THE UNITED REPUBLIC OF TANZANIA

MINISTRY OF EDUCATION, SCIENCE AND TECHNOLOGY

NATIONAL EXAMINATIONS COUNCIL OF TANZANIA

GUIDELINES FOR PREPARING THE LABORATORY FOR PRACTICAL EXAMINATIONS AT SECONDARY AND DIPLOMA IN SECONDARY EDUCATION LEVELS

CHEMISTRY

Prepared by:
The National Examinations Council of Tanzania,
P.O. Box 2624,
Dar es Salaam, Tanzania.

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FOREWORD

The National Examinations Council of Tanzania (NECTA) administers practical examinations in the Certificate of Secondary Education Examination (CSEE), Advanced Certificate of Secondary Education Examination (ACSEE) and Diploma in Secondary Education Examination (DSEE). The examinations aim at assessing knowledge, skills and competencies that have been acquired by candidates in testing facts practically under controlled experimentation and observations. Through practical examinations, skills and competencies of students in the subject are effectively evaluated.

These guidelines are designed to assist chemistry teachers, tutors and laboratory technicians who handle practical preparations in the laboratory to perform their tasks effectively and efficiently. The guidelines provide important instructions on how to organize the laboratory in general and for examinations in particular. They offer a step by step guide on how teachers should prepare and administer practical examinations.

The guidelines can also be used as a tool for promoting teaching and learning process. They will equip those who handle laboratory preparation with skills and procedures for conducting different Chemistry practicals. The guidelines are based on areas for practical assessments stipulated in the subject syllabi. They identify assessment areas, objectives of conducting different practicals, materials, apparatuses, equipment and chemicals to be used in the practicals, procedures for conducting such practicals and sample questions for the practicals. These aspects have been intentionally included to ensure a smooth and fair conduct of the examinations and to ensure the uniformity across all examination centres.

The Examinations Council expects that teachers, tutors and laboratory technicians will use the guidelines to improve their practice in the preparations and conduct of practical examinations.

The Council would like to appreciate valuable contributions from all who participated in the preparation of this guideline.

Dr. Charles E. Msonde
EXECUTIVE SECRETARY

1.0 INTRODUCTION

These guidelines provide instructions for preparing practical examinations administered by the National Examinations Council of Tanzania (NECTA). They cover practical examinations in the Certificate of Secondary Education Examination (CSEE), Diploma in Secondary Education Examination (DSEE) and Advanced Certificate of Secondary Education Examination (ACSEE). The guidelines are aimed to assist chemistry teachers, tutors and laboratory technicians to prepare laboratory for practical examinations within a specified time by properly attending to the requirements stipulated in examinations checklists and advanced practical instructions before practical examination sessions commence.

The guidelines comprise instructions on the laboratory organisation, management and safety and content areas of practical assessment as stipulated in their respective syllabuses. In each area of assessment, the guideline covers key issues in preparing solutions and apparatuses, a sample question for reflection and assessment procedures. However, this guideline is not a substitute for the content stipulated in the syllabus.

It is believed that these practical guidelines will help teachers in identifying key issues to take into account in assessing competencies attained by candidates in practicals.

2.0 LABORATORY ORGANISATION, MANAGEMENT AND SAFETY

The practical examination is successfully prepared and conducted if a laboratory is well organised and managed and if safety procedures are observed. A well-organized and managed laboratory reduces the cost and time of preparing practical examinations through a proper use of ledger books. A proper keeping of laboratory records enables schools and colleges to identify apparatuses and chemicals to be bought when attending to examinations' checklists.

2.1 Laboratory Organization

Laboratory organization refers to the arrangement of apparatuses, chemicals and equipment in a good order. If these items are arranged properly, they can be located easily when needed. This reduces time for the preparation. Moreover, in a well-organized laboratory experiences minimum or no accident during the preparation and execution of practical examinations. The aim of this section is to enable teachers, tutors and laboratory technicians to:

(a) label chemicals, apparatuses and equipment properly, and
(b) store chemicals, equipment and apparatuses appropriately.

2.1.1 Labelling of Chemicals and Apparatuses

A proper labelling of chemicals and apparatuses is essential in organizing a laboratory and to optimize its use. If chemicals and apparatuses are labelled properly, they can be located easily. During this process, the following should be done:

(a) label lockers, drawers and shelves to indicate contents in them;
(b) label bench reagents by indicating their concentration and the date of preparation, and

(c) label chemicals and apparatuses using masking tapes or adhesive tapes while attending to advanced practical instructions. For chemicals stored for a long time, use the plain paper and secure them with a cello tape.

2.1.2 The Storage of Chemicals, Equipment and Apparatuses

A proper storage of chemicals and apparatuses ensures safety and easy access of reagents/chemicals and equipment by laboratory users. For a proper storage, adhere to the following procedures:

(a) arrange chemicals in the store/strong room and in cupboards alphabetically. Arrange them in such a way that chemicals that are flammable, explosive, oxidizing such as HNO_3, $KMnO_4$ and peroxides are kept separate from chemicals they can react with;

(b) store concentrated corrosive chemicals such as H_2SO_4, CH_3COOH, HCl and HNO_3 on the floor or in lower shelves of the cupboard;

(c) arrange apparatuses according to their use. For instance, apparatuses for measuring volume should be kept together, and

(d) separate metallic apparatuses from glassware.

2.2 Laboratory Management

Laboratory management is concerned with ensuring that laboratory activities run smoothly and efficiently. Teachers, tutors and laboratory technicians have to ensure that all facilities and materials necessary to prepare practical examinations are available in the laboratory prior to practical examinations. Laboratory management also involves following rules and regulations properly. Basically, the laboratory manager is obliged to perform the following before examination commence and during the examination:

(a) Management of the Laboratory Before the Commencement of the Examination

When attending a checklist, the teacher, the tutor and the laboratory technician should:

(i) ensure that all apparatuses and chemicals specified in the checklist are available in the laboratory;
(ii) ensure that all chemicals, apparatuses and equipment (bought and those reserved in the store according to records in the ledger book) are functional/calibrated and tested for their efficiency days before attending the advanced practical instructions;
(iii) fill in and update the laboratory ledger book appropriately. Keep records of how much reagent has been used for each experiment and how much is remaining. This will inform the planning of the procurement of apparatuses and chemicals required for practical examinations;
(iv) make use of stock record book(s) in which all available reagents, chemicals and apparatuses are listed for easy picking and use;
(v) ensure that all apparatuses and equipment are clean;
(vi) ensure that there are enough masking tapes for labelling;

(vii) make sure that heat sources such as kerosene burners, Bunsen burners, water baths and other common laboratory fittings are available and functioning. Likewise, ensure that there is no leakage of gases. If any leakage is observed, repair or replace the source immediately;

(viii) test and repair or replace apparatuses such as burette, rubber tubes and loose clips that are non-functional;

(ix) observe safety measures in the laboratory while performing the trial test or the calibration of apparatuses, and

(x) note that organic solvents such as diethyl ether, ethyl acetate and isobutanol can be recovered from previous experiments through fractional distillation. However, applicable only in schools or colleges with advanced equipment for performing the fractional distillation.

(b) Management of the Laboratory During the Preparation of the Practical Examination according to Advanced Practical Instructions

When preparing the practical examination, adhere to the following:

(i) observe safety measures in the laboratory during practical session(s). This should be done in collaboration with the supervisor;

(ii) prepare enough solutions as instructed in the advance instruction. Nonetheless, remember to use chemicals economically, and

(iii) give candidates the hard glasses like pyrex for heating substances because such glasses do not break easily on heating.

2.3 The Safety in the Laboratory

The safety in the laboratory is of utmost importance during practical examinations. Safety must be regarded as the foundation of effective activities in the laboratory. In collaboration with the supervisor and invigilators during the preparation and the administering practical examination, ensure the following:

(i) the procedures are followed for preparing various chemicals correctly, e.g., **always add acid to water and not water to acid**

(ii) the floor is always dry to avoid falling or sliding during practical sessions;

(iii) glassware, heat sources, electrical appliances, gas taps and chemicals are used appropriately during practical examinations;

(iv) candidates use pipette fillers to suck chemicals while using pipettes. Direct sucking of chemicals using the mouth should be discouraged;

(v) candidates use tongs and other holders when touching hot surfaces, water baths and boiling tubes or beakers, instead of using bare hands;

(vi) protective gears like eye goggles, gloves and laboratory coats or aprons are worn during the preparation of practical examinations;

(vii) broken glassware are not be used to avoid the injury during the washing; and

(viii) windows are kept wide open to allow enough ventilation in the laboratory.

3.0 CONTENT AREAS OF PRACTICAL EXAMINATIONS

Distribution of contents for practical examinations in each level is shown in Table 1.

Table 1: Content Areas of Practical Examinations

S/N	Content Area	Type of Practical/Main Teaching Area	Level		
			CSEE	ACSEE	DSEE
1.	Volumetric analysis	(i) Standardization of an acid/base	√	√	√
		(ii) Double indicator method • Determination of the volume of HCl used when Na_2CO_3 is titrated against HCl using POP and MO indicators • Determination of proportions in a mixture of compounds		√	
		(iii) Standardization of sodium thiosulphate using potassium permanganate		√	
		(iv) Standardization of sodium thiosulphate using potassium iodate		√	
		(v) Volumetric estimation of copper in copper(II) sulphate		√	
		(vi) Volumetric titrations	√		√
		(vii) Standard solutions	√		√
		(viii) Volumetric calculations	√		
2.	Chemical kinetics	(i) Factors affecting the rate of chemical reactions	√	√	√
		(ii) Reversible and irreversible reactions	√	√	
		(iii) Equilibrium reactions	√	√	
		(iv) Equilibrium constant		√	
		(v) Order of chemical reactions		√	√
		(vi) Endothermic and exothermic reactions	√	√	
3.	Qualitative analysis	(i) Identification of cation and anion in a single salt	√		√
		(ii) Identification of cation and anion in a single salt through group separation		√	
		(iii) Identification of cation and anion in a mixture of salts through group separation		√	

S/N	Content Area	Type of Practical/Main Teaching Area	Level		
			CSEE	ACSEE	DSEE
4.	Two component liquid systems	Distribution Law		√	

3.1 Volumetric Analysis

Volumetric analysis is a form of quantitative analysis which depends on accurate measurements of volumes of solutions. In a volumetric analysis, a known volume of a solution of an unknown concentration is used to react with an unknown volume of a solution of a known concentration. The common method of volumetric analysis is the titration.

Specific Objectives

The teacher, the tutor and the laboratory technician should be able to prepare experiments for candidates to:

(i) standardize an acid or base;
(ii) determine the volume of HCl used when Na_2CO_3 is titrated against HCl using POP and MO (double indicator method);
(iii) determine the proportions of Na_2CO_3 and NaOH in a mixture through volumetric analysis;
(iv) standardize sodium thiosulphate using potassium permanganate;
(v) standardize sodium thiosulphate using potassium iodate;
(vi) carry out a volumetric estimation of copper in copper(II) sulphate;
(vii) determine the relative atomic mass of an unknown element or compound; and
(viii) determine the percentage purity of an acid or base.

