# PRINCIPLES OF QUALITATIVE INORGANIC ANALYSIS:

## PRECIPITATION, SEPARATION AND IDENTIFICATION OF CATIONS

**Huda S. Alhasan**
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**Bentham Books**

# Principles of Qualitative Inorganic Analysis: Precipitation, Separation and Identification of Cations

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# Principles of Qualitative Inorganic Analysis: Precipitation, Separation and Identification of Cations

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# FOREWORD

Analytical chemistry is divided into qualitative and quantitative analysis. Qualitative analysis is a sequential set of steps in order to characterize the type of substances that are included in the components of the mixture. The qualitative inorganic analysis is used to demonstrate the containment or non-containment of inorganic elements, ions, or compounds in an unknown sample, for example, an analyst working in the environmental field may test a water sample to confirm whether this sample contains dissolved ions of mercury, lead, or barium.

Nowadays, advanced laboratories and scientific companies perform many qualitative analyses effectively using methods, such as infrared and mass spectroscopy, nuclear magnetic resonance, neutron activation analysis, X-ray diffraction, spectroscopy, chromatography, electrophoresis, and others, despite that qualitative analysis with traditional methods is still very important, whether for field tests or the initial examination of instruments, as well as for students in the early stages of learning. This book is divided into ten chapters. The first chapter describes the method and laboratory safety. The second chapter includes an explanation of the basic principles of qualitative analysis, while chapters 3-8 cover several topics that are important in understanding how a particular analytical method works for diagnosing cations in samples, starting from sedimentation and ending with the identification. As for the ninth chapter, it included a summary of diagnosing cations with some given questions about all chapters. Finally, the book concludes with many important tables and information for both students and researchers.

Although this book is primarily designed for students in the first stage and academic teachers, it may also prove useful in teaching staff and workers in different fields of science and industry, because it contains scientific information that helps in understanding the subject of inorganic cations in analytical chemistry. We hope that the book fulfilled the aspirations of the authors.

N.A/ Naser

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# PREFACE

In this practical book, we tried to complete the basics of analytical chemistry. The first chapter describes the laboratory methods and chemical safety with an explanation of how to write the laboratory report. The next five chapters follow the detection of acidic fissures in simple solid compounds or in mixtures. It provides the most important interactions of common cations, which are divided into five groups depending on solubility in a particular solvent. We have taken into consideration the presentation of this book for a student with a detailed explanation of the theoretical basis for each group, which is carried out by the student so that he can recognize the importance of practical experiments and their relationship with theoretical study, while Chapter VII has included observations and general questions. This book is important for undergraduate students (the first class) for each Faculty of Science and some departments of the faculties of engineering and all faculties of the medical group as well as medical institutes. Therefore, we considered writing this book, which is a modest effort to advance the wheel of authorship in the Arab world, in general, and Iraq and Saudi Arabia in particular.

In conclusion, I would like to thank the Ministry of Higher Education and Scientific Research and the University of Babylon/Environmental Research and Studies Centre in Iraq, and the University of Jeddah in Saudi Arabia for providing opportunities to write this book and their continued support for scientific and educational studies.

## CONSENT FOR PUBLICATION

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## CHAPTER 1

# General Guidelines for Laboratory Work

**Abstract:** Chapter one gives the students the information that they need to bear in mind before entering the laboratory. Moreover, students will learn about the safety rules and be familiarized with the laboratory techniques and apparatus. Chapter one will give instructions about recording observations and writing laboratory reports.

**Keywords:** Apparatus, Laboratory reports, Safety, Work techniques.

## GENERAL GUIDELINES FOR LABORATORY WORK

### Instructions for Students

1. The student should wear a lab coat to protect his clothing from pollution and corrosion due to handling concentrated acids and chemicals.
2. Do not smoke and eat food and drink in the laboratory at all.
3. Ensure, upon entering the laboratory, the safety of all electrical and gas connections and the operation of air vacuums.
4. Determine in advance what you want to run from experiments and according to known and specific steps.
5. When the acid is diluted, add the acid to the water in small quantities gradually and make sure that the heat of the diluted solution is not warm (because the process is Exothermic).
6. Use Rubber pipetting bulbs with a pipette when you transfer chemical solutions.
7. When you want to get rid of acid or alkaline solution residues, open the water in the drainage basin before you pour it in and follow with strong water flow.
8. Do not waste the exhaust of chemical compounds that can interact with the water pipes in the drainage basin, but put them in special baskets or containers to be executed in appropriate ways.
9. Be sure to read what is written on the bottles containing chemicals, whether their names or compositions or instructions for how to use or warning of certain risks associated with their use.
10. Return the reagent bottles to their locations after you use them and make sure they are closed.
11. Take only the chemicals you need during weight without loss or waste, and so on for solutions.

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12. Take notes about the experiment and write your results.
13. After finishing work, return the equipment that you used and clean your place well and make sure to close the gas socket you used [1, 2].

## GUIDELINES FOR WORKING INSIDE THE LABORATORY

1. Use small quantities for reagents and chemicals, may be one drop or two drops of reagent are enough for the interaction to get the desired result.
2. The experiments may be a failure as the result of the contamination of your equipment or the glassware that you use; therefore, make sure you clean your apparatus before starting experiments.
3. All necessary precautions should be taken when using toxic and flammable materials. Be careful not to expose flammable material such as benzene or ether to direct flame. This may lead to a fire.
4. All reactions that result in the rise of vapors or gases that are harmful, toxic or irritating to the lungs or eyes, must be conducted in the fume hood.
5. Place only the necessary equipment and the glassware on the laboratory bench that you use in the laboratory.
6. When heating a solution or liquid in a test tube, do not direct a test tube toward yourself or toward your colleague as there is a chance of liquid being shot out of the test tube and harm you or your colleague.
7. Do not heat the standard glassware because its size will change.
8. Mix the chemicals inside the test tube well either by shaking or by a stirring rod.
9. Many chemicals are toxic, so wash your hands before leaving the laboratory.

## REAGENTS AND HOW TO USE THEM

Conducting laboratory experiments depends on the use of various reagents and chemicals, and there is always a laboratory in a specified amount of reagents, part of which is placed on the laboratory benches and store the other in the cabinets that are designed for this purpose.

It is important to know the compositions of the reagents and their main properties, flammability, toxicity and ability to form explosive mixtures with other reagents [2].

Solutions and reagents are kept in glass bottles sealed with glass, rubber or corkscrew stoppers. A piece of paper is affixed to each bottle containing the name and concentration of the reagent [2, 3].

The following primary rules are considered when reagents are used:

1. If there is no clear definition of the amount of reagents to be taken, take as little as possible of reagents and this will give better results, save time and reduce consumption and waste materials.
2. It is strictly forbidden to return the excess quantity of reagent that was taken to the bottle of the reagent in order to prevent contamination and the complete destruction of the reagent.
3. Used reagents must be returned to their places after usage.
4. Do not mix the stoppers of different reagent bottles as well as rubber pipetting bulbs between the pipettes used to take the reagents.
5. Special attention should be paid when dealing with toxic, harmful or flammable substances such as barium salts, mercury, arsenic, Carbon disulphide and others.

## HOW TO WRITE LABORATORY REPORTS

Writing laboratory reports is an important part of the practical sessions and when writing reports, consider the following [1]:

1. The student must be equipped with a notebook to write her/his observations about the practical lesson first hand.
2. The student should submit a report of the experiment, explaining the following:
   a. Name of the experiment.
   b. The purpose of the experiment.
   c. The practical idea is based on experimentation with the explanation of the equations of interaction.
   d. Conclusion.
   e. Difficulties that occurred when conducting the experiment.
   f. Reasons for the failure of the experiment (if a failure occurs).

## THE WORK TECHNIQUES IN THE LABORATORY

The work in the descriptive and quantitative analysis laboratories requires the identification of some general processes, the basis of this practice and its mastery

## CHAPTER 2

# Inorganic Qualitative Analysis

**Abstract:** In this chapter, the different types of analysis are explained. In addition, the importance of the rule of solubility of compounds in water in separation and identification of the cations is emphasised.

**Keywords:** Qualitative analysis, Quantitative analysis, Solubility rules, Soluble compounds.

## INORGANIC QUALITATIVE ANALYSIS

A number of the chemical analysis are used to assign the components of any unknown substance. It could be determining the components and quantitative ratios of those unknown substances. This chemical analysis will be briefly introduced below [4]:

### Firstly: Modern Analysis Methods

Modern analysis methods that are based on the progress and development of science and technology have been achieved in order to design and construct high-sensitivity devices. The working principle of that device is to measure one and more of the physical properties of substances underinvesting that link to components and quantitative ratios of any unknown substance. Photoanalysis, electrochemical analysis or voltage analysis have been used so far.

