THE NATIONAL EXAMINATIONS COUNCIL OF TANZANIA

QUALITATIVE ANALYSIS GUIDE FOR THE CERTIFICATE OF SECONDARY EDUCATION EXAMINATION

032 CHEMISTRY

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CONTENTS

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PREFACE

During the implementation of National Examinations in Chemistry practicals, the National Examinations Council of Tanzania (NECTA) observed that, candidates from different centres have been using varieties of Qualitative Analysis Guides (QAG) to identify ions in the given unknown compounds. Some of the guides were not approved by the Ministry of Education, Science and Technology. Further follow up by the NECTA revealed that, some guidelines had incorrect chemical symbols and formulae, inconsistent information and typographic errors. It was also revealed that, those guides were used by chemistry teachers in teaching practical lessons in schools, hence inconsistency in delivering the subject content to students. The National Examinations Council has therefore prepared this Qualitative Analysis Guide booklet to address those challenges. The objective of constructing this document is to have a identical guide which will be used by candidates when writing their chemistry practical examinations at ordinary level secondary education.

This guide is based on 2010 ordinary level secondary education chemistry syllabus. The first edition of 2016 was planned to be used from 2018 in Certificate of Secondary Education Examination (CSEE). However, recommendations and improvements from stakeholders prompted the NECTA to revise the first edition. In addition to the updating the first edition, the revised QAG contains essential materials which were found to be useful. This edition will be effectively used from 2018 in CSEE.

It is hoped that, this booklet will give an extra guidance to teachers and students in conducting analytical experiments during teaching and learning processes. Teachers and prospective candidates are therefore encouraged to use this booklet effectively so that they can be conversant with the procedures indicated and finally be in a good position of using it properly when doing and writing their examinations.

Dr. Charles E. Msonde
EXECUTIVE SECRETARY

INTRODUCTION

The National Examinations Council of Tanzania (NECTA) has prepared Qualitative Analysis Guide (QAG) to support candidates in identification of ions that are present in unknown compounds through several chemical tests. For Certificate of Secondary Education Examinations (CSEE), 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 Secondary Schools.

Cations: NH_4^+, Na^+, Ca^{2+}, Pb^{2+}, Fe^{2+}, Fe^{3+}, Zn^{2+} and Cu^{2+}

Anions: NO_3^-, SO_4^{2-}, Cl^-, CO_3^{2-} and HCO_3^-

The content of this guide is divided into four sections: A, B, C and D. Section A is a brief note on how to record analytical experiments. Section B is on preliminary tests while Section C is on tests of ions in solution. Section D is on confirmatory tests for ions. Finally, the guide ends up with bibliography.

SECTION A: RECORDING ANALYTICAL EXPERIMENTS

The candidates are required to record experiments performed, observations and results/inferences 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 appearance of the sample and changes which have been observed or identified when a chemical substance is subjected to a test are written in the "Observation" column. These observations include: colour, texture, formation of precipitates, evolution of gases, flame colours, sound and others.

The deductions or what can be inferred from the observation is written in the "Inference" column. These inferences are the ones which lead to the systematic 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. If no reaction occurs, it implies the absence of sulphate ion. The formation of white precipitate indicates the presence of sulphate ion in the unknown sample.

After performing all experiments, it is required to make conclusion about ions present in the sample by performing the confirmatory tests for every deduced ion in the preliminary tests. This can be obtained by combining all the inferences made in the successive tests.

SECTION B: PRELIMINARY TESTS

The preliminary tests are generally for solid samples. As shown in Table 2, the tests include appearance (colour, texture and deliquescence), odour, flame test, action of heat, solubility in water and action of dilute and concentrated acids.

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

Table 2: Preliminary Tests

Experiment	Observations	Inference
1. Appearance of Solid Sample (i) Colour	White	NH_4^+, Na^+, Ca^{2+}, Zn^{2+}, Pb^{2+} may be present. or Transition metals Fe^{2+}, Fe^{3+}, Cu^{2+} may be absent.
	Blue or green.	Cu^{2+} may be present.
	Pale green (light green)	Fe^{2+} may be present.
	Yellowish-brown	Fe^{3+} may be present.
(ii) Texture	Crystalline form	NO_3^-, SO_4^{2-}, Cl^- may be present.
	Powder form	CO_3^{2-}, HCO_3^- may be present.
(iii) Odour	Choking smell	NH_4^+ may be present.
(iv) Deliquescence	Absorbs water from the atmosphere to form a 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 sample followed by heating it on a flame.	Golden yellow flame	Na^+ may be present.
	Brick red flame	Ca^{2+} may be present.
	Bluish-green flame	Cu^{2+} may be present.
	Blue-white (pale-blue) flame	Pb^{2+} may be present.
	Yellow (orange) sparks	Fe^{2+}, Fe^{3+} may be present.
	No definite flame colour observed	Zn^{2+}, NH_4^+ may be present.