3.1.1 The Preparation and Standardization of Acid and Base Solution

The teacher, the tutor or the laboratory technician should be able to:

(a) interpret the information on chemical containers, and
(b) prepare solutions with desirable concentrations

3.1.1.1 The Preparation of Solutions

Solutions are prepared either from solid chemicals or concentrated stocks. The preparation of standard solutions is a key issue prior to the execution of the volumetric analysis practicals. It is, therefore, expected that after the completion of this sub-topic, teachers, tutors and laboratory technicians will be equipped with basic skills in the stated areas.

(a) The preparation of Base or Acid from their Solid Chemicals

To perform such preparations, you have to consider the molarity of the solution needed and the exact volume of the solution required by candidates. Thereafter, follow these steps:

(i) calculate the mass needed from the molar mass of the compound. Dissolve such amount in the required amount of distilled water to get the required concentration.

Use a formula,

$$\text{Grams of chemical} = \frac{\text{molarity of solution} \times \text{molar mass of chemical (g/mol)} \times \text{volume of the required solution (mL)}}{1000\,\text{mL/L}}$$

(ii) weigh the appropriate mass of solute and add it to a beaker containing a small amount of distilled water;

(iii) stir the solution to dissolve the contents;

(iv) Transfer the solution to a volumetric flask;

(v) add more distilled water up to the mark of the flask, and

(vi) cap and invert the flask several times to allow a thorough mixing of the solution.

Note

For direct method of weighing the substances, the chemical should be 100% pure. If the chemical is impure, its purity must be calculated. For example, if 10.6 g Na_2CO_3 is the weighed impure mass with percentage purity of 98.5% then, the actual weight of Na_2CO_3 in the sample is calculated by using this formula;

$$\text{actual weight} = \frac{\text{impure weight}}{\text{percentage purity}} \text{x}100$$

$$\text{actual weight} = \frac{10.6\,\text{g} \times 100}{98.5} = 10.76\ \text{g}$$

Example 1: The preparation of a primary standard solution 0.05 M Na_2CO_3
Chemicals: Na_2CO_3 and distilled water

Apparatuses: A Chemical or a digital balance, a 250 cm^3 beaker, a stirring rod, a volumetric flask, a desiccator, a filter funnel and a source of heat.

The Procedure

(i) Weigh 10.0 g of anhydrous sodium carbonate and put it in an evaporating dish. Heat it gently for about 10 minutes to ensure a complete removal of any trace of water. Allow it to cool in a desiccator.

(ii) Calculate the mass required which is equivalent to 0.05 M Na_2CO_3 with a formula: $m = M \times Mr$,

Where: m = mass in grams of a solid sample,
M = molarity of a solution to be prepared
Mr = Molar mass of the compound in question

Then mass,

$$m = 0.05 \times 106 = 5.3 \text{ g.}$$

(iii) Weigh a clean dry watch glass and record its mass.
(iv) Put 5.3 g of the cooled anhydrous sodium carbonate in the watch glass.
(v) Transfer the chemical into a 500 cm^3 beaker containing distilled water. Stir the mixture using a clean glass rod. Rinse the watch glass and the stirrer while adding the residues into the beaker to ensure that no mass is lost.
(vi) Dissolve all the solute in the beaker by thoroughly stirring the solution with a glass rod.
(vii) Use a funnel to transfer the solution into a 1000 cm^3 clean volumetric flask.
(viii) Add more distilled water in the volumetric flask up to approximately 1000 cm^3.
(ix) Using a dropper, top up the solution with distilled water up to the mark.
(x) Close the flask with a stopper and invert it several times to ensure a thorough mixing of the solution. This is a primary standard solution of 0.05 M Na_2CO_3.

Note

(i) Assemble all apparatuses and materials that you need before starting working.
(ii) Work in a clear space, preferably adjacent to a sink and water supply.
(iii) Wear goggles when dissolving, mixing and pouring chemicals.
(iv) Add concentrated acids, even small quantities, to water.
(v) Always use distilled water when preparing an aqueous solution.
(vi) Warm solution should be cooled to room temperature before being made up to the required volume. This is especially important when volumetric flasks are used given that these flasks are calibrated to contain their designated volumes at precisely 25°C.
(vii) Finely divided solids usually dissolve faster than large lumps. You will save time if you grind up large crystals with a pestle and mortar.
Heating is not necessary if the percentage of purity is considered during calculations.

(b) The Preparation of Solutions of Acids and Bases from their Concentrated Stock Solutions

Standard solutions of mineral acids or bases (especially ammonia solution) can be made by diluting their concentrated stock solutions. The molar concentrations of these stock solutions are not indicated on their stock bottles, but their densities and percentage purities are given. In order to prepare standard solutions of acids from their concentrated liquid, it is first required to understand the following:

(i) the molarity of the solution required, and
(ii) the exact volume of the solution needed

In this regard, the volume of the concentrated acid or base can be calculated and diluted in the required amount of distilled water to obtain the desired volume.

The molarity of the concentrated stock solution can be calculated from specifications given on Winchester bottle of the acid using the following formula:

$$\text{Molarity} = \frac{\%\,\text{assay} \times \rho \times 1000}{\text{Mr} \times 100}$$

Where;

% assay = percentage purity of a concentrated stock solution,

ρ = the density of a stock solution in g/cm^3

Mr = the molar mass of a solution

To obtain the volume of the concentrated stock solution that should be diluted to the required molarity in question, use the dilution law $M_cV_c = M_dV_d$.

Where;

M_c = molarity of the concentrated stock solution calculated from Winchester bottle specifications,

V_c = the volume of the stock solution which should be drawn from Winchester bottle in order to be diluted,

M_d = the required molarity of a solution after the dilution of the concentrated acid or base,

V_d = the volume of the required solution to be prepared after the dilution of concentrated acid or base

Use the following procedures to dilute stock solutions to obtain the required concentration:

(a) pour the concentrated solution from Winchester bottle to a beaker and measure the required volume using a measuring cylinder;

(b) transfer the measured volume into another beaker containing a small amount of water to dilute the acid; **Note: add acid to water and not water to acid**

(c) using a clean glass funnel, transfer the solution into a clean volumetric flask. Wash the beaker several times and add the washing to the flask;

(d) make sure that the flask and its contents are at ambient temperature. Carefully add water to the flask. Use a teat dropper to slowly add water until the bottom of the meniscus touches the calibration on the neck of the volumetric flask; and

(e) stopper the volumetric flask and shake it to ensure that the solution is thoroughly mixed.

Example 2: the preparation of 0.1 M HCl using specifications on Winchester bottle

Chemicals

Concentrated HCl (composition by mass 32.1%, density1.160 g/cm^3, Molar mass 36.5g/mol) and distilled water.

Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician

A 100 cm^3 measuring cylinder, a 250 cm^3 beaker, a stirring rod, a 500 cm^3 volumetric flask and a filter funnel.

The Procedure

(i) Use information given on Winchester bottle, to determine the molarity of HCl before the dilution as follows:

$$\text{molarity} = \frac{\%\ \text{assay} \times \rho \times 1000}{\text{Mr} \times 100}$$

$$\text{molarity} = \frac{32.1 \times 1.160\ \text{g/cm}^3 \times 1000\,\text{cm}^3/\text{L}}{36.5\,\text{g/mol} \times 100} = 10.20\ \text{M.}$$

The molarity of concentrated HCl = 10.20 M.

(ii) Calculate the volume that should be drawn from the concentrated HCl, to prepare 500 cm^3 of 0.1 M HCl solution.

The Procedure

Use the dilution law, $M_cV_c = M_dV_d$.

Where,

M_c = molarity of the HCl before the dilution,

M_d = molarity of the HCl required after the dilution (i.e. 0.1 M),

V_c = volume of HCl to be drawn from Winchester bottle before the dilution,

V_d = the resulting volume after the dilution (i.e. 500 cm^3).

$$V_c = \frac{M_d \times V_d}{M_c}$$

$$V_c = \frac{0.1\ \text{M}\ \text{´}\ 500\ \text{cm}^3}{10.20\ \text{M}} = 4.9\ \text{cm}^3$$

The volume of the concentrated HCl that should be diluted to 500 cm^3 = 4.9 cm^3.

(i) Using a measuring cylinder, measure the calculated volume of concentrated HCl from Winchester bottle (4.9 cm^3).

(ii) Transfer the volume into a beaker containing a small amount of water. **Note: add acid to water and not water to acid.**

(iii) Transfer the diluted acid into a 500 cm^3 volumetric flask by using of a funnel.

(iv) Rinse the beaker with distilled water and pour the contents into the volumetric flask.

(v) Top-up the volumetric flask to the mark by adding distilled water to it.

(vi) Close the volumetric flask and invert it repeatedly to mix the contents thoroughly.

Note: If after shaking and leaving it to settle the volume seems to drop, do not add water to top it up again to the mark.

(c) The Preparation of 0.1 M H_2O_2 Solution Using Specifications on Winchester bottle

The calculations here are based on the volume strength of H_2O_2 given on Winchester bottle as assay volume strength (% v/v),

For example, 100% v/v

From the decomposition equation of H_2O_2 to produce O_2,

From, $2H_2O_2 \rightarrow 2H_2O + O_2$

$2\ M\ H_2O_2 \quad \rightarrow \quad 1\ M\ O_2 = 22.4$ Litres

or $\quad 1\ M\ H_2O_2 \quad \rightarrow \quad ½M\ O_2 = 11.2$ Litres

$\quad ? \quad \rightarrow \quad 100$ Litres

$$\text{Molarity of stock solution} = \text{Volume strength} \times \frac{1}{11.2}$$

$$\text{Molarity of stock solution} = 100 \times \frac{1}{11.2} = 8.93\ \text{M}$$

For example, to prepare 1 litre of 0.1 M H_2O_2 solution,

Using the dilution law $M_cV_c = M_dV_d$

Where,

M_c = molarity of the H_2O_2 before the dilution,

M_d = molarity required after the dilution (i.e. 0.1 M),
V_c = volume of H_2O_2 to be drawn from winchester bottle before the dilution,
V_d = the resulting volume after the dilution (i.e. 1000 cm^3).

$$V_c = \frac{M_d \times V_d}{M_c}$$

$$V_c = \frac{0.1\text{M} \times 1000\ \text{cm}^3}{8.93\text{M}} = 11.198 \approx 11.2\ \text{cm}^3$$

The volume of the concentrated H_2O_2 that should be diluted to 1000 cm^3 = 11.2 cm^3.

Procedure

(i) Using the measuring cylinder, measure the calculated volume of the concentrated H_2O_2 from Winchester bottle (11.2 cm^3).

(ii) Transfer the measured volume into a beaker containing small amount of water.

(iii) Transfer the diluted acid into a 1000 cm^3 volumetric flask by using of a funnel.

(iv) Rinse the beaker with distilled water and pour the contents into the volumetric flask.

