THE NATIONAL EXAMINATIONS COUNCIL OF TANZANIA

QUALITATIVE ANALYSIS GUIDE FOR THE ADVANCED CERTIFICATE OF SECONDARY EDUCATION EXAMINATION

132 CHEMISTRY

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PREFACE

During monitoring of the national examinations, the National Examinations Council of Tanzania (NECTA) observed candidates using different Qualitative Analysis Guides (QAG) in chemistry practical examinations. It was further noted that, some of the guides were written by different authors and were not approved by the Ministry of Education, Science and Technology. It was also found that, some of the guides had technical errors such as incorrect chemical symbols and formulae, inconsistent information and typographic errors. This guide was therefore developed in order to have a uniform document that will be used by candidates when writing their chemistry practical examinations at Advanced Certificate of Secondary Education Examination (ACSEE) level.

This guide is based on 2010 advanced level secondary education chemistry syllabus and was used in 2018 in Advanced Certificate of Secondary Education Examination (ACSEE) assessment. However, it was inevitable for NECTA to accommodate the recommendations and improvements from stakeholders, thus, the need to revise the first edition of 2016. In addition to the updating the first edition, the current one contains essential materials which were found to be useful. This revised QAG will be effectively used from 2019 in ACSEE.

Prospective candidates are therefore encouraged to read and use this booklet effectively so that they can be conversant with the procedures indicated and be in a good position of using it properly when writing their examinations. It is also hoped that the guide will give an extra support to teachers and students in conducting analytical experiments.

Dr. Charles E. Msonde
EXECUTIVE SECRETARY

INTRODUCTION

The National Examinations Council of Tanzania (NECTA) has prepared this Qualitative Analysis Guide (QAG) to support candidates in identification of ions that are present in unknown compounds through several chemical tests. For Advanced Certificate of Secondary Education Examinations (ACSEE), candidates are required to analyze unknowns which are always ionic compounds. This guide is designed to assist candidates to analyze the following ions in accordance to the 2010 Chemistry Syllabus for Advanced Secondary Schools.

Cations: NH_4^+, Na^+, K^+, Mg^{2+}, Ba^{2+}, Ca^{2+}, Sr^{2+}, Zn^{2+}, Mn^{2+}, Ni^{2+}, Co^{2+}, Fe^{3+}, Al^{3+}, Cr^{3+}, Sb^{3+}, Sn^{2+}, Fe^{2+}, Cu^{2+}, Cd^{2+}, Bi^{3+}, Pb^{2+} and Ag^+

Anions: Cl^-, SO_4^{2-}, NO_2^-, NO_3^-, CO_3^{2-}, HCO_3^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, $C_2O_4^{2-}$ and CH_3COO^-

The content in this guide is divided into sections: A, B, C and D. Section A is on preliminary tests, Section B on confirmatory tests of anions, Section C on tests in solution and Section D is on group separation, analysis and confirmatory tests of cations.

RECORDING ANALYTICAL EXPERIMENTS

The candidates are required to record the analytical experiments in a tabular form. Generally tables with three columns should be used, showing a brief explanation of the experimental procedures or tests performed, observations and inferences made as shown in Table 1.

Table 1: Format for Recording Analytical Experiments.

Experiment	Observation	Inference

Tests carried on an unknown solid compound or its solution should be written in the "Experiment" column. Experiments should be reported in simple past tense (in most cases in "passive voice") to explain what was performed. For example, "A small amount of a sample was picked using a clean nichrome wire and heated on a flame". The facts or changes which have been observed or identified when a chemical substance under test is subjected to an experiment should be written in the "Observation" column. These observations include: colour changes, formation of precipitates, evolution of gases, flame colours, sound and other observations. The deductions or what can be inferred from the observation is written in the "Inference" column. These inferences lead to the identification of the unknown salt under investigation.

It should be known that not all tests will give detectable changes. Sometimes if no obvious changes are observed, it can infer to the presence or absence of a particular ion. For instance, addition of barium chloride solution in the unknown solution may or may not give observable changes. In this case if no reaction occurs, it implies the absence of sulphate ion while formation of white precipitate indicates the presence of sulphate ion in the unknown sample.

After performing all experiments, it is required to make conclusions about ions present in the sample by performing the confirmatory tests for every deduced ion. This can be obtained by combining all the inferences made in the successive tests. The following Sections are on the stages of qualitative analysis.

SECTION A: PRELIMINARY TESTS

The preliminary tests included in this QAG are generally for solid samples. The tests include colour, texture, odour, deliquescence, flame test, action of heat, action of dilute and concentrated acids and solubility in water.

[***Safety Precautions***: *Avoid direct smelling of any chemical in the laboratory]*.