Experiment	Observations	Inference
3. Action of Heat on a Solid Sample [***Safety Precautions***: *Hold the test-tube in a slanting position and away from observers and neighbours*] Transfer a small amount (about 0.5 g) of the solid sample in a dry test-tube. Heat gently and then strongly until no further change. Test for any gas evolved and observe the appearance of the residues.	White sublimate and a colourless gas evolves, which turns moist litmus paper from red to blue.	NH_4^+ may be present.
	Reddish brown fumes evolve which turn moist blue litmus paper red and a gas which rekindles a glowing wooden splint.	NO_3^- may be present.
	Colourless gas evolves, which relights a glowing splint.	NO_3^- of Na^+ may be present.
	Colourless gas evolves, which turns lime water milky and moist litmus paper from blue to red.	CO_3^{2-}, HCO_3^- may be present.
	Colourless gas with pungent smell evolves, which turns moist blue litmus paper red or filter paper dipped in acidified potassium dichromate solution from yellow to green.	SO_4^{2-} may be present.
	Colourless gas evolves, which gives dense white fumes with ammonia gas.	Cl^- of hydrated Ca^{2+}, Zn^{2+}, Cu^{2+}, Fe^{2+}, Fe^{3+} salts may be present.
	No gas evolves.	SO_4^{2-} of Na^+, Ca^{2+}, Pb^{2+} may be present.
		Cl^- of Na^+, Pb^{2+} may be present.
		CO_3^{2-} of Na^+ may be present.
	Colourless droplets forming on the cooler parts of the test-tube, which turn anhydrous $CuSO_4$ blue or $CoCl_2$ pink.	Hydrated salt, HCO_3^- may be present.

Experiment	Observations	Inference
	Cracking sound with brown gas.	NO_3^- of Pb^{2+} may be present.
	Cracking sound with no gas evolving.	Cl^- of Na^+ may be present.
	Residue yellow when hot and white when cold.	Zn^{2+} may be present.
	Residue reddish brown when hot and yellow when cold.	Pb^{2+} may be present.
	Black residue.	Cu^{2+} may be present.
	Reddish brown residue.	Fe^{2+}, Fe^{3+} may be present.
	White residue.	Ca^{2+}, Na^+ may be present.
	Blue crystals turn white	SO_4^{2-} of hydrated Cu^{2+} may be present.
4. Action of Dilute HCl on a Solid Sample Transfer a small amount of solid sample in a test-tube followed by 3 drops of dilute HCl.	Effervescence of a colourless gas evolves, which turns lime water milky and moist litmus paper from blue to red.	CO_3^{2-}, HCO_3^- may be present.
	No gas evolves	SO_4^{2-}, NO_3^-, Cl^- may be present.
	White precipitate	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 (c) Hold the test-tube in a slanting position and away from observers and neighbours*]. 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.	Effervescence of a colourless gas evolves. The gas turns lime water milky and moist litmus paper from blue to red.	CO_3^{2-}, HCO_3^- may be present.
	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.

Experiment	Observations	Inference
	Blue crystals which turn white even without warming.	SO_4^{2-} of hydrated Cu^{2+} may be present.
	Brown fumes evolve, which turn moist blue litmus paper red and intensify on addition of copper turnings.	NO_3^- may be present.
	No gas evolves.	SO_4^{2-} may be present.
6. Solubility of Solid Samples Transfer a small amount of the solid sample into the test-tube and add enough cold distilled water to dissolve the solid sample. If the sample does not dissolve warm the contents.	Soluble forming a colourless solution.	Na^+, NH_4^+, NO_3^- may be present.
		CO_3^{2-}, HCO_3^- of Na^+, NH_4^+ may be present.
		Cl^- of Zn^{2+} or Ca^{2+}. may be present.
		SO_4^{2-} of Zn^{2+} may be present.
	Soluble forming blue or green solution.	Cu^{2+} may be present.
	Soluble forming pale green solution.	Fe^{2+} may be present.
	Soluble forming yellowish-brown solution.	Fe^{3+} may be present.
	Insoluble in cold water but soluble in hot water. Crystals reappear on cooling.	Cl^- of Pb^{2+} may be present.
	Insoluble.	CO_3^{2-} of Ca^{2+}, Pb^{2+}, Zn^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+} may be present.
		SO_4^{2-} of Ca^{2+}, Pb^{2+} may be present.