### Secondly: Traditional Analysis Methods

### *1. Qualitative Analysis*

It is defined as a set of tests or chemical processes aimed to identify the material whether unknown material is a pure substance or mixture in a solid state or in a solution without paying attention to the weights of these components or the elements involved in the structure or the proportions of their existence. Therefore, the descriptive analysis is never affected by the loss of the sample for a fraction of its quantity as long as the purpose of this analysis is to determine the components of the relative presence or weights in the sample [2, 4].

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## *2. Quantitative Analysis*

It is used to determine the weights of the components or elements involved in the composition of the mixture or the chemical compound and its proportions. Therefore, the presence of impurities in the material affects the determination of these weights and the choice of analysis method that requires knowledge of the elements that made up the substance under investigation.

A number of producers are required precede to the quantitative analysis:

a) Conduct qualitative analysis to identify the elements and components involved in the composition of the substance and the impurities that contain.

b) Purify the material to be analyzed from the impurities before assigning the proportions of its components.

### ***Scales Using in Qualitative Analysis***

The qualitative analysis can be carried out in different types of scales, as summarized in Table **2.1** [4].

**Table 2.1. The types of measuring scale used in the qualitative analysis.**

| Scale Qualitative Analysis | Macro | Micro | Semi-micro |
|---|---|---|---|
| **Sample size** | 5-100 mL<br>0.5 – 1 g | ≥ 1 mL<br>0.005 – 0.01 g | One drop<br>(no more than 1 mL)<br>0.05 – 0.01 g |

There is no real difference between micro qualitative analysis and semi-micro qualitative analysis, which is the result of the difference in the quantities used in the two cases. The difference is confined to the quantities and small volumes of the sediments and solutions that are used, as well as the use of the type of special tools for each analysis.

There is no advantage in the sensitivity of the semi-micro qualitative analysis. However, there are a number of advantages in another aspect, for example, saving time and chemicals. Filtration, sediment washing, and vaporization processes can take about an hour. These processes can be performed in a few minutes if the semi-micro-method is followed, thus allowing the student to analyse more samples on time and gain broader training in analytical methods [2, 4].

## GLOSSARY IN QUALITATIVE ANALYSIS

**Centrifuge:** It is a device used to accelerate the stability of the sediment, making use of centrifugal force.

**Suspension:** It is the separation of liquid from stable sediment.

**Filtration:** It is the separation of a precipitate from a fluid through a medium. A liquid that passes through the filter called filtrate or filtered liquid, while the residue left on the filter paper is called filtered solid (separation processes are often done in semi-micro-analysis by centrifuge and circulation).

**Sedimentation:** It is the formation of an insoluble solid that forms when reagent reacts with a solution. The insoluble solid is called a precipitate.

**Reagent:** It a substance used to make a chemical change in the material under test, for example, hydrochloric acid is a reagent added to the solution of silver nitrate because it is a precipitate of silver chloride.

**Turbidity:** It is the occurrence of something like clouds as a result of small particles stuck in a solvent.

**Washing:** It is the cleaning of precipitate (usually with water) to remove the suspension of the solution from which it precipitates.

General Rules in the Solubility of Inorganic Compounds in water Inorganic compounds are classified based on their solubility in water into three groups [6, 7]:

i. Soluble compounds: Soluble in water more than 10 g/L.
ii. Low solubility compounds: The water solubility of less than 1 g/L.
iii. Moderately soluble compounds: Compounds that are soluble in water more than 1 g/L and less than 10 g/L.

## CHAPTER 3

# The First Group

**Abstract:** Chapter three provides the procedure for the detection and separation of the first group cations, highlighting the importance of the solubility constant and the effect of pH on the detection and separation of the cations.

**Keywords:** Chlorides, Detection, Separation, Silver group, The solubility constant ksp.

## DETECTION AND SEPARATION OF THE FIRST GROUP OF CATIONS

A number of the chemical analysis are used to assign the components of any unknown substance. It could be determining the components and quantitative ratios of those unknown substances. This chemical analysis will be briefly introduced below [4].

### Silver Group

The first group of cations (silver group) consists of three cations, $Ag^+$ silver ion, $Pb^{2+}$ lead ion, $Hg_2^{2+}$ mercurous ion, and their reagents are diluted in hydrochloric acid because the chlorides of previous cations are insoluble in water ($AgCl$, $Hg_2Cl_2$) or mildly soluble in water ($PbCl_2$) [5].

While the cations of chlorides of the other five groups are dissolved in water, the table below shows that the solubility of the mercurous chloride exhibits the lowest value.

Lead chloride has a significant solubility in water, therefore, it is not completely precipitated and separated in the first group at a room temperature of 25°C. Thus, lead is present in the second group and is precipitated as lead sulphide (Table **3.1**), a list of Solubility constant ksp at 25°C various ionic compounds in water at 25°C is given in Appendices [6, 7].

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**Table 3.1. The solubility constant ksp for chloride; the first group cation at 25°C.**

| Compounds | Solubility Constant $k_{sp}$ at 25°C | Dissolved Amount mg/L at 25°C |
|---|---|---|
| **AgCl** | $1.8 \times 10^{-10}$ | $1.7 \times 10^{-3}$ |
| **$PbCl_2$** | $1.7 \times 10^{-5}$ | $1.6 \times 10^{-2}$ |
| **$Hg_2Cl_2$** | $1.3 \times 10^{-18}$ | $3.8 \times 10^{-4}$ |

Chloride ion obtained from the dissociation of hydrochloric acid in the solution is added to cations of the first group, leading to complete precipitation of $Ag^+$, $Pb^{2+}$ as a result of common ions. However, adding chloride abundantly would lead to a good amount of HCl for dissolving some of the $Ag^+$ and $Pb^{2+}$ and as a result of which the complex ion such as $[AgCl_2]^-$, $[PbCl_3]^-$ that are soluble are formed. For this reason, the reagent of this group is hydrochloric acid achieved according to the following reactions:

$$AgCl + Cl^- \rightarrow AgCl_2^- \qquad \textbf{(3.1)}$$

$$AgCl + 2Cl^- \rightarrow AgCl_3^{2-} \qquad \textbf{(3.2)}$$

$$PbCl_2 + Cl^- \rightarrow PbCl_3^- \qquad \textbf{(3.3)}$$

## Separation of the Cations of Group I

The first group contains the following cations: $Hg_2^{2+}$, $Pb^{2+}$, $Ag^+$

1. Take 1 mL of a solution containing the first group cations and place it in a 5mL conical tube.
2. Add about 15 drops of diluted HCl, shake the solution well, the cations of the first group will be precipitated because their chlorides are insoluble as in the following equations [3, 5]:

$$Ag^+ + Cl^- \rightarrow \underset{\text{White precipitate}}{AgCl} \qquad \textbf{(3.4)}$$

$$Hg_2^{2+} + 2Cl^- \rightarrow Hg_2Cl_2 \qquad \textbf{(3.5)}$$

White precipitate

$$Pb^{2+} + 2Cl^- \rightarrow PbCl_2 \qquad \textbf{(3.6)}$$

White precipitate

3. Place the tube in a centrifuge for 5 minutes, make sure that full precipitation is obtained for the cations by adding a drop of HCl.
4. Filter the solution, the filtered liquid contains the cations from other groups (2, 3, 4, 5 and 6) that is neglected and the precipitate is a mixture of AgCl, $Hg_2Cl_2$ $PbCl_2$.
5. Add 15 drops of hot water to the precipitate (the precipitate of lead chloride dissolve in hot water with stirring). Use the centrifuge to separate the mixture for 5 minutes. The precipitate contains AgCl, $Hg_2Cl_2$ and the filtered liquid contains $Pb^{2+}$. Lead is identified by depositing it in the form of yellow lead chromate.

$$Ag^+ + Cl^- \rightarrow AgCl \qquad \textbf{(3.7)}$$

White precipitate

6. The precipitate contains AgCl. $Hg_2Cl_2$ is separated by ammonia solution and 15 drops of $NH_4OH$ to the precipitate is added, as a consequence of that, the silver chloride is dissolved in the ammonia solution by formation of the complex $[Ag(NH_3)_2]Cl$ silver diamine chloride.

$$AgCl_{(s)} + 2NH_4OH \rightarrow Ag(NH_3)_2^+ + Cl^- + H_2O \qquad \textbf{(3.8)}$$

Soluble complex

The filtered liquid contains $Ag^+$, which is detected by the addition of 5 drops of diluted HCl. A white solid is formed to indicate the presence of silver.

$$Ag(NH_3)_2^+ + 2HCl \rightarrow AgCl_{(s)} + 2NH_4^+ \qquad \textbf{(3.9)}$$

## CHAPTER 4

# The Second Group

**Abstract:** This chapter discusses the detection and separation of the second group of cations. It gives step by step practical directions and a set of rules, which must be followed for successful detection and separation of the cations.