Table 2: Preliminary Tests

S/n	Experiment	Observations	Inference
1.	**Appearance of the Sample**		
	(i) Colour	White	Non-transition metals may be present.
		Blue	Cu^{2+}, Co^{2+} may be present.
		Green	Fe^{2+}, Ni^{2+}, Cr^{3+}, Cu^{2+} may be present.
		Yellow	Fe^{3+}, CrO_4^{2-} may be present.
		Brown/Yellowish-brown	Fe^{3+} may be present.
		Pink	Co^{2+}, Mn^{2+} may be present.
		Orange	$Cr_2O_7^{2-}$ may be present.
	(ii) Texture	Crystalline form	NO_3^-, SO_4^{2-}, Cl^-, $C_2O_4^{2-}$, CrO_4^{2-}, NO_2^-, CH_3COO^-, $Cr_2O_7^{2-}$ may be present.
		Powder form	CO_3^{2-} and HCO_3^- may be present except CO_3^{2-} of NH_4^+, K^+ and Na^+
	(iii) Odour	Choking smell	NH_4^+ may be present.
	(iv) Deliquescence	Absorbs water from the atmosphere to form solution.	NO_3^-, Cl^-, SO_4^{2-} may be present.
2.	**Flame Test** *Cleaning the test apparatus*: Dip a nichrome wire or glass rod or back side of the test-tube in concentrated HCl (in a watch glass) then heat it in a non-luminous flame. *Test*: Dip the cleaned wire (or glass rod or test-tube) in concentrated HCl, then to the	Bright yellow/golden yellow.	Na^+ may be present.
		Brick red	Ca^{2+} may be present.
		Lilac (light purple)	K^+ may be present.
		Green	Ba^{2+} may be present.

S/n	Experiment	Observations	Inference
	sample followed by heating it on a flame.	Red	Sr^{2+} may be present.
		Blue-green	Cu^{2+} may be present.
		Blue	Pb^{2+}, Sb^{2+} may be present.
		Yellow sparks	Fe^{2+}, Fe^{3+} may be present.
3.	**Action of Heat on a Solid Sample** Transfer small amount (about 0.5 g) of a sample in a clean dry test-tube and heat the contents gently and then strongly.	Colourless droplets forming on the cooler part of the test-tube. The droplets turn anhydrous $CuSO_4$ blue or $CoCl_2$ pink.	Hydrated salt, HCO_3^- may be present.
		White sublimate and a colourless gas with chocking smell which turns moist red litmus paper blue.	NH_4^+ may be present.
		Colourless gas evolves which turn moist litmus paper from blue to red and form dense white fumes with ammonia gas.	Cl^- may be present.
		Colourless gas evolves which re-lights a glowing wooden splint.	NO_3^- of Na^+, K^+ may be present.
		Brown fumes evolve which turn moist blue litmus paper red and a gas which re-lights a glowing wooden splint.	NO_3^- may be present except those of Na^+, K^+ and NH_4^+.
		Colourless gas evolves which turns lime water milky.	CO_3^{2-}, HCO_3^- may be present.
		Colourless gas with pungent smell evolves, which turns moist blue litmus paper red or moist potassium dichromate paper green or decolorizes potassium permanganate solution.	SO_4^{2-} may be present.
		Colourless vapour with a smell of vinegar evolves.	CH_3COO^- may be present.

S/n	Experiment	Observations	Inference
		Colourless gas which burns with blue flame evolves.	$C_2O_4^{2-}$ may be present.
		Cracking sound with evolution of brown gas.	NO_3^- of Pb^{2+} may be present.
		Cracking sound with no gas evolving.	Cl^- of Na^+ or K^+ may be present.
		Residue that are reddish brown when hot and yellow when cold.	Pb^{2+} may be present.
		Residue that is yellow when hot and white when cold.	Zn^{2+} may be present.
		Black residue.	Cu^{2+} may be present.
		Reddish brown residue.	Fe^{2+}, Fe^{3+} may be present.
4.	**Action of Dilute HCl on a Solid Sample** Transfer small amount of a sample in a clean test-tube followed by a small amount of dilute HCl. If no reaction, warm the contents gently.	Effervescence of a colourless gas which turns lime water milky and moist litmus paper from blue to red.	CO_3^{2-}, HCO_3^- may be present.
		Brown fumes evolve, which turn moist litmus paper from blue to red.	NO_2^- may be present.
		No gas evolves.	SO_4^{2-}, Cl^-, NO_3^- may be present.
		White precipitate.	Ag^+ may be present.
		White precipitate soluble on warming.	Pb^{2+} may be present.
5.	**Action of Concentrated H_2SO_4 on a Solid Sample** ***[Safety Precautions:*** *Concentrated H_2SO_4 is corrosive. (a) Handle with care (b) Do not boil].* Transfer a small amount of a sample in a clean and dry test-tube. Add a small amount of concentrated H_2SO_4. If no reaction warm the contents gently.	Colourless gas with irritating smell evolves, which turns moist litmus paper from blue to red and forms dense white fumes with ammonia gas.	Cl^- may be present.