(v) Top-up the volumetric flask to the mark by adding distilled water.

(vi) Close the volumetric flask and invert it repeatedly to mix the contents thoroughly.
The suggested volumes of H_2O_2 are summarized in Table 2.

Table 2: The Suggested volumes of H_2O_2 for the preparation of 1 L of 0.1 M H_2O_2 solution

S.No.	Volume strength (%v/v)	Molarity of stock solution (M)	Volume (cm^3) of stock solution to be diluted to 1 L.
1.	20	1.79	55.9
2.	28	2.5	40
3.	30	2.68	37.3
4.	100	8.93	11.2

The Preparation of 0.1 M of Ammonia Solution Using Specifications on Winchester Bottle

Chemicals

Concentrated NH_3 (composition by mass 25%, density 0.91 g/cm^3, molar mass 17 g/mol) and distilled water.

Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician

A 100 cm^3 measuring cylinder, a 250 cm^3 beaker, a stirring rod, a 1000 cm^3 volumetric flask and a filter funnel

The Procedure

(i) use information given on Winchester bottle to determine the molarity of NH_3 before the dilution as follows:

$$\text{molarity} = \frac{\%\ \text{assay} \times \text{density} \times 1000}{\text{Molar mass} \times 100}$$

$$\text{molarity} = \frac{25 \times 0.91\ \text{g/cm}^3 \times 1000\ \text{cm}^3/\text{L}}{17\ \text{g/mol} \times 100}$$

$$= 13.38\ \text{M}$$

The molarity of the concentrated NH_3 solution is 13.38 M.

(ii) calculate the volume that should be drawn from the concentrated NH_3 to prepare 1000 cm^3 of 0.1 M NH_3 solution;

Using the dilution law; $M_cV_c = M_dV_d$

$$V_c = \frac{M_d \times V_d}{M_c}$$

$= 7.48 \approx 7.5\ cm^3$

The volume of the concentrated NH_3 that should be diluted to 1000 cm^3 is 7.5 cm^3;

(iii) using a measuring cylinder, measure the calculated volume of the concentrated NH_3 from Winchester bottle (i.e. 7.5 cm^3);

(iv) transfer the measured volume into a beaker containing a small amount of distilled water and stir it well to mix;

(v) transfer the diluted NH_3 solution into a 1000 cm^3 volumetric flask by means of a funnel;

(vi) rinse the beaker with distilled water and pour the contents into the volumetric flask;

(vii) top up the volumetric flask to the mark by adding more distilled water; and

(viii) close the volumetric flask and invert it repeatedly to mix the contents thoroughly.

Note

If NH_4OH is available instead of NH_3 solution, the same formula used in the preceding paragraph is used for calculating the molarity of the concentrated NH_4OH stock solution. However, the molar mass of 35 g/mol is used instead of 17 g/mol as follows;

$$\text{molarity} = \frac{\%\ \text{assay} \times \text{density} \times 1000}{\text{Molar mass} \times 100}$$

$$\text{molarity} = \frac{25 \times 0.91\ \text{g/cm}^3 \times 1000\ \text{cm}^3/\text{L}}{35\ \text{g/mol} \times 100} = 6.5\ \text{M}$$

The molarity of 25% concentrated NH_4OH stock solution is 6.5 M.

Then use the dilution law to calculate the volume of the concentrated NH_4OH stock

$M_cV_c = M_dV_d$

$$V_c = \frac{M_d \times V_d}{M_c}$$

$$V_c = \frac{0.1\text{M} \times 1000\ \text{cm}^3}{6.5\ \text{M}} = 15.4\ \text{cm}^3$$

The volume of the concentrated NH_4OH that should be diluted to 1000 cm^3 is 15.4 cm^3.

3.1.1.2 The Standardization of Solutions

Solutions can be prepared either by direct or indirect method.

(a) Direct Method

Under this method, a solution is made from a solute which is primarily a standard reagent. A primary standard reagent is a reagent which produces accurately known concentration of solution as per weight of the reagent in a solution.

(b) Indirect Method

Under this method, a solution is made from a reagent which is not primarily a standard reagent, but by standardizing it using a solution of a primary standard reagent.

Primary standard reagents include:

- anhydrous sodium carbonate (Na_2CO_3);
- ethanedioic acid (oxalic acid) ($C_2H_2O_4$);
- sodium hydrogen carbonate ($NaHCO_3$); and
- potassium dichromate ($K_2Cr_2O_7$).

Solutions of non-primary standard reagents are often not standard. Therefore, they must be standardized after the preparation so that their concentration is accurately known.

The Relative Standardization of Solutions

The standardization is done through volumetric analysis; whereby a solution of a primary standard reagent is titrated against a solution of non-primary standard reagent. The determinant of the standardization is the mole ratio, using the chemical equation of the reacting species.

The Standardization of a Solution which is more Concentrated than Expected

Consider a prepared solution with a 0.1 M NaOH which require to be titrated against a prepared solution of HCl which is more concentrated. It is expected that a student will pipette 25.0 cm^3 of NaOH and titrate it against HCl.

Suppose you intend to prepare 5.00 L of 0.1 M HCl, it is expected that the end point will occur when 25.0 cm^3 HCl is used up. However, that is not the case given that you will have prepared the HCl solution from which 20 cm^3 was used up to reach the end point, instead of 25 cm^3. Therefore, the molarity of the acid should be calculated as follows:

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$
Moles of NaOH = Moles of HCl (since its mole ratio is 1:1)
From, $M_aV_a = M_bV_b$
Where,
M_b = the molarity of base,
V_b = volume of base
M_a = molarity of acid
V_a = volume of acid
$M_a \times 20\ cm^3 = 0.1\ M \times 25.0\ cm^3$
$M_a = 0.125\ M$
This concentration is more than expected. To standardize such a solution, we need to add some distilled water to it.

From,
$M_1V_1 = M_2V_2$

Where,
M_2 = required molarity of solution (known)
V_1 = volume of the concentrated solution required to be diluted
M_1 = calculated molarity of a solution (known from the titration experiment)
V_2 = total volume of a prepared solution for the whole experiment
Then,

$$0.125 \times V_1 = 0.1 \times 5.0\ L$$

$$V_1 = 4.0\ L$$

Therefore, we need to add distilled water into 4.00 L solution up to 5.00 L solution to make it standard.

The Standardization of a Solution which is Less Concentrated (dilute) than Expected

Consider a prepared solution with 0.1 M NaOH which require to be titrated against a prepared solution of HCl which is too dilute. It is expected that a student will pipette 25.0 cm^3 of NaOH and titrate it against HCl. Under such a situation, you are required to prepare 5.0 litres of 0.1 M HCl using 12 M stock solution enough for may be 40 students. It is expected that the end point will occur when 25.0 cm^3 of HCl is used up. However, that is not the case, given that you will have prepared the HCl solution from which 30 cm^3 was used up to reach the end point instead of 25 cm^3. Therefore, the molarity of the acid solution that is less concentrated than expected should be calculated as follows:

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$

Moles of NaOH = moles of HCl (since its mole ratio is 1:1)

Let M_b be the molarity of base

V_b = volume of base

M_a = molarity of acid

V_a = volume of acid

Then; $M_aV_a = M_bV_b$

$M_a \times 30\ \text{cm}^3 = 0.1\ \text{M} \times 25.0\ \text{cm}^3$

$M_a = 0.0833\ \text{M}$

In this case, the molarity of acid is less concentrated than expected. To standardize such a solution, add more stock solution to it to raise the concentration such that

$M_bV_b = M_aV_a + \text{Mstock sol.} \times \text{Vstock sol.}$

$0.1 \times 5.0\ \text{L} = 0.0833 \times 5.0\ \text{L} + 12\ \text{M} \times \text{Vstock sol.}$

$\text{Vstock sol.} = 6.96\ \text{cm}^3$

Therefore, add about 7.0 cm^3 of stock solution into 5.00 L solution to make it standard.

Mathematically, this relation can be represented as follows:
$M_1V_4 = M_2V_4 + M_3V_3$
Where,
M_1 = required molarity of a solution (known)
M_2 = calculated molarity of a solution (known from the titration experiment)
M_3 = molarity of a stock solution (known)
V_3 = volume of a stock solution (unknown)
V_4 = total volume of a prepared solution for the whole experiment.

The Procedure

(i) Calculate the molarity of a prepared solution (M_2).
(ii) Calculate the volume required for preparing the solution (V_4).
(iii) Calculate the volume of a stock solution required (V_3) for the prepared solution.

The Standardization Procedure: Add V_3 into V_4 to obtain the required solution.

Note

Sometimes, the same cases may occur in solutions which are not primarily standard reagents like HCl acid and NaOH solution.
It is advised that:

- if HCl has to be standardized, use Na_2CO_3 which is a primary standard reagent that will later on, be used to standardize NaOH; and

- if NaOH has to be standardized, use oxalic acid which is a primary standard reagent that will later be used to standardize HCl.

3.1.2 The Double Indicator Method

In order to prepare an experiment for candidates to determine the volume of HCl used when Na_2CO_3 is titrated against HCl using POP and MO (double indicator method), observe the following key issues:

(a) Apparatuses Needed by the Teacher, the tutor and the Laboratory Technician

A chemical balance, a 250 cm^3 beaker, a stirring rod, a desiccator, a 100 cm^3 measuring cylinder, a 500 cm^3 volumetric flask and a filter funnel.

(b) Apparatuses Needed by the Candidate

- 1 burette
- 1 pipette
- 1 conical flask
- 1 burette stand
- 1 funnel
- 1 stirrer
- 1 white glazed tile

(c) Chemicals

- Sodium carbonate
- Distilled water
- Hydrochloric acid
- Methyl orange
- Phenolphthalein indicator

The Preparation of Solutions

For the preparation of standard solutions of 0.05 M Na_2CO_3 and 0.1 M HCl, follow the procedures as discussed in section 3.1.1.1.

A Sample Question

You are given the following;

AA: a solution of 0.06 M sodium carbonate

BB: a solution containing 1.825 g of hydrochloric acid in 500 cm^3 of aqueous solution

DD: methyl orange

CC: phenolphthalein indicator

The Procedure

(i) Pipette 20 cm^3 or 25 cm^3 of solution AA into a clean conical flask. Add to it two drops of CC.

(ii) Titrate this solution against solution BB from the burette until the colour changes from pink to colourless and record the first titre value.

(iii) Add about two drops of DD and continue to titrate until the second colour changes from yellow to pink and record the titre value.

(iv) Repeat procedures (i) to (iii) three times and record your results as shown in Table 3.

Table 3: Table of Results

Titration number	Pilot	1	2	3
Final reading using POP (cm^3)				
Final reading using MO (cm^3)				
Initial reading				
Titre value using POP (cm^3)				
Titre value using MO (cm^3)				

The Assessment

The candidates' assessment is based on their ability to prepare and fill in the table of results based on the titration experiment. Criteria to take into account when assessing the experimental data are the accuracy of the titre value, the proper placement of readings into cells and the filling in of data in Table 3 in two decimal places.