**Keywords:** Acidic medium, Common Ion, Sulphides, Thioacetamide.

## DETECTION AND SEPARATION OF THE SECOND GROUP CATIONS

### Copper Group

The group includes Mercury (II) $Hg^{2+}$, Copper $Cu^{2+}$, Lead $Pb^{2+}$, Bismuth $Bi^{3+}$, Cadmium $Cd^{2+}$, Tin (II) $Sn^{2+}$, Tin (IV) $Sn^{4+}$, Antimony (III) $Sb^{3+}$, Antimony (V) $Sb^{5+}$ and Arsenic (III) $As^{3+}$, Arsenic (V) $As^{5+}$. This group is divided into two subgroups: the first group is called copper group, which contains Mercury $Hg^{2+}$, Copper $Cu^{2+}$, Lead $Pb^{2+}$, Bismuth $Bi^{3+}$and Cadmium $Cd^{2+}$, whereas the second family is called the arsenic group, which contains: $Sn^{2+}$, $Sn^{4+}$, $Sb^{3+}$, $Sb^{5+}$, $As^{3+}$and $As^{5+}$. The reason for this division is based on their solubility's of some its sulphides in ammonium polysulphide $(NH_4)_2S_x$; x>2 [5].

It is found that the copper family (CuS, PbS, CdS, $Bi_2S_3$ and HgS) does not dissolve in $(NH_4)_2S$, while, the arsenic family ($SnS_2$, SnS, $Sb_2S_5$, $Sb_2S_3$ and $AsS_5$, $As_2S_3$) is soluble in ammonium polysulphide.

The reagent of the second group is hydrosulfuric acid ($H_2S$) in the acidic medium (0.3 N of HCl) because the sulphide of the former elements does not dissolve in this concentration of acid.

It is important to understand the function of hydrosulfuric acid in descriptive analysis and the conditions in which acid is used in the precipitation of the second and fourth groups. The analyst should be aware of the reactions taking place in the test tube and the concepts of equilibrium and solubility product constant.

Hydrosulfuric acid $H_2S$ is a diprotic acid and a weak acid. Its two stages of ionization are:

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$$H_2S \rightarrow HS^- + H^+ \qquad \boldsymbol{K_1 = 1.0 \times 10^{-8}} \qquad \textbf{(4.1)}$$

$$HS^- \rightarrow S^{2-} + H^+ \qquad \boldsymbol{K_2 = 1.3 \times 10^{-15}} \qquad \textbf{(4.2)}$$

By combining these balances, we get the following balance:

$$H_2S \rightarrow S^{2-} + 2H^+ \qquad \boldsymbol{K = 1.3 \times 10^{-23}} \qquad \textbf{(4.3)}$$

Which is equal to its second hitting the two sets $k_2$, $k_1$, *i.e.*, $(S^{2-})\ (H^+)^2 = (H_2S)$. When the solution is saturated, the concentration of $H_2S$ is approximated (0.1) N, therefore, $(S^{2-})\ (H^+)^2 = 1.3 \times 10^{-23}$.

This relationship shows that the concentration of sulphide ions is inversely square of the concentration of hydrogen ion, which means that the sulphide ($S^{2-}$) concentration decreases when the acidity of the solution increases. The concentration of these ions can be controlled by adjusting the acidity of the solution. As the previous relationship shows, a specific acid concentration is used so that the second group is only precipitated. Therefore, the cations of the second group require a lower concentration of the $S^{2-}$ ion to be precipitated in the comparison between the cation of the third and fourth group. In fact, the solubility of the second group sulphide, in general, was lower than the sulphides of the third and fourth groups. Table **4.1** shows the fixed solubility values of some of the first four groups of sulphides [6-8].

**Table 4.1. Solubility constant values of some sulphides compounds.**

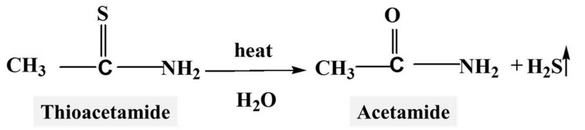
| Metal Sulphide | $K_{sp}$ | Metal Sulphide | $K_{sp}$ |
|---|---|---|---|
| CuS | $6.3 \times 10^{-36}$ | NiS | $1.3 \times 10^{-25}$ |
| CdS | $8 \times 10^{-27}$ | CoS | $5 \times 10^{-22}$ |
| PbS | $8 \times 10^{-28}$ | $Sb_2S_3$ | $<10^{-30}$ |
| HgS | $4 \times 10^{-53}$ | MnS | $2.5 \times 10^{-13}$ |
| $As_2S_3$ | $8 \times 10^{-25}$ | FeS | $6.3 \times 10^{-18}$ |
| $Bi_2S_3$ | $1.6 \times 10^{-72}$ | ZnS | $1.6 \times 10^{-24}$ |
| SnS | $1 \times 10^{-25}$ | $Ag_2S$ | $6.3 \times 10^{-50}$ |

The concentration of about 0.3 N hydrosulfuric acid was chosen, so the concentration of sulphide ion ($S^{2-}$) was sufficient for cadmium sulphide CdS to be precipitated, *i.e.*, cadmium sulphide is more soluble of the second group. At the same time, the concentration of sulphide ion ($S^{2-}$) should be less than required to precipitate zinc sulphide ZnS *i.e.*, zinc sulphide is the lowest solubility of the fourth group. Therefore, the acidity of the solution plays a significant factor in the precipitation of the second group and any error may lead to precipitate the group V [5, 9].

A Mercury (II), Copper (II), Cadmium (II) are located within the transitional elements in the periodic table; the other five elements are located in line between metal and nonmetal Tin (II) and IV, Antimony (III) and (V), Arsenic (III), Bismuth (III) and lead (II). As a result of that, these elements show amphoteric properties that differ in intensity between one element and another. The elements of this group are characterized by their strong tendency to bond to sulfur. This property appears in two forms as a precipitate or the form of metals in nature [1, 4].

## The Method of Work

1. Take 1 mL of a solution containing the group ions, *i.e.*, a copper family that is insoluble in ammonium polysulphide ($HgS$, $Bi_2S_3$, $PbS$, $CuS$, $CdS$).
2. In a conical tube, then add a drop of the methyl violet indicator.
3. Add diluted hydrochloric acid HCl until the colour of the solution turns yellow-green, which means that the acid concentration is 0.3M.
4. Then, add hydrosulfuric acid $H_2S$ 20 drop in the form of thioacetamide solution as seen in Equation (4.4), so the toxicity and disagreeable odor of hydrogen sulphide are eliminated [9].



$$CH_3C(=S)NH_2 \xrightarrow[H_2O]{heat} CH_3C(=O)NH_2 + H_2S\uparrow \tag{4.4}$$

$$Hg^{2+} + S^{2-} \rightarrow HgS_{(s)} \tag{4.5}$$

$$M^{2+} + S^{2-} \rightarrow MS_{(s)} \tag{4.6}$$

## CHAPTER 5

# The Third Group

**Abstract:** This chapter focuses on the third group cations (detection and separation) and describes the method of work, precipitate format, and the impact of pH on the procedure.

**Keywords:** Basic medium, Common ion, Hydroxides, Sulfides.

## DETECTION AND SEPARATION OF THE THIRD GROUP CATIONS

### Group III

This group includes the following cations: Iron (III and II) $Fe^{3+}$ and $Fe^{2+}$, Aluminum $Al^{3+}$, Chromium $Cr^{3+}$, Manganese $Mn^{2+}$, Zinc $Zn^{2+}$, Nickel $Ni^{2+}$, Cobalt $Co^{2+}$. It can be divided into two subgroups depending on the type of precipitate as follows: ($Fe^{3+}$, $Fe^{2+}$, $Al^{3+}$, $Cr^{3+}$) are precipitated in the form of hydroxides. ($Mn^{2+}$, $Zn^{2+}$, $Ni^{2+}$and $Co^{2+}$) are precipitated as sulfides in a basic medium [5, 9].

