperature.
- It gives a clear idea that solubility of substance changes with the temperature.

Procedure

1- Put 2gm of potassium chloride in large test tube then add 20 ml of distilled water the volume of solution must be constant before each new addition.

2- Heat the mixture in a water bath until dissolve salt then put a thermometer in the solution.

3- Cool the solution and record the temperature of the appearance of first crystal.

4- Repeat the previous step by adding four different quantities of the potassium chloride on condition that final amount kCl is 8gm in 20ml of the used water.

Homework:

- Define solution?
- What is the different between solution and mixture?
- Is temperature effect the solubility?
- Explain the effect of concentration on solubility at a given temperature.

Experiment:3
Cosak Method

Cosak Method

In Bronshted-Lory view, an acid is any substance that is capable of donating a proton. Base is any substance that can accept a proton.

$$Ca(OH)_2 + Na_2CO_3 \xrightarrow{heat} CaCO_3 + 2\,NaOH$$

PPt.

Procedure:

1- Add 8gm calcium hydroxide $Ca(OH)_2$ to 100 ml of Na_2CO_3 Solution (0.5M).

2- Heat the solution to boiling point about 10 minute.

3- Transfer the solution to cylinder 100 ml and left to one hour.

4- Separate the filtrate from the precipitation by decantation process.

5- Evaporate the filtrate by hot plate and weight the product.

Homework:

- Write three main different between acid and base compounds.
- Calcium carbonate is non soluble in water. Explain.
- NaOH is known as a strong electrolyte. Why?

Experiment:4
Preparation of Copper (I) iodide

Cuprous Iodide (**Copper (I) iodide**) is the chemical compound with the formula CuI; it is also known as cuprous iodide. Copper (I) iodide is useful in a variety of applications ranging from organic synthesis to cloud seeding. Copper (I) iodide is white, but samples are often tan or even, when found in nature as rare mineral marshite, reddish brown, but such color is due to impurities. It is common for iodides to become discolored because of the easy oxidation of the iodide anion to iodine. Copper (I) iodide can also be prepared by heating iodine and copper in concentrated hydriodic acid, HI.

In the laboratory, copper(I) Iodide is prepared by simply mixing an aqueous solutions of sodium or potassium iodide and a soluble copper(II) salt such copper sulfate.

$$Cu^{2+} + 2I^{-} \rightarrow CuI_2$$

The CuI_2 immediately decomposes to iodine and insoluble copper(I) iodide, releasing I_2.

$$2\ CuI_2 \rightarrow 2\ CuI + I_2$$

This reaction has been employed as a means of assaying copper(II) samples, since the evolved I2 can be analyzed by redox titration. Copper(I) iodide can also prepared by heating iodine and copper in concentrated hydriodic acid.

CuI is poorly soluble in water (0.00042 g/L at 25 °C), but it dissolves in the presence of NaI or KI to give the linear anion $[CuI_2]^{-}$. Dilution of such solutions with water reprecipitates CuI. This dissolution-precipitation process is employed to purify CuI, affording colorless samples.

Properties

Copper iodide is a dense, pure white solide crystallizing with a zincblende structure. It is less sensitive to light than either the chloride or bromide. It is insolubile in water and dilute acide, but dissolve in aqueous solution of ammonia, potassium iodide, potassium cyanide, and sodium thiosulfate. It is decomposed by concentrated sulfuric acid and nitric acid.

Uses of CuI:

1- CuI is used as a reagent in organic synthesis

2- CuI is used in cloud seedin

3- The structural properties of cuI allow cuI to stabilize heat in nylon

4- is used in designing and synthesizing cu(I) clusters, which is polymetal complex compound.

Procedure:

1- weight out 3gm of copper sulfate pentahydrate and dissolve it in

20ml of water.

2- A second solution is prepared by placing 4.55 gm of potassium iodide in a beaker and dissolved in 12ml of water with shaking thoroughly.

3- The second solution is added to the first solution from a burette continuous rapidly stirring, and then 3.5 gm of $NaS_2O_3.5H_2O$ is added to the mixture with continuous stirring.

4- The dense white precipitation is allowed to settle for 15 minute and then collected by filtration.

5- Wash the precipitate with few drop of water, ethanol.

6- Dire and weight the product.

Homework:

- What is the type of the reaction of CuI preparation?
- Show the aim of adding sodium thiosulfate before collecting CuI precipitate?
- Copper (I) iodide is more stable than copper(I) fluoride. Why?

Experiment:5

Preparation of Chrome Alum

Theory: The crystals of chrome alum may be prepared by reducing an acidified solution of potassium dichromate. The reducing can be carried either using ethyl alcohol or starch solution.

$$K_2Cr_2O_7 \longrightarrow \cancel{K_2O} + \cancel{Cr_2O_3} + 3O$$

$$\cancel{K_2O} + H_2SO_4 \longrightarrow K_2SO_4 + H_2O$$

$$\cancel{Cr_2O_3} + H_2SO_4 \longrightarrow Cr(SO_4)_3 + 3H_2O$$

$$\{ CH_3CH_2OH + \cancel{O} \longrightarrow CH_3CHO + H_2O \} \times 3$$

$$K_2Cr_2O_7 + H_2SO_4 + 3C_2H_5OH \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O + 3CH_3CHO$$

Reagents. Potassium dichromate 5.0g

Conc. H2SO4	4ml
Water	20ml
Alcohol	3ml
Starch	1.5g

Procedure:

1- Weight 5g potassium dichromate and dissolve in 20ml of D.W. Then add dropwise 4ml conc. H_2SO_4 with stirring till all the solid dichromate dissolve.

2- Cool the solution by keeping the beaker in an ice bath. Then add dropwise of 3ml of ethyl alcohol.

3- Cool solution in ice bath and the contents of the beaker allow to stand over-night, when crystal of crome alum separate out. Separate crystals from mother liquor, wash with a small amount of cold water, dry and weight product.

Homework:

- Why is the mixture of $K_2Cr_2O_7$ with H_2SO_4 should be cooled?
- Show the reason of using of ethyl alcohol (ethanol) or starch solution during chrome alum preparation?