S/n	Experiment	Observations	Inference
		Brown fumes evolve, which turn moist blue litmus paper red, and intensify on addition of copper turnings.	NO_3^- may be present.
		Colourless vapour with vinegar smell evolves.	CH_3COO^- may be present.
		Upon warming, effervescence of a colourless gas evolves that: (i) turns lime water milky. (ii) burns with a blue flame.	$C_2O_4^{2-}$ may be present.
		Effervescence of a colourless gas which turns lime water milky.	CO_3^{2-}, HCO_3^- may be present.
		No gas evolves.	SO_4^{2-} may be present.
		Blue crystals turn white.	SO_4^{2-} of hydrated Cu^{2+} may be present.
6.	**Solubility of a Solid Sample** Transfer a small amount of a sample in a clean test-tube. Add enough amount of distilled water to dissolve it. If the sample does not dissolve, warm the contents.	Soluble in cold water.	(i) NO_3^-, CH_3COO^-, HCO_3^- may be present. (ii) SO_4^{2-} may be present except those of Ba^{2+}, Sr^{2+}, Ca^{2+} and Pb^{2+}. (iii) Cl^- may be present except those of Ag^+ and Pb^{2+}. (iv) Na^+, K^+, NH_4^+ may be present. (v) CO_3^{2-} of Na^+, K^+, NH_4^+ may be present. (vi) $C_2O_4^{2-}$ of Na^+, K^+, NH_4^+ may be present.
		Soluble in hot water.	Cl^- of Pb^{2+} may be present.
		Insoluble in hot or cold water.	(i) SO_4^{2-} of Ba^{2+}, Sr^{2+}, Ca^{2+}, Pb^{2+} may be present. (ii) Cl^- of Ag^+ may be present. (iii) CO_3^{2-} may be present except those of Na^+, K^+, NH_4^+. (iv) $C_2O_4^{2-}$ may be present except those of Na^+, K^+, NH_4^+.

SECTION B: CONFIRMATORY TESTS FOR ANIONS

Analysis of Anions

The analysis of anions requires the samples to be in aqueous solution. However, some of the salts are insoluble even in hot water. The anions of these salts are made to be soluble in water by converting them into sodium salts by using sodium carbonate, i.e., while the anions are solubilized, the cations are precipitated as carbonates. The precipitates formed during this process are filtered out. The resulting supernatant obtained in this process is called "sodium carbonate extract of the salt".

Preparation of Sodium Carbonate Extract of the Salt

In a clean beaker, mix a small amount (about 1 g) of the sample with a small amount (about 2 g) of solid sodium carbonate. Add distilled water to the mixture up to about half of the 100 cm^3 beaker. Boil the contents in the beaker for few minutes. Filter or centrifuge the mixture to remove the precipitate formed. The supernatant obtained contains sodium salts of the anions and is used for their analysis by following the procedure shown in Table 3.

Table 3: Confirmatory Tests for Anions using Sodium Carbonate Extract of Salt

S/n	Experiment	Observations	Inference
1.	**Confirmatory Tests for SO_4^{2-}**		
	(a) Transfer a small volume (about 1 cm^3) of the extract into the test-tube. Add barium chloride solution followed by dilute HCl or barium nitrate solution followed by dilute HNO_3.	White precipitate is formed insoluble in dilute HCl or HNO_3.	SO_4^{2-} confirmed.
	(b) Transfer a small volume of the extract into the test-tube. Add ethanoic acid followed by lead ethanoate. Divide the resulting mixture into two portions. In one portion add dilute HCl and in another add ammonium ethanoate solution.	White precipitate insoluble in dilute HCl but soluble in ammonium ethanoate solution is formed.	SO_4^{2-} confirmed.
2.	**Confirmatory Tests for Cl^-**		
	(a) Transfer a small volume of the extract into the test-tube. Add dilute HNO_3 followed by $AgNO_3$ solution then $NH_3(aq)$.	White precipitate soluble in dilute ammonia solution is formed.	Cl^- confirmed.
	(b) Transfer a small amount of the original solid sample into the test-tube. Add potassium dichromate solution followed by few drops of concentrated H_2SO_4.	Red orange vapour is observed.	Cl^- confirmed.

S/n	Experiment	Observations	Inference
	(c) Transfer a small amount of the original solid sample into the test-tube. Add equal amount of solid MnO_2 followed by few drops of concentrated H_2SO_4.	Greenish yellow gas evolves which bleaches moist red litmus paper.	Cl^- confirmed.
3.	**Confirmatory Tests for NO_2^-** (a) Transfer a small volume of the extract into the test-tube. Add dilute H_2SO_4 followed by freshly prepared $FeSO_4$ solution.	Brown solution is formed.	NO_2^- confirmed.
	(b) Transfer a small volume of the extract into the test-tube. Add KI solution followed by concentrated HCl.	Dark brown precipitate is formed.	NO_2^- confirmed.
4.	**Confirmatory Tests for NO_3^-** (a) Transfer a small volume of the extract into the test-tube. Add dilute H_2SO_4 then freshly prepared $FeSO_4$ solution followed by careful addition of concentrated H_2SO_4 along the side of the test-tube.	Brown ring is formed at the junction of the liquids.	NO_3^- confirmed.
	(b) Transfer a small amount of the original solid sample into the test-tube. Add copper turnings followed by concentrated H_2SO_4 then warm.	Brown fumes evolve.	NO_3^- confirmed.
	(c) Transfer a small amount of the original solid sample into the test-tube. Add Al or Zn metal followed by NaOH solution, then heat.	A colourless gas with a choking smell which turns moist red litmus paper to blue.	NO_3^- confirmed.
5.	**Confirmatory Tests for $C_2O_4^{2-}$** (a) Transfer a small volume of the extract into the test-tube. Add a small volume of $CaCl_2$ solution drop wise. Divide the resulting mixture into two portions. In one portion add dilute ethanoic acid. In another portion add dilute HCl or dilute HNO_3.	White precipitate forms, insoluble in dilute ethanoic acid but soluble in dilute HCl or HNO_3.	$C_2O_4^{2-}$ confirmed.