This group ($Fe^{3+}$, $Fe^{2+}$, $Al^{3+}$, $Cr^{3+}$) contains Ammonium Hydroxide $NH_4OH$ as the group reagent, the precipitates of this group are in the form of hydroxides because the hydroxides of these ions are insoluble in water, and the constants of solubility are low

| | | |
|---|---|---|
| $Fe(OH)_3$ | $K_{sp} = 6x10^{-38}$ | pH=3 |
| $Fe(OH)_2$ | $K_{sp} = 2x10^{-15}$ | pH=7 |
| $Al(OH)_3$ | $K_{sp} = 7x10^{-31}$ | pH=5 |
| $Cr(OH)_3$ | $K_{sp} = 1.4x10^{-30}$ | pH=6 |

The precipitation of metal hydroxide depends largely on the value of the hydroxide-soluble constant and the concentration of hydroxide ions in the solution or on the concentration of hydrogen ions (pH), where

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$$pH = -\log\left[H^+\right] \qquad pOH = -\log\left[OH^-\right]$$

$$pK_a = -\log\left[K_a\right] \qquad pK_b = -\log\left[K_b\right]$$

$$pK_w = -\log\left[K_w\right] \qquad pK_w = pH + pOH = 14$$

Where $K_w$, $K_a$, and $K_b$ are the autoionization water constant, the acid ionization constant, and base ionization constant, respectively. It could be controlled by the concentration of hydroxide by using a weak electrolyte, such as ammonium hydroxide (ammonia in water), and adding different concentrations of ammonium ions in the form of ammonium chloride salt. The added salt contains a common ion, *i.e.*, ammonium. The common ion is defined as the symmetric ion produced by two or more electrolyte ionization. If one electrolyte solution is added to another solution, one of the ions in the first solution leads to the concentration of sodium chloride to increase chloride ion concentration:

$$NaCl \rightarrow Na^+ + Cl^- \tag{5.1}$$

$$HCl \rightarrow H^+ + Cl^- \tag{5.2}$$

Adding ammonium chloride to the ammonia solution will increase the concentration of ammonium ion $NH_4^+$, but it will reduce the ionization of ammonia (the effect of the common ion and therefore the concentration of hydroxide ion)

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \tag{5.3}$$

$$NH_4Cl \rightarrow NH_4^+ + Cl^- \tag{5.4}$$

Controlling the concentration of hydroxide ions in the solution leads to precipitates of hydroxide and less soluble hydroxide.

Using a mixture of ammonium chloride and ammonia solution can precipitate selective ions. Some cations of the third group, for example, iron and chromium are located within the transition elements in the periodic table, therefore, these ions show the characteristics of the transition elements as follows:

1. Multi oxidations state as due to the proximity of the energy sublevels of 4p and 3d.
2. Coloured ions.
3. The tendency to form complex ions.

Aluminum is not from transition metal but it has similar properties to chromium and iron because these three have the same charge ions and the value of ionic radius is very close. The colors of the solution and precipitated ions are distinct, therefore the analyst should pay attention to these characteristics because they help a lot in predicting the identity of the solution, which leads to curtailing many of the necessary steps and processes.

## Procedure

Group III is precipitated and separated from other groups and then their ions are detected in the mixture to be analyzed using the following steps [5, 9]:

1. Heat the filtered liquid that is produced by the separation of the second group to the boiling point until the evaporation of hydrogen sulfide gas is completely stopped.
2. Add 3-4 mL of concentrated nitric acid and continue boiling until all iron $Fe^{2+}$(II) is oxidized to iron $Fe^{3+}$ (III) if present.
3. Add about 0.2 mg of solid ammonium chloride, then add the ammonia solution with stirring until the solution becomes basic and detected using the red litmus paper (until the smell of ammonia appears).
4. The previous solution is placed on a water bath for 3 minutes. After removing the solution from the water bath, the filtered liquid and solid precipitate are separated using the centrifuge device (C.F) for 5 minutes. This step is quick (step of separation), showing that the precipitate contains group III hydroxides. The color of the final formed precipitate was yellow-brown.

$$Al^{3+} + NH_4OH \xrightarrow{NH_4Cl} Al(OH)_{3(s)} \qquad \textbf{(5.5)}$$

White precipitate

$$Fe^{3+} + NH_4OH \xrightarrow{NH_4Cl} Fe(OH)_{3(s)} \qquad \textbf{(5.6)}$$

Brown precipitate

## CHAPTER 6

# The Fourth Group

**Abstract:** This chapter discusses the method of detection and separation of the fourth group cations, precipitate format, the impact of pH on the procedure.

**Keywords:** Acidic medium, Black precipitate, Pink precipitate, Sulfides, White precipitate.

## DETECTION AND SEPARATION OF THE FOURTH GROUP CATIONS

### Group IV

This group contains Manganese $Mn^{2+}$, Cobalt $Co^{2+}$, Nickel $Ni^{2+}$, and Zinc $Zn^{2+}$. These ions are divalent. The group reagent is hydrogen sulfide in a weakly acidic medium. To control the acidic medium, the mixture of ammonium chloride $NH_4Cl$ and ammonium hydroxide $NH_4OH$ is used. Ammonium hydroxide is a weak base. However, the alkalinity of Ammonium hydroxide is high. Therefore, the alkalinity decreases with the addition of ammonium chloride, which leads to the following balance shift to the left [1, 5].

$$NH_4OH \leftrightarrow NH_4^+ + OH^- \tag{6.1}$$

The solution contains $H_2S$ and ammonium chloride $NH_4Cl$ and ammonium hydroxide $NH_4OH$. Therefore, another balance is as follows:

$$H_2S \leftrightarrow 2H^+ + S^{2-} \tag{6.2}$$

Since an appropriate amount of ammonium hydroxide is added, so the medium is basic $[OH] > 10^{-7}$. Therefore, the equilibrium in Equation 6.2 is shifted to the right so that the group reagent contains a good concentration of $S^{2-}$ ion which is much larger than the concentration of $S^{2-}$ of the group reagent for the second group because the medium is acidic in that group. Thus, the precipitated medium here contains $S^{2-}$ ion and $OH^-$ with a relatively higher concentration. Both of these ions are precipitated because sulfide and hydroxide are more insoluble minerals [1, 5].

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The cations of this group are not precipitated when the cations of the second group are precipitated because these sulfides are dissolved in the acidic medium of HCl (0.3M) as observed previously. In other words, the concentration of $S^{2-}$ ion is not sufficient for precipitation of the group IV ions in the conditions of the second group. The group IV ions are precipitated in the form of sulfides as follows:

$$Co(NO_3)_2 + H_2S_{(g)} \rightarrow CoS_{(s)} + 2HNO_3 \tag{6.3}$$

Black precipitate

$$NiCl_2 + H_2S_{(g)} \rightarrow NiS_{(s)} + 2HCl \tag{6.4}$$

Black precipitate

Black precipitates of CoS and NiS are dissolved in hot nitric acid and Aqua regia, respectively. However, these precipitates are insoluble in hydrochloric acid.

$$MnCl_2 + H_2S_{(g)} \rightarrow MnS_{(s)} + 2HCl \tag{6.5}$$

Pink precipitate

$$ZnCl_2 + H_2S_{(g)} \rightarrow ZnS_{(s)} + 2HCl \tag{6.6}$$

White precipitate

Both ZnS and MnS are dissolved in the mineral acids. Besides, zinc sulfide is dissolved in acetic acid.

## Methodology

Group IV is precipitated and separated from the rest of the groups in the analysed mixture using the following steps (see Flow chart **6.1**):

1. Take filtered liquid produced by separating the ions of the third group, add 1-2 g of $NH_4Cl$, and then heat for 2-1 minutes on a water bath.
2. Add the ammonia solution to filtered liquid until it becomes alkaline (use red litmus paper, *i.e.*, change the color of the paper from red to blue). Then, add 2 drops of the thioacetamide solution and heat it for 15 minutes. The ions of this group will be precipitated as sulfides.