3.1.3 The Preparation of an Experiment for Candidates to Determine the Proportions of Na_2CO_3 and NaOH in a Mixture through Volumetric Analysis

In this section, the teacher, the tutor and the laboratory technician are expected to prepare an experiment for candidates to determine the proportions of Na_2CO_3 and NaOH in a mixture through volumetric analysis.

Key Issues

(a) Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician

A chemical balance, a 250 cm^3 beaker, a stirring rod, a dissector, a 100 cm^3 measuring cylinder, a 500 cm^3 volumetric flask and a filter funnel.

(b) Apparatuses Needed by the Candidate

- 1 burette
- 1 pipette (20 cm^3 or 25 cm^3)
- 1 conical flask
- 1 burette stand
- 1 funnel
- 1 stirrer
- 1 white glazed tile

(c) Chemicals

- Sodium carbonate, Na_2CO_3
- Hydrochloric acid
- Sodium hydroxide
- Methyl orange indicator
- Phenolphthalein indicator

The Preparations of Solutions

(a) Procedures for preparing a primary standard solution of Na_2CO_3 and HCl have been covered in the previous section.

(b) Sodium hydroxide 0.304 M
Molar mass of NaOH = 40 g/mol

To prepare a solution of NaOH from its solid chemical, the following factors should be considered: (i) the molarity of the solution to be prepared and (ii) the exact volume of the solution required.

From these requirements, the mass needed can be calculated and dissolved in the required amount of distilled water to obtain the required concentration. This can be obtained using a formula:

$$\text{Grams of chemical} = \frac{\begin{matrix}\text{molarity of} \\ \text{solution}\end{matrix} \times \begin{matrix}\text{molar mass} \\ \text{of chemical (g / mol)}\end{matrix} \times \begin{matrix}\text{volume of the} \\ \text{required solution (mL)}\end{matrix}}{1000\,\text{mL / L}}$$

Note: The percentage of purity should be considered.

This weight is thus directly measured by a chemical balance.

Therefore, mass to be dissolved in 1 litre $= \frac{0.304\ \text{mol / L} \times 40\ \text{g / mol} \times 2000\,\text{ml}}{1000\,\text{mL / L}}$

= 24.32 g

Hence, 24.32 g will be dissolved in 1litre to make 0.304 M.

A Sample Question

You are given the following;

A: a solution containing a mixture of sodium carbonate and sodium hydroxide in aqueous solution;

B: a solution containing 9.125 g of pure hydrochloric acid in 0.25 dm^3 of aqueous solution;

MO: a methyl orange indicator; and

POP: a phenolphthalein indicator.

The Procedure

(i) Pipette 20 cm^3 of solution A into a conical flask.
(ii) Add two or three drops of POP.
(iii) Titrate this solution against solution B until a colour change is observed.
(iv) Record the first titre value.
(v) Add two drops of MO to the same solution.
(vi) Continue to titrate until a second colour change is observed.
(vii) Record the second titre value.
(viii) Repeat the titration procedures (i) to (vii) to obtain three more readings.

3.1.4 The Preparation of an Experiment for Candidates to Standardize Sodium Thiosulphate using Potassium Permanganate

In this section, the teacher, the tutor and the laboratory technician are expected to prepare an experiment for candidates to standardize a solution of sodium thiosulphate using potassium permanganate.

Key Issues

(a) Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician

- A 50 cm^3 burette
- A 10 cm^3 pipette
- A conical flask
- A beaker
- A measuring cylinder

(b) Chemicals

- Potassium permanganate
- Sodium thiosulphate
- Dilute sulphuric acid
- Potassium iodide
- Starch solution
- Distilled water.

The Sample preparations

The Preparation of 0.02 M Potassium Permanganate Solution

(i) Dissolve 3.16 g of potassium permanganate in a beaker containing a small amount of water
(ii) Stir the mixture to dissolve the contents
(iii) Warm it on a water bath for a few minutes
(iv) Allow the solution to cool
(v) Transfer the solution to a 1 L volumetric flask
(vi) Add water up to the 1000 cm^3 mark

Note

The following points should be adhered to when preparing solutions:

(i) add concentrated acids, even small quantities, to water, not the vice-versa;

(ii) always use distilled water in preparing an aqueous solution;

(iii) do not heat potassium permanganate directly from a source; and

(iv) warm solutions should be cooled to room temperature before being measured to the required volumes.

3.1.5 The Preparation of an Experiment for Candidates to Standardize Sodium Thiosulphate Using Potassium Iodide

In this section, the teachers, the tutors and the laboratory technicians should be able to prepare solutions of sodium thiosulphate, potassium iodate, starch, and concentrated hydrochloric acid for practical examinations.

Key issues

(i) Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician

- A 50 cm^3 burette
- A 10 cm^3 pipette
- 3 Conical/Erlenmeyer flasks
- 2 L beakers
- Three 10 mL measuring cylinders
- 1 L storage bottle
- Burners and tripods

(ii) Chemicals

- Sodium thiosulphate
- Potassium iodate
- Potassium iodide
- Starch
- Concentrated hydrochloric acid

The Preparation of 0.1 M $Na_2S_2O_3.5H_2O$

This chemical can be bought in hydrated ($Na_2S_2O_3.5H_2O$) or anhydrous form ($Na_2S_2O_3$). However, despite that, any form can be used to prepare an experiment, the mass of the chemical to be dissolved will vary depending on specific instructions from *advanced practical instructions*.

The common concentration of sodium thiosulphate usually ranges from 0.1 to 0.5 M $Na_2S_2O_3$. To avoid a problem of premature precipitation of Sulphur in a stock solution before practical session, it is advised that you add about 0.2 g of sodium carbonate per litre of the prepared solution if water used to prepare the sodium thiosulphate solution has a pH value below 7.

Molecular weight of $Na_2S_2O_3.5H_2O$ = 248 g/mol
Hence, to prepare 0.1 M of $Na_2S_2O_3.5H_2O = 0.1\ mol/dm^3 \times 248\ g/mol$
= 24.8 g

Procedure

- (i) Take about 100 mL of water in a clean and dry 1000 mL volumetric flask.
- (ii) Add 24.8 g of sodium thiosulphate with constant stirring.
- (iii) Add about 0.2 g of sodium carbonate while constantly stirring.
- (iv) Add more water to the solution while stirring to make up the volume of 1000 mL solution. Mix it thoroughly and leave it to stand.
- (v) Store the solution.

Note

It is recommended that you complete to prepare sodium thiosulphate solution at least 30 minutes before commencing a practical examination.

The Preparation of Starch Indicator

Prepare a litre of starch indicator as follows:

- (i) prepare a paste by adding about 10 g of soluble starch (starch powder) to about 100 mL of deionized water;
- (ii) pour the paste into 900 mL of boiling distilled water for about 5 minutes;
- (iii) stir it until all starch is dissolved; and
- (iv) leave the solution to cool down. If precipitate is observed, decant the supernatant for use as indicator solution.

The Preparation of 10% KI

Mwt = 166 g
For a solid dissolving in a liquid, 10% is usually prepared on weight/volume (w/v) basis.

The Procedure

- (i) Weigh 100 g of KI and put it in a clean dry beaker.
- (ii) Add a small amount of distilled water and stir it to dissolve the content.
- (iii) Transfer the solution to a volumetric flask (1000 cm^3.) and fill it to the mark.
- (iv) For every one litre of KI solution prepared, add about 3 drops of the concentrated HCl, and mix it thoroughly.
- (v) Protect the prepared solution from direct light by storing it in amber bottles in a cool and dark place.

The Preparation of KIO_3

Molecular weight = 39 + 127+ (16 x 3)

= 214 g

To prepare 0.02 M solution of KIO_3

= 0.02 x 214

= 4.28 g of KIO_3 to be dissolved in a litre of distilled water.

Note: Consider the percentage purity of KIO_3 during the preparation.

The Procedure

(i) Weigh about 4.3 g of KIO_3 and transfer into a beaker containing a small amount of water.

(ii) Stir it to dissolve the content.

(iii) Pour the solution into a 1000 ml volumetric flask and add distilled water to it up to the mark.

(iv) Prepare a solution that corresponds to the number of candidates.

3.1.6 The Volumetric Estimation of Copper in Copper(II) Sulphate

Copper can be estimated volumetrically using potassium iodide solution. The theory behind it is that a copper solution oxidizes iodide in a solution while itself is reduced to Cu^+, the reaction that yields iodine

$2Cu^{2+} + 4I^- \rightarrow Cu_2I_2 \text{ or } (2CuI) + I_2$.

The Preparation of Standard $CuSO_4$ solution

(i) Weigh accurately the mass of $CuSO_4$ required

(ii) Transfer the sulphate into a 250 cm^3 volumetric flask

(iii) Dissolve it in distilled water. Add about 5 cm^3 acetic acid and shake it well

Note: The percentage purity of $CuSO_4$ should be taken into account during the preparation.

Key Issues

(a) Apparatuses Needed by the Teacher, the Tutor and the Laboratory technician

- A digital or Chemical balance
- A 500 cm^3 beaker
- A stirring rod
- A 100 cm^3 measuring cylinder
- A 250 cm^3 volumetric flask
- A filter funnel.

(b) Chemicals

- Solid $CuSO_4$
- Solid KI or 10% KI solution
- Starch
- Acetic acid
- $Na_2S_2O_3$
- Na_2CO_3
- Distilled water

(c) Apparatuses Needed by the Candidate

- 1 burette
- 1 pipette
- 1 conical flask
- 1 burette stand
- 1 funnel
- 5 beakers

Note

The preparation of HCl solutions and 10% KI have been discussed in sections 3.1.1.1 and 3.1.5, respectively.

The Reflection on Key Issues in the Preparation and Assessment

- A solution of $CuSO_4$ solution was prepared by reacting 4.50 g of impure CuO, with dilute H_2SO_4 and made up to 250 cm^3 of solution after the filtration and a solution of 24.80 g of $Na_2S_2O_3.5H_2O$ in a litre of solution, a 10% KI and a freshly prepared starch indicator.
- Copper(II) sulphate reacts with potassium iodide to liberate iodine. The liberated iodine is then titrated with the standard solution of the reducing agent such as sodium thiosulphate.

  $$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$$

- In this reaction, iodine is used as the connecting path since thiosulphate cannot react with oxidizing agent other than iodine. In the end, the amount of iodine is not considered in the calculations. The calculations will involve mole ratio between thiosulphate and copper(II) sulphate from the following reaction:

  $$2Cu^{2+} + 4I^- \rightarrow Cu_2I_2 \text{ or } (2CuI) + I_2 \text{..................(i)}$$

  $$2S_2O_3^{2-} + 2I_2 \rightarrow S_4O_6^{2-} + 4I^- \text{............................(ii)}$$

  $$2Cu^{2+} + 2S_2O_3^{2-} + 2I^- \rightarrow Cu_2I_2 + S_4O_6^{2-} \text{.......Overall}$$

  The mole ratios are the same. Hence, the concentration of copper(II) sulphate solution can be calculated using an equivalent concentration of the thiosulphate. However, the thiosulphate is standardised before use.