S/n	Experiment	Observations	Inference
	(b) Transfer a small volume of the extract into the test-tube. Add dilute H_2SO_4 followed by one drop of potassium permanganate solution and warm.	Acidified potassium permanganate is decolourized.	$C_2O_4^{2-}$ confirmed.
	(c) Transfer a small volume of the extract into the test-tube. Add $AgNO_3$ solution. If precipitate is formed, add dilute ammonia solution.	White precipitate, soluble in dilute ammonia solution is formed.	$C_2O_4^{2-}$ confirmed.
	(d) Transfer a small volume of the extract into the test-tube. Add concentrated H_2SO_4.	Colourless gas which turns lime water milky and burns with a blue flame evolves.	$C_2O_4^{2-}$ confirmed.
	(e) Transfer a small volume of the extract into the test-tube. Add $BaCl_2$ or $Ba(NO_3)_2$ solution. If precipitate is formed, add dilute ammonium chloride or dilute HCl.	White precipitate soluble in dilute ammonium chloride or dilute HCl is formed.	$C_2O_4^{2-}$ confirmed.
6.	**Confirmatory Tests for CrO_4^{2-} and $Cr_2O_7^{2-}$** Transfer a small volume of the extract into the test-tube. Add dilute HNO_3 followed by ammonia solution until the solution becomes neutral then boil. (a) To the neutral solution, add $BaCl_2$ solution followed by dilute HCl.	Yellow precipitate, soluble in dilute HCl.	CrO_4^{2-} confirmed.
	(b) To the neutral solution, add $Pb(CH_3COO)_2$ or $Pb(NO_3)_2$ solution.	Yellow precipitate is formed.	CrO_4^{2-} confirmed.
	(c) Transfer a small volume of the extract into the test-tube. Add few drops of dilute NaOH solution.	The solution changes from orange to yellow.	$Cr_2O_7^{2-}$ confirmed.
	(d) To the neutral solution, add $Pb(CH_3COO)_2$ or $Pb(NO_3)_2$ solution.	Orange precipitate is formed.	$Cr_2O_7^{2-}$ confirmed.

S/n	Experiment	Observations	Inference
7.	**Confirmatory Test for CH_3COO^-** Transfer a small volume of the extract into the test-tube. Add dilute HNO_3 followed by ammonia solution until the solution becomes neutral then boil. (a) To the neutral solution, add $FeCl_3$ solution.	Deep red colour is observed.	CH_3COO^- confirmed.
	(b) To the neutral solution, add concentrated H_2SO_4 followed by ethanol solution.	Smell of vinegar.	CH_3COO^- confirmed.

Table 3: Confirmatory Test for CO_3^{2-} and HCO_3^-

S/n	Experiment	Observations	Inference
1.	**Confirmatory Test for Soluble CO_3^{2-} and HCO_3^-** (a) Transfer a small volume of the solution of the original sample into a test-tube. Add few drops of $MgSO_4$ solution. If no precipitate is formed, warm the contents.	White precipitate is formed before warming the contents.	CO_3^{2-} confirmed.
		White precipitate is formed after warming the contents.	HCO_3^- confirmed.
	(b) Transfer a small volume of the solution of the original sample into a test-tube. Add few drops of $CaCl_2$ solution. If no precipitate is formed warm the contents.	White precipitate is formed before warming the contents.	CO_3^{2-} confirmed.
		White precipitate is formed after warming the contents.	HCO_3^- confirmed.
	(c) Transfer a small volume of the solution of the original sample into a test-tube. Add barium chloride solution followed by dilute HCl (or barium nitrate solution followed by dilute HNO_3).	White precipitate soluble in dilute HCl (or HNO_3) is formed.	CO_3^{2-} confirmed.
2.	**Confirmatory Test for Insoluble CO_3^{2-}** Transfer a small amount of the original sample in a test-tube. Add a small volume of dilute HNO_3.	Effervescence of a colourless gas, which turns lime water milky.	CO_3^{2-} confirmed.

SECTION C: TESTS IN SOLUTION

Action of NaOH(aq) and NH_4OH(aq) on Solutions of Samples

In these experiments the solutions of samples are treated with alkali solutions drop-wise until in excess. **However, these may be used for guided qualitative analysis and not to be included in systematic qualitative analysis**. The expected observations are summarized in Table 4.

Table 4: The Action of NaOH(aq) and NH_4OH(aq) on Sample Solutions

Addition of 1 or 2 drops of NaOH(aq) or NH_4OH(aq)	Addition of Excess NaOH(aq)	Addition of Excess NH_4OH(aq)	Inference
White precipitate.	Precipitate dissolves.	Precipitate dissolves.	Zn^{2+} may be present.
White precipitate.	Precipitate dissolves.	Insoluble precipitate.	Sn^{2+}, Pb^{2+} may be present.
White precipitate.	Insoluble precipitate.	Insoluble precipitate.	Sb^{3+}, Bi^{3+}, Mg^{2+} may be present.
White precipitate.	Precipitate dissolves.	Slightly soluble precipitate.	Al^{3+} may be present.
White precipitate.	Insoluble precipitate.	No precipitate.	Ca^{2+}, Sr^{2+}, Ba^{2+} may be present.
No precipitate.	–	–	K^{+}, Na^{+} may be present.
No precipitate.	Ammonia gas evolves on warming.	–	NH_4^{+} may be present.
Brown precipitate.	Insoluble precipitate.	Insoluble precipitate.	Ag^{+} may be present.
White precipitate which turns brown on exposure to air.	Insoluble precipitate.	Insoluble precipitate.	Mn^{2+} may be present.
Dirty green precipitate turns brown on exposure to air.	Insoluble precipitate.	Insoluble precipitate.	Fe^{2+} may be present.
Reddish brown precipitate.	Insoluble precipitate.	Insoluble precipitate.	Fe^{3+} may be present.
Pale green precipitate.	Insoluble precipitate.	Green solution.	Ni^{2+} may be present.
Pale blue precipitate (black on heating).	Insoluble precipitate.	Deep blue solution.	Cu^{2+} may be present.
Blue precipitate (turns pink on heating).	Green solution.	Insoluble precipitate.	Cr^{3+} may be present.
Blue precipitate (turns pink on warming).	Insoluble precipitate.	Precipitate dissolves.	Co^{2+} may be present.
White precipitate.	Precipitate dissolves.	Insoluble precipitate.	Cd^{2+} may be present.