3. Separate the filtered liquid from the precipitate after placing a solution in the centrifuge for 5 minutes. Keep the filtered liquid to detect subsequent group ions if present.
4. Group IV ions are separated from each other and their ions are detected by following scheme (6.1).
5. The precipitate contains sulfide of insoluble cobalt, nickel, zinc, and magnesium. Add about 10 drops of HCl to the solution with continuous stirring and heat it on a water bath for 3-5 minutes, the following chlorides will be obtained:

$$NiS + 2HCl \rightarrow NiCl_{2(s)} + H_2S_{(g)} \qquad \textbf{(6.7)}$$

Precipitate chloride

$$CoS + 2HCl \rightarrow CoCl_{2(s)} + H_2S_{(g)} \qquad \textbf{(6.8)}$$

Precipitate chloride

$$MnS + 2HCl \rightarrow MnCl_2 + H_2S_{(g)} \qquad \textbf{(6.9)}$$

Soluble chloride

$$ZnS + 2HCl \rightarrow ZnCl_2 + H_2S_{(g)} \qquad \textbf{(6.10)}$$

Soluble chloride

6. The filtered liquid ($ZnCl_2$, $MnCl_2$) is separated from the precipitate ($CoCl_2$, $NiCl_2$). Nine drops from the Aqua Regia ($1HNO_3$:$3HCl$) is added to the precipitate and heated for 1-2 minutes. Both precipitates will dissolve. The filtered liquid is divided into two parts:

   **The First Section:** It detects the presence of $Co^{2+}$ ion by adding a few drops of ammonium thiocyanate ($NH_4SCN$). A blue color solution appears indicating the presence of cobalt.

$$CoCl_2 + 2NH_4SCN \rightarrow (NH_4)_2[CoSCN_4] + NH_4Cl \qquad \textbf{(6.11)}$$

   **The Second Section:** The presence of $Ni^{2+}$ ion is detected by the addition of (1spatula) $NH_4Cl_{(S)}$ and then the solution is made basic by $NH_3$ solution and tested with litmus paper. Then, 7 drops of the Dimethylglyoxime (DMG)

# CHAPTER 7

# The Fifth Group

**Abstract:** This chapter elaborates on the the detection and separation of the fifth group cations. It explains step-by-step practical directions and a number of rules that must be followed for successful detection and separation of the cations.

**Keywords:** Ammonium chloride, Ammonium hydroxide, Carbonates, White precipitate.

## DETECTION AND SEPARATION OF THE FIFTH GROUP CATIONS

### Group V

This group includes calcium ions $Ca^{2+}$, strontium ions $Sr^{2+}$ and barium ions $Ba^{2+}$, which belong to alkaline earth metals in the periodic table. These metals produce soluble chlorides, sulphides and hydroxides in conditions that allow precipitation of groups I, II and III. However, these ions precipitate in the form of carbonates using ammonium carbonate (buffer solution) from ammonium hydroxide and ammonium chloride [1, 5].

The purpose of using ammonium chloride in the mixture of [$NH_4Cl + NH_4OH + (NH_4)_2CO_3$] is to precipitate ions of this group and control the number of carbonate ions that are precipitated so that the magnesium ions are not precipitated with this group, neither in the form of carbonate nor in the form of hydroxide. The presence of ammonium ions in a large quantity reduces the concentration of carbonates precipitated by moving the equilibrium to the right. Thus, it possible to get rid of the carbonate where the bicarbonate gets dissociated when the solution is heated. The bicarbonates are converted into carbonates by neutralizing the ammonia solution [5].

$$NH_4^+ + CO_3^{2-} \rightarrow NH_3 + HCO_3^- \quad \textbf{(7.1)}$$

The presence of ammonium in a large amount reduces the concentration of hydroxyl ions that are produced when the ammonium hydroxide is ionised.

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$$NH_4OH \leftrightarrow NH_4^+ + HO^- \quad \textbf{(7.2)}$$

But the concentration of ammonium ion $NH_4^+$ should not be too large that may have an impact on the equilibrium.

If the equilibrium moves to the right, this could lead to reducing the concentration of carbonates to the level that is not sufficient to precipitate the ions of group V. The presence of ammonium hydroxide prevents the previous behaviour, as a consequence of hydroxides ions that are produced from the dissociation of ammonium hydroxide combined with the increase of ammonium ions, that leads to the equilibrium move to the left.

In other words, the buffer solution ($NH_4OH$ + $NH_4Cl$) provides an appropriate concentration of carbonate ions that is sufficient to precipitate the ions of group V but is insufficient to precipitate magnesium carbonate.

$$Ba^{2+} + CO_3^{2-} \rightarrow \underset{\text{White precipitate}}{BaCO_{3(s)}} \quad \textbf{(7.3)}$$

$$Sr^{2+} + CO_3^{2-} \rightarrow \underset{\text{White precipitate}}{SrCO_{3(s)}} \quad \textbf{(7.4)}$$

$$Ca^{2+} + CO_3^{2-} \rightarrow \underset{\text{White precipitate}}{CaCO_{3(s)}} \quad \textbf{(7.5)}$$

**Warning:** Do not direct the test tube while heating up toward you or toward your colleagues, and heat the centre of the tube with the continuous movement to distribute the heating on all parts of the liquid.

## The Method of Work

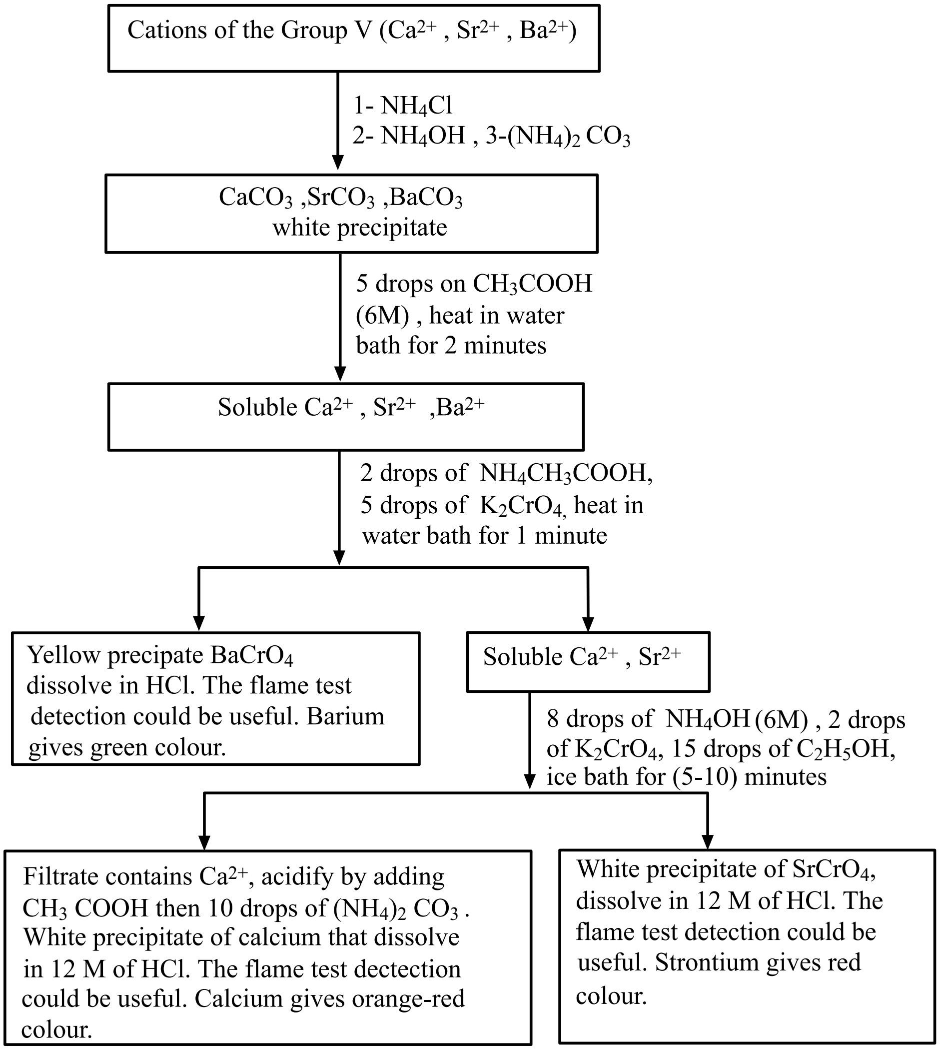
Group V ions are precipitated and separated from the group VI ions by the following steps (See [9]):

1. Heating the filtered liquid produced by filtering group IV at the boiling point in order to remove ammonium salts and then diluting the solution by adding a small amount of distilled water. Following this, the medium of the solution is

changed to make it basic by adding several drops of $NH_4OH$ followed by the addition of a few drops of $NH_4Cl$ and about 4-5 drops of $NH_4CO_3$. The mixture is heated in a water bath for 2 minutes.

2. Filtering the mixture and separating the precipitates that contain the ions of group V in the form of carbonates. The filtered liquid is retained that contains the ions of group V.