SECTION C: TESTS IN SOLUTION

Preparation of the Stock Solution of the Sample

Transfer a small amount (about 1 g) of the solid sample in a test-tube. Add enough amount of distilled water (about 15-20 cm^3) and shake thoroughly. If the sample is insoluble in cold water, warm the contents. If the sample is insoluble in hot water, transfer (about 1 g) of the new solid sample in a test-tube and then dissolve it in dilute nitric acid (to about 15-20 cm^3 of the final solution). Perform the tests as shown in Table 3.

Table 3: Tests in Solution

Experiment	Observations	Inference
1. Action of NaOH Solution on a Sample Solution To a small volume (about 1 cm^3) of the original sample solution, add sodium hydroxide solution drop-wise until in excess.	White precipitate is formed, soluble in excess.	Zn^{2+}, Pb^{2+} may be present.
	White precipitate is formed, insoluble in excess.	Ca^{2+} may be present.
	Blue precipitate is formed, insoluble in excess.	Cu^{2+} may be present.
	Green precipitate is formed, insoluble in excess, which turns brown on standing.	Fe^{2+} may be present.
	Reddish-brown precipitate is formed, which is insoluble in excess.	Fe^{3+} may be present.
	No precipitate is formed; on warming, a colourless gas with a choking smell which turns moist litmus paper from red to blue evolves.	NH_4^+ may be present.
2. Action of NH_3 Solution on a Sample Solution To a small volume of the original sample solution, add ammonia solution drop-wise until in excess.	White precipitate is formed, insoluble in excess.	Pb^{2+} may be present.
	White gelatinous precipitate is formed, soluble in excess.	Zn^{2+} may be present.
	No precipitate is formed.	Ca^{2+}, Na^+ may be present.

Experiment	Observations	Inference
	Pale blue precipitate is formed, soluble in excess forming a deep blue solution.	Cu^{2+} may be present.
	Green precipitate is formed, insoluble in excess.	Fe^{2+} may be present.
	Reddish-brown precipitate is formed, insoluble in excess.	Fe^{3+} may be present.

SECTION D: CONFIRMATORY TESTS

Table 4: Confirmatory Tests for Cations

Experiment	Observations	Inference
1. Confirmatory Tests for Ca^{2+} (i) To a small volume of the original sample solution, 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.
2. Confirmatory Tests for Pb^{2+} (i) To a small volume of the sample solution, add K_2CrO_4 solution.	Yellow precipitate is formed.	Pb^{2+} confirmed.
(ii) To a small volume of the sample solution, add KI solution. Warm and cool the mixture.	Yellow precipitate which disappears on warming but re-appears on cooling.	Pb^{2+} confirmed.
3. Confirmatory Tests for Zn^{2+} (i) To a small volume of the sample solution, add potassium hexacyanoferrate(II) solution followed by few drops of dilute HCl.	Bluish-white precipitate insoluble in dilute HCl.	Zn^{2+} confirmed.
(ii) To a small volume of the sample solution, add ammonia solution until in excess.	White gelatinous precipitate soluble in excess.	Zn^{2+} confirmed.

Experiment	Observations	Inference
4. Confirmatory Test for NH_4^+ Transfer a small amount (about 0.2 g) of the original solid sample in a test-tube, add sodium hydroxide solution just to cover the whole solid then warm gently. Test for gas evolved.	Colourless gas evolves which turns moist litmus paper from red to blue.	NH_4^+ confirmed.
5. Confirmatory Test for Na^+ Perform flame test.	Golden yellow flame.	Na^+ confirmed.
6. Confirmatory Tests for Cu^{2+} (i) To a small volume of the original sample solution, add ammonia solution drop-wise until in excess.	Pale blue precipitate soluble in excess of aqueous ammonia forming a deep blue solution.	Cu^{2+} confirmed.
(ii) To a small volume of the original sample solution, add few drops of potassium hexacyanoferrate(II).	Reddish-brown precipitate.	Cu^{2+} confirmed.
7. Confirmatory Tests for Fe^{2+}, Fe^{3+} (i) To a small volume of the sample solution, add few drops of potassium hexacyanoferrate(III).	Deep blue precipitate.	Fe^{2+} confirmed.
(ii) To a small volume of the sample solution, add few drops of potassium hexacyanoferrate(II).	Light blue precipitate.	Fe^{2+} confirmed.
(iii) To a small volume of the sample solution, add few drops of potassium hexacyanoferrate(II).	Deep blue precipitate.	Fe^{3+} confirmed.
(iv) To a small volume of the sample solution, add few drops of potassium or ammonium thiocyanate solution.	Deep blood- red solution.	Fe^{3+} confirmed.