The Assessment

The candidates' assessment is based on their ability to fill in the table of results, the accuracy of their practical data, the observation of 2 decimal points as well as the manipulation of experimental data.

3.1.7 The Preparation of an Experiment for Candidates to Determine the Relative Atomic Mass of Unknown Element in a Solution

Key Issues

(a) Apparatuses for the Teacher, the Tutor and the Laboratory Technician

A digital or a Chemical balance, a 250 cm^3 beaker, a stirring rod, a 100 cm^3 measuring cylinder, a 500 cm^3 volumetric flask and a filter funnel.

(b) Apparatuses Needed by the Candidate

Apart from specific instructions in the advanced instructions, each candidates will use the following apparatuses:

- 1 burette
- 1 pipette
- 1 conical flask
- 1 burette stand
- 1 funnel
- 1 white glazed tile

(c) Chemicals

- Sodium hydroxide or potassium hydroxide
- Distilled water.
- Hydrochloric acid
- Methyl orange

The Preparation of Solutions

(i) KOH

Weigh 5.6 g of KOH. Dissolve it in distilled water to get 1 litre of a solution. If NaOH is used, weigh 4.0 g and dissolve it in 1 litre of a solution.

Note: The percentage purity should be considered in the calculations.

(ii) HCl

Has been discussed in section 3.1.1.1 (b)

Sample Question

You are provided with the following chemicals:

SS: A solution of 0.1 M potassium hydroxide

HH: A solution containing 1.8 g of hydrochloric acid in 500 cm^3 of aqueous solution

MO: Methyl orange

Procedure

(i) Pipette 20 cm^3 or 25 cm^3 of solution SS into a clean conical flask. Add to it two drops of **MO**.

(ii) Titrate this solution against solution **HH** from the burette until the colour change is observed and record the titre value.

(iii) Repeat the above procedure three times and record your results in Table 4.

Table 4: Table of results

Titration number	**Pilot**	**1**	**2**	**3**
Final reading (cm^3)				
Initial reading (cm^3)				
Titre value (cm^3)				

Assessment

Candidates are assessed based on their ability to determine the molarity of KOH in a solution. The assessment will be based on the accuracy of the titre value and manipulation of data. It also involves the candidate's competence in deducing the molecular mass of compounds and their constituent elements.

3.2 Chemical Kinetics and Energetics

In this content area of assessment, the teacher, the tutor or the laboratory technician are required to prepare a practical examination to assess candidates' competencies in determining the effects of various factors on the rate of chemical reaction and to determine reaction orders practically.

3.2.1 Chemical Kinetics

Chemical Kinetics refers to the rates of chemical reactions. It essentially investigates conditions that influence a speed at which reactants are converted into products.

(a) The Effect of the Concentration

Key Issues

(i) Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician:

Apart from the common fittings in the laboratory, it is recommended that each teacher, tutor and laboratory technician should have the following:

- Volumetric flask (1000 mL or 500 mL or 250 mL) depending on the number of candidates;
- A weighing balance (digital balance is recommended for quick measurements);
- At least two big containers (plastic or glass) with enough capacity to accommodate the volume of solutions to be prepared, and
- A blue or black pen/marker pen for uniform labelling of marks.

(ii) Apparatuses Needed by the Candidate/One Experimental Set up

Apart from specific instructions in the advanced instructions, each candidate will use the following apparatuses:

- 2 beakers (100 cm^3)
- a stop watch
- a white piece of paper/white tile,
- a measuring cylinder (100 cm^3),
- a thermometer (-10 to 110 ºC),
- 2 boiling tubes

(iii) Chemicals

- **Sodium thiosulphate**
  Its preparation has been covered in section 3.1.5
- **Sulphuric acid**
  The precipitation of sulphur occurs when thiosulphate reacts with an acid. The common concentration required to precipitate enough amount of Sulphur to obscure a given mark is given at different concentrations of the acid, depending on the advanced practical instructions. For example, a 0.5 M sulphuric acid solution can be prepared as follows:

$$\text{Molarity} = \frac{\text{density} \times \text{percentage purity} \times 1000}{\text{Molecular weight} \times 100}$$

$$= \frac{1.84\,\text{g/cm}^3 \times 98 \times 1000\,\text{cm}^3/\text{L}}{98.08\,\text{g/mol} \times 100}$$

$$= 18.38\,\text{M}$$

Then follow the following procedure

Dilute 18.38 M of an acid to get 0.5 M solution as follows:

(i) Use the dilution Law to get a volume of the pure acid enough to make 0.1 M sulphuric acid, thus:

$$M_c V_c = M_d V_d$$

M_c = molarity of concentrated solution

V_c = volume of concentrated solution

M_d = molarity of dilute solution

V_d = volume of dilute solution

(ii) Take about 1000 cm^3 of water in a clean volumetric flask, So that,

$$18.38\,\text{M} \times V_c = 0.5\,\text{M} \times 1000\,\text{cm}^3$$

$$= 27.2\,\text{cm}^3 \text{ of the concentrated sulphuric acid.}$$

Thereafter, measure about 27.2 cm^3 of the concentrated acid.

(iii) Add the acid slowly to water in a container while constantly mixing it.
(iv) Add water up to a volume of 1000 cm^3 solution. Mix it thoroughly

Note: In this experiment, it is not necessary to standardize the acid prepared because the experiment is qualitative.

This approach used in the dilution of sulphuric acid can be used in the preparation of dilute solutions from other concentrated acid solution (hydrochloric acid and nitric acid) required to prepare a specified molar concentration.

Example: The preparation of the dilute nitric acid (assay = 70%)

$$\text{Molarity} = \frac{\text{Density} \times \text{Percentage purity} \times 1000}{\text{Molecular weight} \times 100}$$

$$= \frac{1.82\,g/cm^3 \times 70 \times 1000\,cm^3/L}{63\,g/mol \times 100}$$

$$= 20.22\,M$$

$$M_cV_c = M_dV_d$$

M_c = molarity of concentrated solution

V_c = volume of concentrated solution

M_d = molarity of dilute solution

V_d = volume of dilute solution

Then,

$$20.22\,M \times V_c = 0.5\,M \times 1000\,cm^3$$

$$= 24.7\,cm^3 \text{ of the concentrated Nitric acid}$$

Note

Always add acid slowly to water while constantly mixing it. This will prevent a rapid heat generation and possible spattering of the mixture.

$$\text{Molarity} = \frac{\text{density} \times \text{percentage purity} \times 1000}{\text{Molecular weight} \times 100}$$

$$= \frac{1.84\,g/cm^3 \times 98 \times 1000cm^3/L}{98.08\,g/mol \times 100}$$

$$= 18.38\,M$$

Therefore, dilute 18.38 M of acid to get 0.5 M solution using the Dilution Law:

$$M_cV_c = M_dV_d$$

M_c = molarity of the concentrated solution

V_c = volume of the concentrated solution

M_d = molarity of the dilute solution

V_d = volume of the dilute solution

Then,

$$18.38\,M \times V_c = 0.5\,M \times 1000\,cm^3$$

$$= 27.2\,cm^3 \text{ of the concentrated sulphuric acid}$$

Note

Always add acid slowly to water with constantly mixing it. This will prevent a rapid heat generation and spattering of the mixture.

Then, follow the following procedure:

(i) Take about 500 mL of water in a cleaned flask (always fill your container about half way or more with distilled water);

(ii) Measure about 28 mL of the concentrated acid;

(iii) Add the acid slowly to water in a container while constantly mixing it., and

(iv) Make up the volume to 1000 mL with water. Mix thoroughly.

Note

In this experiment, it is not necessary to standardize the acid prepared because the experiment is qualitative. However, for titration experiments (quantitative analysis) you will need to standardize the acid as previously described in section 3.1.1.1. This approach can be used to calculate the volume of the concentrated acid solution (sulphuric acid, hydrochloric acid and nitric acid) required to prepare the specified molar concentration.

Nitric acid

$$\text{Molarity} = \frac{\text{Density} \times \text{Percentage purity} \times 1000}{\text{Molecular weight} \times 100}$$

$$= \frac{1.82\,g / cm^3 \times 70 \times 1000\,cm^3 / L}{63g / mol \times 100}$$

$$= 20.22\,M$$

$$M_cV_c = M_dV_d$$

M_c = molarity of the concentrated solution

V_c = volume of the concentrated solution

M_d = molarity of the dilute solution

V_d = volume of the dilute solution

Then,

$$20.22\,M \times V_c = 0.5\,M \times 1000\,cm^3$$

$$= 24.7\,cm^3 \text{ of the concentrated nitric acid}$$

Table 5 provides various volumes of preparations depending on the specifications of stock solution.

Table 5: The Percentage of Purity (assay), Density and Suggested Volume of HCl Required to Prepare 1 M HCl Solution

S/n	Percentage purity	Density (g/cm^3)	Approximate volume (cm^3) of the conc. Acid to be diluted to make 1 L solution
1.	31-32	1.16	100
2.	34	1.18	91
3.	35	1.18	88.5
4.	36	1.18	85.2
5.	37	1.18	83.5
6.	38	1.18	81.3

Table. 6: Suggested Volume of the Conc. HNO_3 Required to Prepare 0.1 - 0.5 M HNO_3 Solution (ρ 1.82 g/cm^3, % Purity 70%, Mr. 63 g/mol)

S/N	Concentration (mol/dm^3)	Approximate volume (cm^3) of the conc. Acid to be diluted to make 1L solution
1.	0.1	5
2.	0.2	10
3.	0.3	15
4.	0.4	20
5.	0.5	25

Table. 7: Suggested Volume of Conc. H_2SO_4 Required to Prepare 0.1 - 0.5 M H_2SO_4 Solution (ρ 1.84 g/cm^3, % Purity 98%, Mr, 98 g/mol)

S/N	Concentration (mol/dm^3)	Volume of the conc. Acid (cm^3) to be added to water to make make 1 L solution
1.	0.1	5.4
2.	0.2	11
3.	0.3	16.3
4.	0.4	22
5.	0.5	27.2

Table 8: Suggested Mass of $Na_2S_2O_3.5H_2O$ (Mr. 248 g) Required to Prepare 1 L of 0.1 - 0.5 M

S/N	Concentration (mol/dm^3)	Mass of $Na_2S_2O_3.5H_2O$ (g)
1.	0.1	24.8
2.	0.2	49.6
3.	0.3	74.4
4.	0.4	99.2
5.	0.5	124.0

Table 9: Suggested Mass of $Na_2S_2O_3$ (158 g) Required to Prepare 1 L of 0.1 - 0.5 M

S/N	Concentration (mol/dm^3)	Mass of $Na_2S_2O_3$ (g)
1.	0.1	15.8
2.	0.2	31.6
3.	0.3	47.4
4.	0.4	63.2
5.	0.5	79.0

A Sample Question

(i) Measure 50 cm^3 of 0.1 M sodium thiosulphate ($Na_2S_2O_3$) solution in a beaker.