   The cations of group V are separated from each other and then detected. (see Flow chart **7.1).**



Cations of the Group V ($Ca^{2+}$ , $Sr^{2+}$ , $Ba^{2+}$)

1- $NH_4Cl$
2- $NH_4OH$ , 3-$(NH_4)_2$ $CO_3$

$CaCO_3$ ,$SrCO_3$ ,$BaCO_3$
white precipitate

5 drops on $CH_3COOH$ (6M) , heat in water bath for 2 minutes

Soluble $Ca^{2+}$ , $Sr^{2+}$ ,$Ba^{2+}$

2 drops of $NH_4CH_3COOH$, 5 drops of $K_2CrO_4$, heat in water bath for 1 minute

Yellow precipate $BaCrO_4$ dissolve in HCl. The flame test detection could be useful. Barium gives green colour.

Soluble $Ca^{2+}$ , $Sr^{2+}$

8 drops of $NH_4OH$ (6M) , 2 drops of $K_2CrO_4$, 15 drops of $C_2H_5OH$, ice bath for (5-10) minutes

Filtrate contains $Ca^{2+}$, acidify by adding $CH_3$ COOH then 10 drops of $(NH_4)_2$ $CO_3$ . White precipitate of calcium that dissolve in 12 M of HCl. The flame test dectection could be useful. Calcium gives orange-red colour.

White precipitate of $SrCrO_4$, dissolve in 12 M of HCl. The flame test detection could be useful. Strontium gives red colour.

**Flow chart 7.1.** The analysis of cations group V.

## CHAPTER 8

# The Sixth Group

**Abstract:** This chapter focuses on the sixth group cations (detection and separation), and elaborates on the method and step-by-step practical directions for the flame test.

**Keywords:** Alkali ions, Ammonium chloride, Flame test, Group reagent.

## DETECTION AND SEPARATION OF THE SIXTH GROUP CATIONS

### Group VI

This group contains magnesium ions $Mg^{2+}$, sodium $Na^+$, potassium $K^+$ and ammonium $NH_4^+$. These ions are not precipitated with any of the previous *via* groups. Magnesium ions may be partially precipitated in the form of a hydroxide or a carbonate with the fourth and fifth groups if the amount of ammonium chloride is not added sufficiently during precipitation [5, 9].

There is no general group regent for group VI. Therefore, all common sodium, potassium and ammonium salts are soluble in water. There are some complexes that have intermediate solubility, as the result of the presence of alkali ions in their composition. These complexes are used in the detection of ions. We will talk about each while talking about the analytical properties of each ion.

In case of magnesium ions, its hydroxide, carbonate, phosphates, and oxalate are insoluble in water. Therefore, this method is used for the detection of $Mg^{2+}$ ions [1, 5].

## THE ANALYTICAL PROPERTIES OF THE IONS OF THIS GROUP

$Mg^{2+}$ have ions similar to the group of alkaline earth minerals, while sodium and potassium follow the group of alkali minerals. Since $NH_4^+$ is similar to the $K^+$ ion, so it is classified in this group [9].

The metals of this group are active metals since they easily lose the electrons from the outer surface. Most of their ionic compounds are solid. Alkali ions seldom form complex compounds because of their large size and a small charge. The ions are colourless because there are no unpaired electrons in the last layer.

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**Caution:** Since sulfuric acid and potassium dichromate are very acidic, their mixture has similar properties. We must avoid using this solution, and it could be replaced by known commercial cleaning solutions.

## THE WORK METHOD

### Detection of $Mg^{2+}$

Firstly, it is important to make sure that all the former groups' ions especially barium and strontium ions, were isolated during the analysis of these groups by adding a mixture of 0.5 mL of ammonium sulphate $(NH_4)_2SO_4$ and 0.5 mL of ammonium oxalate $C_2H_8N_2O_4$ to the filtrate of the fifth group [1, 9].

Following this, the mixture is heated and left aside for a few minutes and then the solution is filtered whenever the precipitate appears. The supernatant contains the ions of the sixth group. The filtrates are kept and and the precipitates are discarded.

$NH_4OH$ is added to the filtrate until the medium becomes basic, followed by the addition of five more drops of $NH_4OH$. After this, 1 mL of $Na_2HPO_4$ is added and the solution is stirred well with a stirring rod to scratch the walls with the test tube. After a few minutes of wait, if a white crystalline solid appears, this is taken as evidence of the presence of $Mg^{2+}$ (see Flow chart **8.1**).

### Detection of $NH_4^+$

3 mL of the original solution is taken and placed in an evaporating dish. After this, 3 mL of concentrated NaOH or KOH is added and mixed well with a stirring rod and covered with a watch glass containing a piece of moist red litmus paper inside. It is heated gently until the vapors start to release. If the colour of the paper uniformLy turns into blue, this indicates the presence of ammonium in the solution. (see Flow chart **8.2**)

### Detection of $Na^+$

In 3 mL of the original solution in a test tube, a concentrated solution of KOH is added until it becomes alkaline. Following this, about 3 mL of a concentrated solution of $K_2CO_3$ is added and boiled for a short period, and then cooled down and filtered to make sure that the precipitation process is complete. Following this, the supernatant is placed in the crucible, heated over a gentle fire to dryness. The remaining salt in the crucible is dissolved with 1.5 mL of water. Sodium is detected by either by adding uranyl acetate or magnesium acetate or antimony potassium tartrate. First a yellowish-green precipitate is formed and then a white precipitate. In the flame test, an intense yellow bulky flame appears if $Na^+$ is present.

### Detection of $K^+$

In 3 mL of the original solution into a test tube, a concentrated solution of NaOH is added until it becomes alkaline.

After this 3 mL of concentrated solution $Na_2CO_3$ is added and the mixture is heated. The mixture in then cooled down and filtered to make sure that the precipitation process is complete. The supernatant is placed in the crucible, heated over a gentle fire to dryness. The remaining salt in the crucible is dissolved with 1 mL of water and divided into two equal portions (see Flow chart **8.2**).

**The First Portion:** Several drops of acetic acid are added until the solution is slightly acidic followed by the addition of 1 mL of sodium cobalt nitrite $Na_3Co(NO_2)_6$ . The appearance of a yellow precipitate indicates the presence of $K^+$ .

**The Second Portion:** A flame test is carried out, where a violet flame appears if $K^+$ is present.

### ADDITIONAL INFORMATION:

1. The detection of sodium is a very sensitive process since the residues of sodium ions that are found in most solutions and even in glass bottles could make the solutions attain a colour. Therefore, it is necessary to compare the detection of the unknown solution with a solution that contains sodium ions. If intense yellow colour appears for five seconds or more, that is the evidence of the presence of sodium ions, but if the colour of yellow is not dense and remains for a short period (less than five seconds), sodium ions are not present [1, 9].
2. Potassium compounds are more volatile than sodium compounds. Therefore, the potassium compounds take less time to produce a colour in flame than the sodium compounds. Violet colour produced by the potassium flame is changed by the yellow colour of the sodium flame.
   It is, therefore, possible to observe the colour of the flame through a cobalt glass as it absorbs the yellow colour and releases the red-violet colour. The flame

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## CHAPTER 9

# Summary of Groups Analysis with General Questions

## SUMMARY ANALYSIS OF ALL GROUPS CATIONS (UNKNOWN SOLUTION)

| | |
|---|---|
| Precipitates contain the cations of the first group $PbCl_2$, $Hg_2Cl_2$, AgCl<br>White precipitate | Add 2M of HCl acid to an unknown solution, then cool the solution and filter it; make sure to acidify the filtered liquid. |
| Precipitate shows the existence of the cation of the second group.HgS (black), CuS(black), $Bi_2S_3$(brown), SnS(black) , SnS (yellow), CdS (yellow), $Sb_2S_3$ (orange), PbS(black) | Pass $H_2S_{(g)}$ to the acidic solution using thioacetamide. The first group can be precipitated to form sulphur in the form of yellow precipitation of $H_2S$ |
| Precipitate shows the existence of the cation of the third group. $Al(OH)_3$ (white), $Cr(OH)_3$ (green) , $Fe(OH)_3$ (brown) | Heat the filtered liquid to remove $H_2S$, then add cons of $HNO_3$ and heat it again to convert $Fe^{2+}$ into $Fe^{3+,}$ then add 2M of $NH_4Cl$, 2 M of NHOH to make the solution basic. |
| Precipitate shows the existence of the cation of the Fourth group. CoS (black), NiS(black), MnS(pink), ZnS(white). | Add 2M of $NH_4Cl$, 2 M of NHOH to the filtered liquid then pass $H_2S_{(g)}$ |
| Precipitate shows the existence of the cation of the fifth group. $CaCO_3$ (white) , $SrCO_3$ (white) , $BaCO_3$ (white) | Heat the filtered liquid to remove $H_2S$, then add 2 M of $(NH_4)_2CO_3$, 2M of $NH_4Cl$, 2 M of NHOH |
| Sixth group: $Mg^{2+}$ , $Na^+$ , $K^+$ | The filtered liquid of the fifth group contains the cations of the sixth group. |

* If the pH of the solution is too low, cadmium and zinc can be precipitated.