Table 5: Confirmatory Tests for Anions

Experiment	Observations	Inference
1. Confirmatory Tests for SO_4^{2-} (i) Transfer a small volume of the original sample solution into the test-tube. Add barium chloride followed by dilute HCl or barium nitrate followed by dilute HNO_3.	White precipitate insoluble in dilute HCl or dilute HNO_3.	SO_4^{2-} confirmed.
(ii) Transfer a small volume of the original sample solution into the test-tube. Add ethanoic acid followed by lead ethanoate solution. 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.	SO_4^{2-} confirmed.
2. Confirmatory Tests for NO_3^- (i) Transfer a small volume of the original solid sample solution into the test-tube. Add dilute H_2SO_4 and 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.
(ii) Transfer a small amount of the original solid sample into a test-tube. Add copper turnings followed by concentrated H_2SO_4 then warm.	Brown fumes evolve.	NO_3^- confirmed.
3. Confirmatory Tests for CO_3^{2-}, HCO_3^- (i) Transfer a small volume of the original sample solution 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.

Experiment	Observations	Inference
(ii) Transfer a small volume of the original sample solution into a test-tube. Add $BaCl_2$ solution. If the precipitate forms, add dilute HCl.	White precipitate soluble in dilute HCl is formed.	CO_3^{2-} confirmed.
(iii) Transfer a small amount of water-insoluble solid sample in a test-tube. Add a small volume of dilute nitric acid.	Effervescence of a colourless gas, which turns lime water milky.	CO_3^{2-} confirmed.
4. Confirmatory Tests for Cl^- (vi) To a small volume of the original sample solution, add about 3 drops of dilute nitric acid followed by about 3 drops of silver nitrate solution and then add excess ammonia solution.	White precipitate soluble in excess ammonia solution is formed.	Cl^- confirmed.
(vii) Transfer a small amount of the original solid sample into a test-tube. Add a small amount of MnO_2 followed by concentrated H_2SO_4 and warm the mixture.	Greenish-yellow gas evolves which bleaches moist red litmus paper.	Cl^- confirmed.

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Table 2: Preliminary Tests

Experiment	Observations	Inference
1. **Appearance of solid Sample** (i) Colour	White	NH_4^+, Na^+, Ca^{2+}, Zn^{2+}, Pb^{2+} may be present. or Transition metals Fe^{2+}, Fe^{3+}, Cu^{2+} may be absent.
	Blue or green.	Cu^{2+} may be present.
	Pale green (light green)	Fe^{2+} may be present.
	... brown	Fe^{3+} may be present.
(ii) Texture		NO_3^-, SO_4^{2-}, Cl^- may be present.
		... may be present.
(iii) Odour		
(iv) Deliquescen...		

Table 3: Tests in Solution

Experiment	Observations	Inference
1. **Action of NaOH Solution on a Sample Solution** To a small volume (about 1 cm³) of the original sample solution, add sodium hydroxide solution drop-wise until in excess.	White precipitate is formed, soluble in excess.	Zn^{2+}, Pb^{2+} may be present.
	White precipitate is formed, insoluble in excess.	Ca^{2+} may be present.
	Blue precipitate is formed, insoluble in excess.	Cu^{2+} may b...
	Green precipitate is formed, insoluble in excess, wh... turns brown on standi...	
	Reddish-bro... formed ... exc...	

Table 4: Confirmatory Tests for Cations

Experiment	Observations	Inference
1. **Confirmatory Tests for Ca^{2+}** (i) To a small volume of the original sample solution, 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.
2. **Confirmatory Tests for Pb^{2+}** (i) To a small volume of the sample solution, add K_2CrO_4 solution.	Yellow precipitate is formed.	Pb^{2+} confirmed.
(ii) To a small volume of the sample solution, add KI solution. Warm and cool the mixture.	Yellow precipitate which disappears on warming but re-appears on cooling.	Pb^{2+} confirmed.