SECTION D: GROUP SEPARATION, ANALYSIS AND CONFIRMATORY TESTS FOR CATIONS

Group Separation

Preparation of Solutions for Group Separation

[***Safety Precautions***: *Concentrated acids are corrosive. Handle them with care*]

Dissolve the sample in cold solvent and if the sample is insoluble warm the contents. Follow the following order:

(i) Water.
(ii) Dilute HCl.
(iii) Concentrated HCl.
(iv) Dilute HNO_3.
(v) Concentrated HNO_3.
(vi) Aqua regia (3 parts concentrated HCl and 1 part concentrated HNO_3).

Note: *Retain the precipitate obtained in each stage of group separation for group analysis in Table 6.*

Table 5: Group Separation

Step	Experiment	Observations	Inference
1.	**GROUP I** Add dilute HCl to the solution of the sample. If no precipitate forms proceed directly to group (II) separation with the same solution.	If precipitate forms, filter or centrifuge and use the supernatant for Step 2.	Pb^{+}, Ag^{+} may be present.
2.	**GROUP II** Add hydrogen sulphide solution or pass hydrogen sulphide gas to the supernatant or solution from Step 1. If no precipitate forms proceed to Step 3 using the same solution or the original sample solution.	If precipitate forms, filter or centrifuge and then warm the supernatant to evaporate hydrogen sulphide. The resulting solution is used for Step 3.	Cu^{2+} (black precipitate) Sb^{3+} (orange precipitate) Sn^{2+} (brown precipitate) Cd^{2+} (yellow precipitate) Bi^{3+} (brown precipitate) may be present.
3.	**GROUP III** Add few drops of concentrated HNO_3, heat then cool. Add solid NH_4Cl	If precipitate forms, filter or centrifuge and use the supernatant in Step 4.	Fe^{3+} (brown precipitate) Al^{3+} (white precipitate) Cr^{3+} (green precipitate) may be present.

Step	Experiment	Observations	Inference
	followed by ammonia solution to the supernatant or solution from group II. If no precipitate forms go for Step 4.		
4.	**GROUP IV** Warm the solution and cool. Add hydrogen sulphide solution or ammonium sulphide solution or pass H_2S gas for few minutes (not less than 3 minutes). If no precipitate forms proceed directly to Step 5.	If precipitate forms, filter or centrifuge and use the supernatant in step 5	Co^{2+} (black precipitate) Ni^{2+} (black precipitate) Mn^{2+} (light pink precipitate) Zn^{2+} (dirty white precipitate) may be present.
5.	**GROUP V** Warm the solution or supernatant from step 4, add $(NH_4)_2CO_3$ solution. If no precipitate forms proceed to Step 6.	If precipitate forms, filter or centrifuge and use the supernatant in Step 6.	Ba^{2+} (white precipitate) Ca^{2+} (white precipitate) Sr^{2+} (white precipitate) may be present.
6.	**GROUP VI** Evaporate to dryness the supernatant or the solution from Step 5.	White residue remains.	Na^+, K^+, Mg^{2+}, NH_4^+ may be present.

Table 6: Group Analysis and Confirmatory Tests for Cations

S/n	Experiment	Observation	Inference
1.	**GROUP I** Add enough water to cover the precipitate and boil, then add ammonia solution.	Precipitate soluble in warm water before addition of ammonia solution.	Pb^{2+} may be present.
		Precipitate insoluble in warm water before addition of ammonia solution.	Ag^+ may be present.
		Precipitate soluble in ammonia solution.	Ag^+ may be present.
		Precipitate insoluble in ammonia solution.	Pb^{2+} may be present.