*The main reason for the formation of sulphur is iron (III) ions triangular, chromate ions or sulfite ions.

## SUMMARY OF THE PROPERTIES OF CATIONS GROUPS (GROUP I - GROUP VI)

| **Groups** | **Ions** | **Groups Regent** | **Common Characteristic** |
|---|---|---|---|
| First | Silver $Ag^+$, mercuros (I) $Hg_2^{2+}$, lead $Pb^{2+}$ | HCl | Their chlorides do not dissolve in hydrochloric acid |
| Second | Mercuric(II) $Hg^{2+}$, copper $Cu^{2+}$, bismuth $Bi^{3+}$, cadmium $Cd^{2+}$, Arsenic $As^{3+}$, Tin ($Sn^{4+}$, $Sn^{2+}$) | $H_2S$ in acidic medium, *i.e.*, HCl | Their sulfides do not dissolve in HCl acid, but the last three sulfide elements dissolve in $(NH_4)_2$ S |

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*(Table cont).....*

| | | | |
|---|---|---|---|
| Third | Iron $Fe^{3+}$, Aluminium $Al^{3+}$, Chrome $Cr^{3+}$ | $NH_4Cl$ and $NH_4OH$ | Precipitate in the form of hydroxides |
| Fourth | Zinc $Zn^{2+}$, Magnesim $Mn^{2+}$, Nickle $Ni^{2+}$, Cobalt $Co^{2+}$ | $NH_4Cl$, $NH_4OH$ and $H_2S$ | Precipitate in the form of sulphides in the basic medium. |
| Fifth | Calcium $Ca^{2+}$, Strontium $Sr^{2+}$, Barium $Ba^{2+}$ | $NH_4Cl$, $NH_4OH$ and $(NH_4)_2CO_3$ as well as the flame test. | Precipitate in the form of carbonate dissolved in mineral acids. |
| Sixth | Ammonium $NH_4^+$, sodium $Na^+$, potassium $K^+$, magnesium $Mg^{2+}$ | They did not have a specific group regent but the flame test can be characteristic of some of them. | They do not form precipitate expect magnesium. |

## GENERAL QUESTIONS

1. How to separate the $Ag^+$ silver ion from lead ion into the mixture:
   a) An unknown solid that is likely to be $AgNO_3$ or $Hg_2Cl_2$ or pb$(CH_3COO)_2$. Will this be unidentified if it gives purple-red precipitate when adding $K_2CrO_4$ to this solution?
   b) Precipitate also turns white when adding HCl.
2. Are silver halides affected by light? Describe the results.
3. Why the acidity should not be greater than (0.3 N) when cation group II is precipitated? Why should not it be much lower than 0.3 N?
4. Solution contains ions: $Cu^{2+}$, $pb^{2+}$, $Bi^{3+}$, $Hg^{2+}$
   a) Which ion will precipitate when a few of $NH_4OH$ ions are added to the previous solution so that it becomes weak alkaline?
   b) What precipitates when HCl is added to this solution?
   c) What is precipitated when the $NH_4OH$ is added to the original solution?
5. How to separate the ions from each other?
6. Which is the reagent that dissolves CuS and does not dissolve HgS?
7. Which is the reagent that dissolves $Cu(OH)_2$ and does not dissolve $Bi(OH)_3$?
8. Write the chemical equations that represent:
   a) The solubility of HgS in Aqua Regia.

b) Effect of concentrated $NH_4OH$ on copper ion?

9. Using ammonium sulphide solution,

   a) How to separate the ions of the cation group II?

10. What are the ions that have an amphoteric property? Explain this with the chemical equations.
11. Why does $Al(OH)_3$ dissolve in an excess amount of NaOH and $NH_4OH$?
12. Which reagent is to be used to separate the following pairs?

    a) $Cr^{3+}$, $Fe^{3+}$

    b) $Al^{3+}$, $Fe^{3+}$

13. Which cations of group IV

    a) form a complex with ammonia?

    b) form amphoteric hydroxides?

14. If adding an excess amount of ammonium hydroxide to an unknown solution that may contain $Co^{2+}$, $Ni^{2+}$, $Mn^{2+}$, no precipitate is formed. What is your conclusion?
15. The acidic solution contains the following ions:
    ($Ni^{2+}$, $Al^{3+}$, $Sb^{3+}$, $Ag^{+}$)

    a) 5 drops of this solution are added in a test tube and then about 2 ml of water is added, and white precipitates appear. Explain your result.

    b) What happens when you add a little of $NH_4OH$ to this solution?

    c) What happens if an excess amount of $NH_4OH$ is then added?

    d) What will be precipitated in the original solution if the pH is 0.3 and $H_2S$ is passed through the solution?

    e) How to separate these five ions from each other?

    f) What will be precipitated in the original solution if it is alkaline and $H_2S$ free?

16. Cations of group II and group IV are precipitated in the form of sulfides, discuss these terms, indicating the fundamental difference between them.

*Principles of Qualitative Inorganic Analysis*, **2021, 57-61**

# APPENDIX

## APPENDIX

**Table 1. The solubility constant for compounds at 25°C.**

| **Compounds** | **Formula** | **$K_{sp}$** |
|---|---|---|
| Barium carbonate | $BaCO_3$ | $2.58 \times 10^{-9}$ |
| Barium sulphate | $BaSO_4$ | $1.08 \times 10^{-10}$ |
| Cadmium carbonate | $CdCO_3$ | $1.0 \times 10^{-12}$ |
| Cadmium hydroxide | $Cd(OH)_2$ | $7.2 \times 10^{-15}$ |
| Calcium carbonate | $CaCO_3$ | $3.36 \times 10^{-9}$ |
| Calcium hydroxide | $Ca(OH)_2$ | $5.02 \times 10^{-6}$ |
| Calcium phosphate | $Ca_3(PO_4)_2$ | $2.07 \times 10^{-33}$ |
| Calcium sulphate | $CaSO_4$ | $4.93 \times 10^{-5}$ |
| Cobalt(II) hydroxide | $Co(OH)_2$ | $5.92 \times 10^{-15}$ |
| Copper(I) chloride | $CuCl$ | $1.72 \times 10^{-7}$ |
| Copper(I) thiocyanate | $CuSCN$ | $1.77 \times 10^{-13}$ |
| Lead(II) carbonate | $PbCO_3$ | $7.40 \times 10^{-14}$ |
| Nickel(II) carbonate | $NiCO_3$ | $1.42 \times 10^{-7}$ |
| Nickel(II) hydroxide | $Ni(OH)_2$ | $5.48 \times 10^{-16}$ |
| Silver(I) carbonate | $Ag_2CO_3$ | $8.46 \times 10^{-12}$ |
| Strontium carbonate | $SrCO_3$ | $5.60 \times 10^{-10}$ |
| Zinc hydroxide | $Zn(OH)_2$ | $3 \times 10^{-17}$ |

**Table 2. List of some acids.**

| **Name** | **Formula** |
|---|---|
| Acetylsalicylic acid | $CH_3CO_2\text{-}C_6H_4\text{-}COO\underline{H}$ |
| Adipic acid | $\underline{H}OOC\text{-}(CH_2)_4\text{-}COO\underline{H}$ |
| Benzoic acid | $C_6H_5\text{-}COO\underline{H}$ |
| Chlorobenzoic acid | $ClC_6H_4\text{-}COO\underline{H}$ |
| Malic acid | $\underline{H}OOC\text{-}CHOH\text{-}CH_2\text{-}COO\underline{H}$ |

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***(Table 2) cont.....***

| | |
|---|---|
| Malonic acid | $\underline{H}OOC\text{-}CH_2\text{-}COO\underline{H}$ |
| Oxalic acid dehydrate | $\underline{H}OOC\text{-}COO\underline{H}.\ 2H_2O$ |
| Phenylacetic acid | $C_6H_5\text{-}CH_2\text{-}COO\underline{H}$ |
| Salicylic acid | $\underline{H}O\text{-}C_6H_4\text{-}COO\underline{H}$ |
| Succinic acid | $\underline{H}OOC\text{-}(CH_2)_2\text{-}COO\underline{H}$ |
| Tartaric acid | $\underline{H}OOC\text{-}CHOH\text{-}CHOH\text{-}COO\underline{H}$ |
| Trichloroacetic acid | $Cl_3C\text{-}COO\underline{H}$ |