(ii) Draw a cross on a white piece of paper and place the beaker or a conical flask on it.

(iii) Add 50 cm^3 of the sulphuric acid and immediately start a stopwatch. The flask should be swirled once or twice before it is placed on the piece of paper.

(iv) Look down vertically on the cross and record the time taken for the cross to disappear.

(v) Repeat procedures (i) – (iv) using 40, 30, 20 and 10 cm^3 of sodium thiosulphate. Always top up the solution with distilled water to make 50 cm^3 before adding the acid solution.

(vi) Tabulate the data from each experiment in columns indicating the volume of sodium thiosulphate solution (cm^3), the volume of water (cm^3), time (s), and the rate (s^{-1}) as shown in Table 10.

Table 10: The Experimental Data

Experiment	Vol. of $Na_2S_2O_3$ (cm^3)	Vol. of H_2O (cm^3)	Time (s)	Rate (s^{-1})
1.	50	0		
2.	40	10		
3.	30	20		
4.	20	30		
5.	10	40		

(vii) Plot a graph of volume (concentration) of sodium thiosulphate against time. What does the shape of the graph looks like and what does it indicate?

(viii) Plot a graph of the volume (concentration) of sodium thiosulphate against reciprocal of time (1/t). Give an interpretation of the shape of the graph.

(b) The Effect of Temperature

Key issues

- The teacher, the tutor and the laboratory technician should observe all issues pointed out under the effect of the concentration in section 3.2.1 (a).

- In addition, the teacher, the tutor and the laboratory technician should prepare a thermometer, source of heat and a stopwatch for each candidate who is to take the experiment. The instruments should be checked before distribution.

- The task of checking the instruments for their functioning should be performed early, preferably when attending the checklist or as a normal routine to ensure that all common fittings in the Laboratory are functioning. This saves time during the final preparations for practical examinations.

- The teacher, the tutor and the laboratory technician should ensure that all thermometers work properly. No broken thermometer(s) should be given to a candidate(s) or be placed in a set up.

- The teacher, the tutor and the laboratory technician should ensure that if there is a shift during the practical examination, each candidate cleans apparatuses properly and rinses them with distilled water. This will reduce/minimize the contamination and save time for preparing the laboratory, especially if a school/centre/college has more than one session in one alternative practical paper.

- If a centre has a shift of candidates in this question, all chemicals used by a previous candidate should be discarded so that the next candidate is given fresh chemicals from the stock. The teacher, the tutor and the laboratory technician should ensure that candidates are given the recommended amount of chemicals as specified in the advanced instructions.

- The teacher, the tutor and the laboratory technician should ensure that beakers (50 cm^3 or 100 cm^3) are available in the laboratory. The use of beakers which are not specified in the checklist/advanced practical instructions, greatly affects results of an experiment. They also take excess time and cause panic to candidates. Hence, in this experiment, all candidates should use beakers with uniform volume/capacity.

A Sample Question

An experiment to determine the effect of temperature on the rate of reaction.

(i) Draw a mark X on a square white sheet of paper.
(ii) Measure 10 cm^3 of 0.25 M of sodium thiosulphate solution into a conical flask and place the flask on the white paper above the cross.
(iii) Add 40 cm^3 of water to make a total volume of 50 cm^3.
(iv) Heat the solution to a temperature of 30 ºC.
(v) To the solution, add 4 cm^3 of 2 M hydrochloric acid and immediately start a stopwatch.
(vi) Note the time that the cross on the white paper disappears.
(vii) Repeat the procedures above with a solution heated to 40 ºC, 50 ºC, 60 ºC and 70 ºC.

(viii) Record the data in Table 11, indicating the changes of the rates of reaction according to the temperature.

Table 11: The Experimental Data

Temperature (^oC)	30	40	50	60	70
Time (t) in seconds					
Rate (1/t) sec^{-1}					

(i) Plot a graph of the reaction time (t) against the temperature (T). Interpret the graph.

(ii) Plot a graph of the reciprocal reaction time (1/t) against the temperature (T). Interpret the graph.

The Assessment

Candidates' assessment is based on their ability to record the experimental data and fill in the table, the accuracy of their data, the manipulation of data and plotting of graph(s) meanwhile observing the essentials of a graph such as:

(a) the title
(b) scales (Horizontal and Vertical)
(c) labelled axes
(d) the line of best fit
(e) indication of the points used to calculate the slope

Note: The graph should be as large as the paper allows.

(c) The Surface Area or the Particle Size

The surface area or the particle size is one of the determinants of the speed at which chemical reactions take place. In ordinary conditions, the particles with large surface area have little chance of contacting the second reactant. Therefore, in order for the reaction to be faster, the reacting substance has to be changed into small granules since the smaller the size of the reacting solid particles, the faster the reaction.

Key Issues

(i) Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician:

Apart from the common fittings in the laboratory, each teacher, tutor and laboratory technician is instructed to prepare the following:

- a volumetric flask (1000 mL or 500 mL or 250 mL), depending on the number of candidates;
- a weighing balance (digital balance is recommended for quick measurements);
- at least two containers (plastic or glass) with enough capacity to

accommodate the volume of solutions to be prepared;

- a blue or black pen for uniform labelling of marks;
- plain papers for making labels, and
- a stopwatch/clock.

(ii) Apparatuses Needed by the Candidate

Apart from specific instructions in the advanced instructions, the candidate will require:

- a stopwatch
- a measuring cylinder (100 cm^3),
- a thermometer (-10 to 110 ºC),
- a weighing balance
- a gas jar/gas syringe

(iii) Chemicals

- **$CaCO_3$**
  This chemical can be bought in a form of marble chips or powdered form.
- **Dilute H_2SO_4**
  The common concentration required to react with the carbonate is 0.1 M sulphuric acid solution. The preparation of the dilute acids has been described in section 3.1.1.1

  An example of chemical reactions that are affected by the particle size is insoluble carbonates with acid. This reaction can be done in two ways: by investigating the change in the rate of reaction; and by varying the weight of the marble or determining the rate of reaction by observing the rate of production of carbon dioxide evolved.

An Experiment to Determine the Effect of Surface Area Using Marble Chips

Key Issues

(i) Apparatuses Needed by the Teacher, the tutors and the Laboratory Technician

- a weigh balance, preference should be the digital balance to save time
- a volumetric flask
- several beakers/watch glasses for keeping the marble chips that have been measured

(ii) Apparatuses Needed by the Candidate

- a conical flask
- a stop watch/clock
- a water trough
- a measuring cylinder
- a gas jar

- a delivery tube
- rubber stoppers

(iii) Chemicals

The preparations of HCl, HNO_3 and H_2SO_4 have been described in section 3.1.1.1 and 3.2.1

The Procedure

(i) Put 10 g of dry calcium carbonate in a 250 cm^3 conical flask.

(ii) Fill a 500 cm^3 measuring cylinder with water and invert it over a beehive shelf placed in a trough which is three-quarter full of water. Make sure that air bubbles do not penetrate into the measuring cylinder.

(iii) Connect the flask and the measuring cylinder placed in a trough using a delivery tube. Use a tight rubber cork at the mouth of the conical flask.

(iv) Open the rubber bung and pour in 10 cm^3 of 0.1 M hydrochloric acid on carbonate chips in the conical flask. Cover the flask and start the stopwatch/clock immediately.

(v) Read the level of gas in the measuring cylinder after every 30 seconds.

(vi) Continue taking readings until no more acidified water remains in the measuring cylinder.

(vii) Grind another 10 g chips to a very fine powder. Repeat the whole experiment by using the carbonate powder. Record your readings as shown in Table 12.

Table 12: The Experimental Data

Large marble chips			**Powdered marble chips**		
Time (s)	Vol. of CO_2 evolved (cm^3)	Rate 1/t (s^{-1})	Time (s)	Vol. of CO_2 evolved (cm^3)	Rate 1/t (s^{-1})
30			30		
60			60		
90			90		
120			120		
150			150		

(viii) Plot two graphs on the same axes, one for each of the two experimental data.

(ix) Plot volume (y-axis) against time (x-axis) on the same graph paper. Label a graph of the large marble chips as L and that of the powdered chip as P.

(d) The Catalyst

A catalyst is a chemical substance added to reactants to alter (increase or decrease) the speed at which the reaction occurs. The catalyst lowers the activation energy so as to increase the effective collision of reacting particles per time.

Common chemical catalysts and reactions they catalyse are:

(i) manganese(IV) oxide in the preparation of oxygen gas from hydrogen peroxide;

(ii) iron in the preparation of ammonia by Haber Process

(iii) copper(II) oxide in the laboratory preparation of oxygen using potassium chlorate;

(iv) vanadium(V) oxide in the preparation of sulphuric acid by the contact Process.

Key Issues

(i) Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician:

In addition to the common laboratory fittings, the following are required:

- a volumetric flask (1000 mL or 500 mL or 250 mL) depending on the number of candidates;
- a weighing balance (digital balance is recommended for quick measurements);
- at least two or three big containers (plastic or glass) which are big enough to accommodate the volume of solutions to be prepared;
- a blue or black pen/marker pen for uniform labelling of marks;
- plain papers for making labels; and
- a stopwatch/clock.

(ii) Apparatuses Needed by the Candidate

- A stop watch
- 2 test tubes
- A splint
- A spatula
- A filter funnel and a filter paper

(iii) Chemicals

- Hydrogen peroxide
- Manganese(IV) oxide

A Sample Question: An experiment to investigate the effect of catalyst on the rate of chemical reaction

The Procedure

(i) Fill each test tube with hydrogen peroxide and label them as P and Q. Test if there is any oxygen has evolved by holding a glowing splint at the mouth of each test tube.

(ii) Use a spatula to add a small amount of MnO_2 to test tube P. Test the gas and observe the extent of the flame when MnO_2 is added.

(iii) Filter the content of test tube P and examine any change in the weight of MnO_2.

(iv) Scrap MnO_2 from the filter paper and add it to test tube Q and observe the extent of the flame.

(v) Record the results.

Discussion

- What is the function of a splint in this experiment?
- Why were there variations in the flame, of a glowing splint when it was placed at the mouth of test tube Q and in P when manganese(IV) oxide was added?
- Which other solid substances can be used in this experiment instead of manganese(IV) oxide?

(e) Activation Energy

Activation energy is the minimum energy required to initiate a collision of reacting molecules or atoms to change it into products.

Key Issues

(i) Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician

Common laboratory fittings and the following additional requirements:

- a volumetric flask (1000 mL or 500 mL or 250 mL) depending on the number of candidates;
- a weighing balance (digital balance is recommended for quick measurements)
- At least two or three big containers (plastic or glass) enough to accommodate the volume of solutions to be prepared.
- A blue or black pen/marker pen for uniform labelling of marks
- Plain papers for making labels
- a stopwatch/clock

(ii) Apparatuses Needed by the Candidate

Apart from the specific requirements instructed in the advanced instructions, each candidate will use the following apparatuses:

- a thermometer (0 – 100 °C)
- a stopwatch
- beakers
- a blue or black pen for making labels

(iii) Chemicals

0.05 M sodium thiosulphate solution and 0.1 Nitric acid solution.

A Sample Question

The laboratory experiments aimed at determining activation energy. Perform an experiment to find out the activation energy of the reaction between sodium thiosulphate and dilute acid.