S/n	Experiment	Observation	Inference
	Confirmatory Tests for Pb^{2+} (i) Add K_2CrO_4 to the solution of the sample.	Yellow precipitate is formed.	Pb^{2+} confirmed.
	(ii) Add KI solution to the solution of the sample. Warm and cool the mixture.	Yellow precipitate is formed, which disappear on warming but re-appear on cooling.	Pb^{2+} confirmed.
	Confirmatory Tests for Ag^{+} (i) To a small volume of the sample solution, add excess dilute HCl.	White precipitate is formed.	Ag^{+} confirmed.
	(ii) To a small volume of the sample solution, add K_2CrO_4.	Brick red precipitate is formed.	Ag^{+} confirmed.
2.	**GROUP II** **(a) Separation of Sub-groups, IIa and IIb** Wash the precipitate by adding enough water to cover it, centrifuge and then decant. Add sodium hydroxide solution followed by few drops of yellow ammonium sulphide.	Precipitate insoluble in ammonium sulphide is formed.	Group IIa (Pb^{2+}, Bi^{2+}, Cu^{2+}, Cd^{2+}) may be present.
		Precipitate soluble in ammonium sulphide.	Group IIb (Sb^{3+}, Sn^{2+}) may be present.
	(b) Analysis of Group IIa Cover the precipitate with water and warm, followed by addition of concentrated HNO_3. (i) To the solution add dilute H_2SO_4.	White precipitate is formed.	Pb^{2+} may be present.
	(ii) To the filtrate of (i) add concentrated ammonia solution.	White precipitate is formed.	Bi^{3+} may be present.
		Blue solution is formed	Cu^{2+} may be present.
	(iii) To the filtrate of (i) add concentrated ammonia solution followed by H_2S gas or ammonium sulphide solution.	Colourless solution giving yellow precipitate on addition of H_2S gas.	Cd^{2+} may be present

S/n	Experiment	Observation	Inference
	Confirmatory Tests for Pb^{2+} (i) To a small volume of the original sample solution, add K_2CrO_4.	Yellow precipitate is formed.	Pb^{2+} confirmed.
	(ii) Add KI solution to the solution of the sample. Warm and cool the mixture.	Yellow precipitate is formed, which disappears on warming but re-appears on cooling.	Pb^{2+} confirmed.
	Confirmatory Tests for Cu^{2+} (i) Add excess ammonia solution to the solution of the sample.	Blue precipitate soluble in excess ammonia forming a deep blue (royal) solution.	Cu^{2+} confirmed.
	(ii) Add potassium hexacyanoferrate(II) solution to the solution of the sample.	Brownish-red gelatinous precipitate is formed.	Cu^{2+} confirmed.
	Confirmatory Tests for Bi^{3+} (i) Add potassium iodide solution to the solution of the sample.	Dark-brown precipitate which on dilution forms an orange precipitate.	Bi^{3+} confirmed.
	(ii) Add excess dilute HCl to the solution of the sample.	White precipitate soluble in excess dilute HCl.	Bi^{3+} confirmed.
	(iii) Add sodium hydroxide solution dropwise until in excess to the solution of the sample.	White precipitate which turns faint yellow on heating.	Bi^{3+} confirmed.
	Confirmatory Tests for Cd^{2+} (i) Add dilute HCl to the solution of the sample followed by H_2S gas.	Yellow precipitate insoluble in yellow ammonium sulphide.	Cd^{2+} confirmed.
	(ii) Add dilute ammonia solution to the solution of the sample.	White precipitate soluble in excess ammonia solution.	Cd^{2+} confirmed.
	(c) Analysis of Group IIb (i) Acidify the supernatant obtained in 2 (a) using dilute HCl.	Precipitate is formed.	Sb^{3+}, Sn^{2+} may be present.

S/n	Experiment	Observation	Inference
	(ii) Dissolve the precipitate obtained in 2 (c) (i) with hot concentrated HCl and divide the resulting solution into two portions.	Solution is formed.	Sb^{3+}, Sn^{2+} may be present.
	• To the first portion add equal volume of water followed by H_2S gas.	Orange precipitate is formed.	Sb^{3+} may be present.
	• To the second portion add a clean iron wire.	White precipitate which may turn grey.	Sn^{2+} may be present.
	Confirmatory Tests for Sn^{2+} (i) To the solution of the sample, add excess sodium hydroxide solution followed by silver nitrate solution.	Black precipitate is formed.	Sn^{2+} confirmed.
	(ii) To the solution of the sample add dilute H_2SO_4 followed by ammonium molybdate (VI).	Blue precipitate is formed.	Sn^{2+} confirmed.
	Confirmatory Tests for Sb^{3+} (i) To the solution of the sample add ammonium sulphide followed by small amount of solid oxalic acid.	Orange precipitate is formed.	Sb^{3+} confirmed.
	(ii) To the solution of the sample add potassium iodide solution.	Yellow solution is formed.	Sb^{3+} confirmed
3.	**GROUP III** (a) Wash the precipitate with warm water. Add sodium hydroxide solution followed by H_2O_2 and warm.	Brown precipitate is formed.	Fe^{3+} may be present.
		Solution is formed.	Cr^{3+}, Al^{3+} may be present.
	(b) Divide the solution obtained in 3 (a) into two portions. (i) Into one portion, add dilute ethanoic acid and few drops of lead(II) ethanoate.	Yellow precipitate is formed.	Cr^{3+} may be present.