**Note:** *The acidic hydrogen atoms are underlined.*

**Table 3. List of acids with their information.**

| **Acid** | **Sp.gr** | **Percentage** | **M.Wt** |
|---|---|---|---|
| HCl | 1.18 | 36 % | 36.45 |
| $HNO_3$ | 1.42 | 70 % | 63 |
| $H_2SO_4$ | 1.835 | 98 % | 98.06 |
| $H_3PO_4$ | 1.75 | 85 % | 97.97 |
| $HClO_4$ | 1.54 | 61 % | 100.45 |
| HI | 1.70 | 75 % | 127.9 |
| HBr | 1.49 | 48 % | 80.9 |
| HF | 1.125 | 98 % | 20 |
| $CH_3COOH$ | 1.05 | 99.5 % | 60 |

**Table 4. List of strong acids and weak acids.**

| **Strong Acids** | **Weak Acids** |
|---|---|
| Hydrochloric acid, HCl | Acetic acid, $CH_3COOH$ |
| Hydrobromic acid, HBr | Hydrocyanic acid, HCN |
| Hydriodic acid, HI | Hydrofluoric acid, HF |
| Nitric acid, $HNO_3$ | Nitrous acid, $HNO_2$ |
| Sulfuric acid, $H_2SO_4$ | Sulphurous acid, $H_2SO_3$ |
| Perchloric acid, HClO | Hypochlorous acid, HOCl |
| Periodic acid, $HIO_4$ | Phosphoric acid,$H_3PO_4$ |

**Table 5. List of strong Bases and list of weak Bases.**

| Strong Bases | Weak Bases |
|---|---|
| Sodium hydroxide $NaOH$ | Ammonia $NH_3$ |
| Potassium hydroxide $KOH$ | Sodium carbonate $Na_2CO_3$ |
| Calcium hydroxide $Ca(OH)_2$ | Potassium carbonate $K_2CO_3$ |
| Barium hydroxide $Ba(OH)_2$ | Aniline $C_6H_5NH_2$ |
| Sodium phosphate $Na_2PO_4$ | Trimethylamine $(CH_3)_3N$ |

**Table 6. Conjugate Acid-Base Pairs.**

| Acid | Conjugate Base |
|---|---|
| Hydrochloric acid $HCl$ | Chloride ion $Cl^-$ |
| Sulfuric acid $H_2SO_4$ | Hydrogen sulphate ion $HSO_4^-$ |
| Hydronium ion, $H_3O^+$ | Water $H_2O$ |
| Hydrogen sulphate ion $HSO_4^-$ | sulphate ion $SO_4^{2-}$ |
| hypochlorous acid $HOCl$ | Hypochlorite ion $ClO^-$ |
| Dihydrogen phosphate ion $H_2PO_4^-$ | Monohydrogen phosphate ion $HPO_4^{2-}$ |
| Ammonium ion $NH_4^+$ | Ammonia $NH_3$ |
| Hydrogen carbonate ion $HCO_3^-$ | Carbonate ion $CO_3^{2-}$ |
| Water $H_2O$ | Hydroxide ion $OH^-$ |
| **Conjugate acid** | **Base** |

**Table 7. The concentration of strong acids and bases and the value of Kw.**

| Solution | $[H_3O^+]M$ | $[HO^-]M$ | $K_w = [H_3O^+][HO^-]$ |
|---|---|---|---|
| Pure water | $1.0\ x10^{-7}$ | $1.0\ x10^{-7}$ | $1.0\ x10^{-14}$ |
| 0.10 M strong acid | $1.0\ x10^{-1}$ | $1.0\ x10^{-13}$ | $1.0\ x10^{-14}$ |
| 0.010 M strong acid | $1.0\ x10^{-2}$ | $1.0\ x10^{-12}$ | $1.0\ x10^{-14}$ |
| 0.10 M strong base | $1.0\ x10^{-13}$ | $1.0\ x10^{-1}$ | $1.0\ x10^{-14}$ |

***(Table 7) cont.....***

| | | | |
|---|---|---|---|
| 0.010 M strong base | 1.0 x10$^{-12}$ | 1.0 x10$^{-2}$ | 1.0 x10$^{-14}$ |
| 0.025 M strong acid | 2.5 x10$^{-2}$ | 4.0 x10$^{-13}$ | 1.0 x10$^{-14}$ |
| 0.025 M strong base | 4.0 x10$^{-13}$ | 2.5 x10$^{-2}$ | 1.0 x10$^{-14}$ |

**Table 8. pH values at specified $[H_3O^+]$.**

| **Solution** | **$[H_3O^+]$M** | **pH** |
|---|---|---|
| 1.00 L of $H_2O$ | 1.0 x10$^{-7}$ | 7.00 |
| 0.100 mol HCL in 1.00 L of $H_2O$ | 1.0 x10$^{-1}$ | 1.00 |
| 0.0100 mol HCL in 1.00 L of $H_2O$ | 1.0 x10$^{-2}$ | 2.00 |
| 0.100 mol NaCl in 1.00 L of $H_2O$ | 1.0 x10$^{-7}$ | 7.00 |
| 0.0100 mol NaOH in 1.00 L of $H_2O$ | 1.0 x10$^{-12}$ | 12.00 |
| 0.100 mol NaOH in 1.00 L of $H_2O$ | 2.5 x10$^{-13}$ | 13.00 |

**Table 9. Transition ranges of some indicators.**

| **Indicator Name** | **Acid Colour** | **Transition Rang (pH)** | **Base Colour** |
|---|---|---|---|
| Thymol blue | Red | 1.2-2.8 | Yellow |
| Methyl orange | Red | 3.1-4.4 | Orange |
| Litmus | Red | 5.0-8.0 | Blue |
| Bromothymol blue | Yellow | 6.0-7.6 | Blue |
| Thymol blue | Yellow | 8.0-9.6 | Blue |
| Phenolphthalein | Colourles | 8.0-9.6 | Red |
| Alizarin yellow | Yellow | 10.1-12.0 | Red |

**Table 10. Relative strengths of acids and Bases.**

| Increasing acid strength | **Acid** | **Formula** | **$K_a$ of Acid** | **Conjugate Base** | **Formula** | Increasing base strength |
|---|---|---|---|---|---|---|
| | Hydronium ion | $H_3O^+$ | $5.53 \times 10^1$ | Water | $H_2O$ | |
| | Hydrogen sulphate ion | $HSO_4^-$ | $1.23 \times 10^{-2}$ | Sulphate ion | $SO_4^{2-}$ | |
| | Formic acid | $HCOOH$ | $1.82 \times 10^{-4}$ | Formate ion | $HCOO^-$ | |
| | Benzoic acid | $C_6H_5COOH$ | $6.46 \times 10^{-5}$ | Benzoate ion | $C_6H_5COO^-$ | |
| | Acetic acid | $CH_3COOH$ | $1.75 \times 10^{-5}$ | Acetate ion | $CH_3COO$- | |
| | Carbonic acid | $H_2CO_3$ | $4.30 \times 10^{-7}$ | Hydrogen carbonate ion | $HCO_3^-$ | |
| | Dihydrogen phosphate ion | $H_2PO_4^-$ | $6.31 \times 10^{-8}$ | Monohydrogen phosphate ion | $HPO_4^{2-}$ | |
| | Hypochlorous acid | $HOCl$ | $2.95 \times 10^{-9}$ | Hypochlorite ion | $ClO^-$ | |
| | Ammonium ion | $NH_4^+$ | $5.57 \times 10^{-10}$ | Ammonia | $NH_3$ | |
| | Hydrogen carbonate ion | $HCO_3^-$ | $4.68 \times 10^{-11}$ | Carbonate ion | $CO_3^{2-}$ | |
| | Monohydrogen phosphate ion | $HPO_4^{2-}$ | $4.47 \times 10^{-13}$ | Phosphate ion | $PO_4^{3-}$ | |
| | Water | $H_2O$ | $1.81 \times 10^{-16}$ | Hydroxide ion | $OH^-$ | |
| | **Conjugate acid** | **Formula** | **$K_a$ of acid** | **Base** | **Formula** | |

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This is one of the fundamental books that university students benefit from in the first stage of their studies for many colleges, including the College of Sciences and Faculties of Engineering because it contains scientific information that helps in understanding the separation process and the detection of cations in inorganic compounds. Besides, this book contains a description of practical methods and chemical safety in laboratories with an explanation of how to write a laboratory report. The importance of the book is not limited to undergraduate students, but also to graduate students, academics, and workers in many fields of science and various industries.