The reaction follow this equation:

$$S_2O_3^{2-}(aq) + 2H^+(aq) \rightarrow H_2O(l) + SO_2(g) + S(s)$$

The Procedure

(i) Place a 50 cm^3 beaker on top of a letter X marked on a sheet of paper provided.
(ii) Prepare a water bath using a 250 cm^3 or 300 cm^3 beaker.
(iii) Measure 10 cm^3 of sodium thiosulphate and 10 cm^3 of hydrochloric acid and put them into two different test tubes.
(iv) Place a thermometer in one of the test tubes, then place both test tubes into the water bath prepared in step (ii) and warm the test tube contents to 40 ºC.
(v) Immediately pour hot solutions of sodium thiosulphate and hydrochloric acid in the 50 cm^3 beaker in step (i) and simultaneously start a stopwatch/clock. Record the time taken in seconds, for the mark to disappear completely.
(vi) Repeat procedure (i) to (v) at different temperatures as shown in Table 13.

Table 13: Experimental Data

Temperature		**Time (s)**	**Reaction rate (1/t) s^{-1})**	**1/T /K^{-1} (x 10^{-3})**	**log 1/t (s^{-1})**
T (°C)	**T (K)**				
40	313				
50	323				
60	333				
70	343				
80	353				

(vii) Plot a graph of log 1/t against 1/T

(f) Determination of the Order of Reactions

Teachers, tutors and laboratory technicians would be required to prepare a practical examination for candidates to determine 1st and 2nd orders.

Key Issues

(i) Apparatuses Needed by the Teacher, the Tutor or the Laboratory Technician

Apart from the common fittings in the laboratory, it is recommended that each teacher/Laboratory Technician needs to have the following:

- a volumetric flask (1000 mL or 500 mL or 250 mL) depending on the number of candidates;
- a weighing balance (digital/chemical);
- at least two or three big containers (plastic or glass) enough to accommodate the volume of solutions to be prepared, and
- a blue or black pen/marker pen for uniform labelling of marks.

(ii) Apparatuses Needed by the Candidate/one Experimental Set up

Apart from specific instructions in the advanced instructions, each candidate will use the following apparatuses:

- 1 small beaker (50 or 100 cm^3);
- 1 stopwatch;
- 2 measuring cylinders of 10 cm^3 ;
- a thermometer (-10 to 110 ºC);
- 2 boiling tubes; and
- a piece of white paper marked as per advanced practical instructions.

Note

- Small beakers should be uniform for all candidates. This is important to enable the candidates' experimental data to fall within the required range, given that the size of a beaker greatly affects the recorded time.
- Two measuring cylinders should be prepared for each candidate/one experimental set up to avoid/minimize the contamination.
- Check/inspect all stopwatches and change batteries in them whenever necessary.
- It is strongly advised that all apparatuses in the set up be disconnected and **cleaned thoroughly** so that the next user of the set up starts afresh.

(iii) Chemicals

- 0.20 M sodium thiosulphate solution
- 0.10 M hydrochloric acid solution
- Distilled water

The Preparation

Prepare chemicals as instructed in other sections.

Note

To avoid the premature precipitation of sulphur due to the acidic nature of water, add about 0.2 g Na_2CO_3 to each litre of sodium thiosulphate solution.

A Sample Question

The Procedure

(i) Put a small beaker on top of a mark drawn on a piece of white paper such that it is seen through the bottom of the beaker.

(ii) By using a measuring cylinder, measure 2 cm^3 of sodium thiosulphate and 8 cm^3 of distilled water and put them in a beaker.

(iii) Use another measuring cylinder to measure 10 cm^3 of hydrochloric acid and pour it into the beaker containing sodium thiosulphate and water. Immediately, start a stopwatch/clock.

(iv) Record the time taken until enough precipitation of sulphur obscures the mark.

(v) Repeat the experiment using various volumes of sodium thiosulphate solution and water.

Assessment

Candidates will be assessed on their ability to collect experimental data within the acceptable range. They will also be assessed on how they manipulate experimental data through plotting a graph and performing related calculations. Essentials of a graph such as title, scale, labelling of axes, slope and best line will also be observed.

3.2.2 Chemical Energetics

Chemical reactions are associated with energy changes. This energy change is usually in the form of heat and at the constant pressure. It is defined as the heat of reaction or enthalpy change (ΔH), which can be absorbed or released. For example, to form 1 mole of a compound from its constituent elements, the necessary amount of enthalpy change occurs and this change is defined as the enthalpy of formation.

In this section, candidates will be assessed on their ability to determine practically if the heat is absorbed (endothermic) or released (exothermic). In so doing, the teacher, the tutor and the laboratory technician should be able to prepare an experiment for candidates to:

(i) determine the heat of neutralization; and
(ii) determine the heat of solution.

(a) The Standard Enthalpy of Neutralization

The standard enthalpy change of neutralization occurs when acid solution and alkali solution react together under standard conditions to produce 1 mole of water. Enthalpy change of neutralization is always measured per moles of water formed. The values of enthalpy change of neutralization are always negative (exothermic reaction).

Key issues

(i) Apparatuses Needed by the Teacher, the Tutor or the Laboratory Technician

- a volumetric flask (1000 mL or 500 mL or 250 mL) for preparing the standard solution depending on the number of candidates, and
- a weighing balance (digital or chemical balance).

(ii) Apparatuses Needed by the Candidate

Apart from the common fittings in the laboratory, each teacher, tutor and laboratory technician should have the following:

- a thermometer (-10 to 110 ºC);
- a polystyrene cup/insulated vessel and a lid;
- 2 beakers (100 or 150 cm^3), and
- 1 measuring cylinder (10 cm^3).

All candidates should be given a calorimeter/plastic beaker/polystyrene cup/Styrofoam cup of the same **nature** and **size**. This is important in increasing the precision of the experimental values.

The Assessment

Candidates are assessed on their ability to record and manipulate experimental data. Nonetheless, there is a tolerable range of experimental data.

(iii) Chemicals

0.5 M HCl and 0.5 M NaOH (follow specified concentrations in the advanced instructions)

The procedure on how to prepare HCl has been provided in the previous sections

To prepare 0.5 M NaOH, first find the molecular weight of NaOH, which is 40 g. Then, the mass of NaOH required to make 0.5M NaOH is 0.5 M × 40 g/mol = 20 g

Note

The percentage purity of NaOH should be considered during the preparation.

The Procedure

(i) measure about 20.0 g of solid sodium hydroxide on a watch glass.
(ii) take the measured sodium hydroxide into a beaker containing some amount of distilled water.
(iii) using a wash bottle, clear all small particles remaining on the watch glass into the beaker.
(iv) add distilled water into the beaker while stirring until all solute dissolves.
(v) transfer this solution into a 1000 cm^3 volumetric flask. Use a wash bottle to clear all remaining solution from the beaker into the volumetric flask, and finally
(vi) add distilled water to the mark.

A Sample Question

The following is an experiment to determine the enthalpy change of neutralization.

(i) Measure 100 cm^3 of 0.5 M HCl using a measuring cylinder.
(ii) Pour this solution into a clean calorimeter/insulated vessel and record the temperature of the content.
(iii) Rinse the used measuring cylinder with distilled water and reuse it to measure 100 cm^3 of solution 0.5 M NaOH.
(iv) Use a thermometer to measure the temperature of solution in the measuring cylinder.
(v) Pour the contents of the measuring cylinder into the beaker containing a solution.
(vi) Stir the mixture gently and use the thermometer to determine and record the final temperature reached.
(vii) Record all your readings in a tabular form.

The Assessment

Candidates will be assessed based on their ability to experimentally determine the enthalpy of neutralization. Criteria for assessing the experimental data will be set whereby the candidates' data should be within the tolerance limit. The manipulation of experimental data will be assessed. The essentials of a graph (title, scales, labelling of

Cartesian planes, best line/nature of a graph as well as the indication of y-intercept and slope) will be taken into account during the assessment.

(b) The Enthalpy of Solution

Key Issues

(i) Apparatuses Needed by the Candidate

Apart from specific instructions in the advanced practical instruction, prepare the following:

- 1 calorimeter/styrofoam cup/beaker;
- 1 stopwatch/clock;
- 1 glass rod;
- 1 measuring cylinder (50 or 100 or 150 cm^3 capacity);
- 1 thermometer (0 -100 °C); and
- cotton wool/blanket/saw dust (for improvising a calorimeter or an alternative to styrofoam cups).

(ii) Chemicals

- 2 g of each of the salts, NH_4NO_3, $CaCl_2$, NaOH, NaCl
- Distilled water

For a quick performance of this task, a school/centre/college is advised to have at least two digital balances.

If styrofoam cups are not available, improvise a calorimeter by immersing a small beaker into a large beaker. Fill spaces between the beakers with cotton wool, saw dust or blanket.

The Assessment

Candidates will be assessed on ability to determine the enthalpy/molar enthalpies of the given salts practically.

A Sample Question

You are provided with 2 g of each of the following salts: ammonium nitrate; calcium chloride; sodium chloride and sodium hydroxide.

The Procedure

(i) Use a measuring cylinder to measure 40 cm^3 or 50 cm^3 of distilled water and pour it into a styrofoam cup.
(ii) Place a thermometer in the cup containing water and record its temperature.
(iii) Add ammonium nitrate and immediately start your stopwatch/clock. Swirl it to mix and continue taking the temperature data at 30 seconds intervals while swirling the solution occasionally for six minutes.
(iv) Repeat steps (i) to (iii) for each of the given salts.

(v) Record your results in a tabular form.

3.3 Qualitative Analysis

A Qualitative analysis is a process involving the use of bench reagents in the identification of components of a chemical substance. Qualitative analysis involves identifying substances present in a sample qualitatively. It is used to determine the presence of anions and cations in a given sample. However, teachers, tutors and laboratory technicians face several challenges preparing bench reagents needed for qualitative analysis practical works.

A Specific Objective

In this section, the teacher, the tutor and the laboratory technician should be able to prepare necessary bench reagents for carrying out chemistry experiments to determine unknown cations and anions from given samples.

Key Issues

Apparatuses Needed by the Teacher, the Tutor or the Laboratory Technician

- A Chemical balance or a digital balance
- Volumetric flasks
- Beakers, watch glasses
- Filter papers
- Reagent bottles (clear and brown)
- Glass rods
- Wash bottles
- Labels.

Apparatuses Needed by the Candidate

- Boiling tubes (pyrex)
- Wash bottle
- A test tube holder
- A nichrome or a platinum wire
- Watch glasses

Note: Each candidate should be given a Qualitative Analysis Guide (QAG) sheet which is provided by the National Examinations Council of Tanzania (NECTA). The guide is easily downloaded at https://www.necta.go.tz/approved_exam_guides. Make copies of the QAG to be enough for all of your candidates.