S/n	Experiment	Observation	Inference
	(ii) Acidify the second portion with dilute HCl followed by one drop of litmus solution. Add ammonia solution to make the resulting solution just alkaline.	White precipitate which decolourizes the litmus dye is formed.	Al^{3+} may be present.
	Confirmatory Test for Fe^{2+} To the solution of the original sample add few drops of potassium hexacyanoferrate(III) (Potassium ferricyanide).	Dark blue precipitate is formed.	Fe^{2+} confirmed.
	Confirmatory Tests for Fe^{3+} (i) Add few drops of potassium hexacyanoferrate(II) (Potassium ferrocyanide) into the solution of the sample.	Dark blue precipitate is formed.	Fe^{3+} confirmed.
	(ii) To the solution of the sample add few drops of potassium or ammonium thiocyanate solution.	Deep blood-red colouration is formed.	Fe^{3+} confirmed.
	Confirmatory Tests for Cr^{3+} (i) To the solution of the sample add excess dilute NaOH until all grey precipitate dissolves, and then add dilute H_2O_2 solution followed by pentanol (amyl alcohol) and dilute H_2SO_4. Shake gently.	Blue colour concentrating in pentanol is formed.	Cr^{3+} confirmed.
	(ii) To the solution of the sample add excess dilute NaOH followed dilute H_2O_2 solution.	Yellow solution is formed.	Cr^{3+} confirmed.
	(iii) To the solution of the sample add dilute acetic acid followed by few drops of lead acetate.	Yellow precipitate is formed.	Cr^{3+} confirmed.
	Confirmatory Test for Al^{3+} Acidify the solution with dilute HCl and add few drops of litmus solution followed by ammonia solution.	Blue lake precipitate is formed.	Al^{3+} confirmed.

S/n	Experiment	Observation	Inference
4.	**GROUP IV** (i) Wash the precipitate by adding enough water to cover it, warm then filter or centrifuge and decant. After that add water to the precipitate followed by dilute HCl.	Solution is formed.	Mn^{2+}, Zn^{2+} may be present.
		Black residue is formed.	Ni^{2+}, Co^{2+} may be present.
	(ii) To the solution of 4 (i) add excess NaOH solution.	White precipitate which turns brown on exposure to air.	Mn^{2+} may be present.
		White precipitate soluble in excess NaOH solution.	Zn^{2+} may be present.
	(iii) To the residue in 4 (i) add concentrated HCl followed by crystals of $KClO_3$. Heat until all crystals have dissolved, then evaporate to nearly dryness.	Green-yellow solution depositing yellow crystals.	Ni^{2+} may be present.
		Pink solution depositing blue crystals.	Co^{2+} may be present.
	Confirmatory Tests for Mn^{2+} (i) To the solution of the sample, add dilute NaOH solution.	White precipitate which darkens on exposure to air is formed.	Mn^{2+} confirmed.
	(ii) To the solution of the sample add sodium bismuthate(V) solution followed by adding concentrated HNO_3 drop-wise.	Purple solution is formed.	Mn^{2+} confirmed.
	(iii) Boil the solution of the sample. Add solid lead dioxide and concentrated HNO_3.	Purple solution is formed.	Mn^{2+} confirmed.
	Confirmatory Tests for Zn^{2+} (i) To the solution of the sample add potassium hexacyanoferrate(II) solution.	Bluish-white precipitate is formed.	Zn^{2+} confirmed.
	(ii) To the solution of the sample add dilute $NaOH/NH_4OH$ solution until in excess.	White precipitate soluble in excess.	Zn^{2+} confirmed.

S/n	Experiment	Observation	Inference
	Confirmatory Tests for Co^{2+} (i) To the neutral or acidic solution of the sample add ammonium thiocyanate.	Blue solution is formed.	Co^{2+} confirmed.
	(ii) To the solution of the sample add NaOH solution followed by ammonia solution.	Blue precipitate which turns pink upon warming. The precipitate is soluble in ammonia solution.	Co^{2+} confirmed.
	(iii) To the neutral solution of the sample add concentrated KNO_2 solution.	Yellow precipitate is formed.	Co^{2+} confirmed.
	Confirmatory Tests for Ni^{2+} (i) To the solution of the sample add sodium hydroxide solution followed by ammonia solution.	Green precipitate is formed which is soluble in ammonia solution to give a deep blue solution.	Ni^{2+} confirmed.
	(ii) To the solution of the sample add dilute ammonia solution until just alkaline, followed by dimethyglyoxime reagent.	Red precipitate is formed.	Ni^{2+} confirmed.
	(iii) To the solution of the sample add ammonia solution until in excess.	Green precipitate soluble in excess ammonia solution forming a blue solution.	Ni^{2+} confirmed.
	(iv) To the solution of the sample add potassium hexacyanoferrate(II).	Green precipitate is formed.	Ni^{2+} confirmed.
	(v) To the solution of the sample add potassium hexacyanoferrate(III).	Brown precipitate is formed.	Ni^{2+} confirmed.
5.	**GROUP V** Wash the precipitate in hot water. Dissolve the precipitate in small amount of ethanoic acid. (i) To the prepared sample solution add potassium chromate(VI) solution. Leave it to stand for a few minutes.	Yellow precipitate is formed.	Ba^{2+} may be present.
	(ii) To the prepared sample solution, add $(NH_4)_2SO_4$ solution. Leave it to stand for a few minutes.	White precipitate is formed.	Sr^{2+} may be present.

S/n	Experiment	Observation	Inference
	(iii) To the prepared sample solution, add dilute H_2SO_4 until in excess, centrifuge or filter and discard any residue. To the supernatant add dilute ammonia solution to neutralize excess acid followed by ammonium oxalate solution.	White precipitate is formed.	Ca^{2+} may be present.
	Confirmatory Tests for Ba^{2+} (i) To the solution of the sample add potassium chromate(VI) solution.	Yellow precipitate is formed.	Ba^{2+} confirmed.
	(ii) To the solution of the sample add dilute NaOH solution followed by ammonium oxalate solution, and then add ethanoic acid.	White precipitate soluble in ethanoic acid is formed.	Ba^{2+} confirmed.
	(iii) To the solution of the sample add Na_3PO_4 solution followed by dilute HCl or HNO_3.	White precipitate soluble in dilute HCl or HNO_3.	Ba^{2+} confirmed.
	(iv) Perform flame test.	Green flame.	Ba^{2+} confirmed
	Confirmatory Tests for Sr^{2+} (i) To the solution of the sample add $(NH_4)_2SO_4$ solution or dilute H_2SO_4.	White precipitate is formed.	Sr^{2+} confirmed.
	(ii) Perform flame test.	Red flame.	Sr^{2+} confirmed.
	Confirmatory Tests for Ca^{2+} (i) To the solution of the sample add excess ammonia solution followed by ammonium oxalate solution.	White precipitate is formed.	Ca^{2+} confirmed.
	(ii) Perform flame test.	Brick red flame.	Ca^{2+} confirmed.
6.	**GROUP VI** To the solution of the sample, add aqueous ammonium oxalate. Filter and discard any precipitate. **Confirmatory Tests for Mg^{2+}** (i) To the prepared sample solution, add dilute ammonia	White crystalline precipitate is formed.	Mg^{2+} confirmed.

S/n	Experiment	Observation	Inference
	solution, solid ammonium chloride and disodium hydrogen phosphate.		
	(ii) To the prepared sample solution add few drops of magneson I reagent followed by excess NaOH solution.	Sky-blue precipitate is formed.	Mg^{2+} confirmed.
	Confirmatory Tests for K^+ and Na^+ (i) Evaporate the supernatant to dryness and use the residue formed to perform flame test.	Lilac/light purple flame.	K^+ confirmed.
		Golden-yellow flame.	Na^+ confirmed.
	(ii) To the solution of the sample, add sodium hexanitritocobaltate(III) solution, followed by ethanoic acid.	Yellow precipitate is formed.	K^+ confirmed.
	Confirmatory Test for NH_4^+ To a small amount of a solid sample add dilute NaOH and warm. Pass moist litmus paper to the mouth of the test-tube containing the mixture. Dip a glass rod in concentrated HCl and pass it to the mouth of a test-tube containing the mixture.	Colourless gas which turns moist red litmus paper blue and forms white fumes with concentrated HCl evolves.	NH_4^+ confirmed.

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Table 2: Preliminary Tests

S/n	Experiment	Observations	Inference
1.	**Appearance of the Sample**		
	(i) Colour	White	Non-transition metals may be present.
		Blue	Cu^{2+}, Co^{2+} may be present.
		Green	Fe^{2+}, Ni^{2+}, Cr^{3+}, Cu^{2+} may be present.
		Yellow	Fe^{3+}, CrO_4^{2-} may be present.
		Brown/Yellowish-brown	Fe^{3+} may be present.
		Pink	Co^{2+}, Mn^{2+} may be present.
		Orange	$Cr_2O_7^{2-}$ may be present.
		Crystalline form	NO_3^-, SO_4^{2-}, Cl^-, $C_2O_4^{2-}$, CrO_4^{2-}, NO_2^-, CH_3COO^-, $Cr_2O_7^{2-}$ may be present.
			CO_3^{2-} and HCO_3^- may be present except CO_3^{2-} of NH_4^+, K^+ and Na^+
			NH_4^+ may be present.
			...$^{2-}$ may be present.

Table 3: Confirmatory Tests for Anions using Sodium Carbonate Extract of Salt

S/n	Experiment	Observations	Inference
1.	**Confirmatory Tests for SO_4^{2-}** (a) Transfer a small volume (about 1 cm^3) of the extract into the test-tube. Add barium chloride solution followed by dilute HCl or barium nitrate solution followed by dilute HNO_3.	White precipitate is formed insoluble in dilute HCl or HNO_3^-.	SO_4^{2-} confirmed.
	(... small volume of the ... test-tube. Add ... by lead ...	White precipitate insoluble in dilute HCl but soluble in ammonium ...ate solution is	SO_4^{2-} confirmed.

Table 4: The Action of NaOH(aq) and NH_4OH(aq) on Sample Solutions

Addition of 1 or 2 drops of NaOH(aq) or NH_4OH(aq)	Addition of Excess NaOH(aq)	Addition of Excess NH_4OH(aq)	Inference
White precipitate.	Precipitate dissolves.	Precipitate dissolves.	Zn^{2+} may be present.
White precipitate.	Precipitate dissolves.	Insoluble precipitate.	Sn^{2+}, Pb^{2+} may be present.
White precipitate.	Insoluble precipitate.	Insoluble precipitate.	Sb^{3+}, Bi^{3+}, Mg^{2+} may be present.
White precipitate.	Precipitate dissolves.	Slightly soluble precipitate.	Al^{3+} may be present.
White precipitate.	Insoluble precipitate.	No precipitate.	Ca^{2+}, Sr^{2+}, Ba^{2+} may be present.
No precipitate.	No precipitate.	No precipitate.	K^+, Na^+ may be present.
No precipitate.	Ammonia gas evolves on warming.	No precipitate.	NH_4^+ may be present.
Brown precipitate.	Insoluble precipitate.	Insoluble precipitate.	Ag^+ may be present.