Chemicals

- Hydrochloric acid
- nitric acid
- sulphuric acid
- sodium hydroxide

- ammonium hydroxide
- calcium hydroxide
- barium chloride
- ferrous sulphate
- silver nitrate
- potassium iodide
- methyl orange
- phenolphthalein
- potassium ferrocyanide
- potassium ferricyanide
- potassium thiocyanate
- alcohol (ethanol)
- acetic acid
- ammonium carbonate
- ammonium oxalate
- ferric chloride
- lead acetate
- magnesium sulphate
- potassium chromate
- potassium dichromate
- copper powder
- distilled water

3.3.1 The Preparation of Bench Reagents

Bench reagents are prepared by dissolving the appropriate amount of solid in water or by diluting concentrated stock solutions. For all solutions, the weight of a substance contained in a litre of a molar solution is given and from this figure, other concentrations may be calculated.

Since some chemicals are expensive or sparingly soluble, special concentrations of such solutions are generally used. These are, therefore, included in a list of reagents requiring a special preparation. Distilled water should be used for all aqueous solution unless otherwise stated. The following should be adhered to in the process of bench reagent preparation:

(a) the preparation of bench reagents has to involve the accurate measurements of masses and volumes. Precautions should be taken in handling chemical substances such as corrosive acids and irritating solids like sodium hydroxide;
(b) the prepared reagents should be placed in the labelled containers showing the concentrations and the date of preparation;
(c) reagents should be placed in the appropriate reagent bottles, and
(d) an appropriate interpretation of information on chemical containers is quite necessary in the preparation of bench reagents.

Tables 14 and 15 show how to prepare the bench reagents for a qualitative analysis from solid chemicals and concentrated solutions, respectively.

Table 14: The Preparation Guideline for Bench Reagents from Solid Chemicals

S/No.	Name of the Reagent	How to Prepare it	Concentration
1.	Ammonium acetate	Dissolve 231 g of the salt in 1 litre of solution.	3 M
2.	Ammonium molybdate	Dissolve 45 g of ammonium molybdate in 40 ml concentrated ammonia solution, add 60 ml water and 100 g NH_4NO_3 slowly with vigorous stirring. Add water to make 1 litre.	-
3.	Ammonium carbonate	Dissolve 192 g of the salt in a mixture of 140 mL conc. ammonia solution and water to make 1 litre of solution.	2 M
4.	Ammonium chloride	Dissolve 160.5 g of the salt to make 1 litre of solution.	3 M
5.	Ammonium nitrate	Dissolve 80 g of the salt to make 1 litre of solution.	1 M
6.	Ammonium oxalate	Dissolve 62 g of the crystalline salt to make 1 litre solution.	0.5 M
7.	Ammonium sulphate (saturated solution)	Dissolve 750 g of the salt to make 1 litre of solution.	5.7 M
8.	Aqua Regia solution	Mix 3 parts of the concentrated hydrochloric acid with one part of the concentrated nitric acid.	-
9.	Ammonium sulphide	Use the yellow commercial product	-
10.	Ammonium thiocyanate	Dissolve 38 g of the salt to make1 litre of solution.	0.5 M
11.	Barium chloride	Dissolve 69.8 g of the salt to make 1 litre of solution	0.25 M
12.	Bromine water	Shake 35 g or 11 mL of liquid bromine with water to make a saturated aqueous solution. Add more bromine if necessary a slight excess.	-
13.	Calcium hydroxide,	Boil water vigorously for one hour. Cool it in a flask fitted with a soda lime guard tube. Add 2 g of calcium hydroxide to a litre of water and shake it well. After several hours, siphon, or filter off the clear liquid and store it in a bottle fitted with a guard tube. **OR** Dissolve 2 g of $Ca(OH)_2$ to make 1 litre of	-

S/No.	Name of the Reagent	How to Prepare it	Concentration
		solution.	
14.	Calcium chloride	Dissolve 55 g of the hexahydrated ($CaCl_2.6H_2O$) or 27.8 g of anhydrous to make 1 litre of solution.	0.25 M
15.	Calcium sulphate	Shake 2.04 g of the salt with 1 litre of water, filter and decant the saturated solution after several hours.	0.015 M
16.	Chlorine water	Saturate 250 mL of water with chlorine, the chlorine may be prepared by dropping conc. HCl upon $KMnO_4$. Preserve it in a dark coloured bottle.	6.8 g/l
17.	Cobalt nitrate	Dissolve 44 g of the hexahydrate salt or 27.5 of anhydrous to make 1 litre of solution.	0.15 M
18.	Copper sulphate	Dissolve 125 g of the hydrated salt or 80 g of anhydrous salt in 1 litre of water containing 3 mL of the conc. sulphuric acid.	0.5 M
19.	Ferric chloride	Dissolve 135.2 g of $FeCl_3.6H_2O$ in water containing 20 mL of the concentrated HCl, dilute to 1 L with water and filter if necessary.	0.5 M
20.	Ferrous sulphate	Dissolve 139 g of ferrous sulphate heptahydrate or 76 g of anhydrous ferrous sulphate in water containing 7 ml of the concentrated sulphuric acid to make 1 L of solution.	0.5 M
21.	Hydrogen sulphide	H_2S generated from a Kipps apparatus.	(~42 g/l)
22.	Iodine solution	Dissolve 12.7 g of iodine in a solution of 20 g of pure KI in 30 mL of water, and dilute it to 1 litre of solution.	0.05 M
23.	Lead acetate	Dissolve 95 g of $Pb(C_2H_3O_2)_2.3H_2O$ to make 1 L water. Add sufficient dilute acetic acid to clear the solution.	0.25 M
24.	Magnesium sulphate	Dissolve 62 g of $MgSO_4.7H_2O$ in 1000 mL water.	0.25 M
25.	Methyl orange,	Dissolve 1.0 g of methyl orange in 800 mL of water and then add 200 mL of ethanol.	-
26.	Phenolphthalein indicator solution	Dissolve 5 g in 500 mL ethanol; add 500 mL water with constant stirring.	-
27.	Potassium chromate	Dissolve 49 g of the salt to make 1 litre of solution.	0.25 M
28.	Potassium dichromate	Dissolve 35 g of the salt to make 1 litre of	0.12 M

S/No.	Name of the Reagent	How to Prepare it	Concentration
		solution	
29.	Potassium ferricyanide	Dissolve 55 g of the salt to make 1 litre of solution.	0.167 M
30.	Potassium ferrocyanide	Dissolve 53 g $K_4Fe(CN)_6.6H_2O$ of the salt to make 1 litre of solution.	0.125 M
31.	Potassium iodide	Dissolve 83 g of the salt to make 1 litre of solution.	0.5 M
32.	Potassium permanganate	Dissolve 1.58 g of the salt in hot water, dilute to 1 litre, and filter through glass wool	0.01 M
33.	Potassium hexacyanoferrate(II)	Dissolve 105 g of the salt to make 1 litre of solution.	0.25 M
34.	Potassium thiocyanate	Dissolve 48.5 g of the salt to make 1 litre of solution.	0.5 M
35.	Silver nitrate	Dissolve 17 g of the salt to make 1 litre of solution.	0.1 M
36.	Sodium acetate	Dissolve 408 g of the salt to make 1 litre of solution.	3 M
37.	Sodium carbonate	Dissolve 429 g of the decahydrate salt or 159 of the anhydrous salt to make 1 litre of solution.	1.5 M
38.	Zinc nitrate	Dissolve 94.7 g of the salt to make 1 litre of solution.	0.5 M
39.	Sodium hydroxide	Dissolve 200 g of the salt to make 1 litre of solution.	5 M
40.	Silver nitrate	Dissolve 43 g of the salt to make 1 litre of solution.	0.25 M

Table 15: The Preparation Guideline for Bench Reagents from the Concentrated solutions (1 L of 1 M)

S/No.	Acids	Molarity	%	S.G.	Required volume (mL)
1.	Acetic acid, MW=60.05	17.4 M	99	1.05	58
2.	HCl, MW=36.46	11.6 M	36	1.18	86
3.	Nitric acid, MW=63.01	16.4 M	69	1.42	61
4.	H_2SO_4, MW=98.07	17.6 M	95	1.84	57
5.	Ammonia solution, MW=35.05	14.5 M	28	0.88	69

Assessment

- The candidates will be assessed in their ability to follow systematic procedures for identifying samples given (CSEE, ACSEE and DSEE).

- The candidates will also be assessed in their ability to carry out group separation (ACSEE).
- The candidate will be assessed on their ability to follow the guided procedures and write observations and inferences (CSEE, ACSEE and DSEE).

3.3.2 A Sample Procedure for Identifying Ions in a Salt Sample

Qualitative analysis procedures involve some steps in identifying components of a salt sample. The following are the steps necessary to be followed in determining the identity of the salt sample.

(a) Preliminary Tests

(i) The Appearance of a Compound

The appearance of a compound can help in its identification, i.e., colour and texture. Ionic compounds are generally white or colourless. Ions of transition elements tend to be coloured, e.g.

Cu^{2+} - blue
Fe^{2+} - pale green
Fe^{3+} - yellowish brown

(ii) The Solubility

Some salts are very soluble in water, examples are KI, $CuSO_4$, NH_4Cl. Some salts are extremely insoluble in water, e.g., $CaCO_3$ and $PbCO_3$. Some salts are partially soluble. These include AgCl, $PbCl_2$ and $Ca(OH)_2$. Soluble compounds such as NaOH, Na_2CO_3, $(NH_4)_2CO_3$ and KI are used to precipitate other salts from solutions. A qualitative analysis work depends very much on whether one knows which salts are soluble and which ones are not.

(iii) The Flame Test

A flame test involves mixing solid ions with the concentrated hydrochloric acid solution and heating them on a nichrome wire over a non-luminous flame. The flame observed shows the characteristic colour of the element. The following are the characteristic flame colours of some metal ions.

Sodium, Na is golden yellow
Potassium, K is pale violet/lilac
Calcium, Ca is brick red
Copper, Cu is green/blue

(iv) The Action of Heat

Different substances decompose upon the heating to give different products. If a compound is heated and a colourless liquid condenses on the side of the test tube, the compound is most likely to be an hydrated and the liquid is probably water. A test to confirm the presence of water is performed using Cobalt(II) chloride paper and anhydrous Copper(II) sulphate. If a brown gas is given off, the compound is more likely to be a nitrate.

(v) The Reaction with Sodium Hydroxide

Add dilute sodium hydroxide solution drop by drop to a test solution. Stir or shake the mixture and observe any reaction. If the precipitate forms continue, add sodium hydroxide solution in excess. If no precipitation is formed, warm the mixture.

(vi) The Reaction with Aqueous Ammonia

Add dilute aqueous ammonia drop by drop to a test solution. Stir or shake the mixture and observe any reaction. If a precipitate forms, continue adding aqueous ammonia.

(b) Confirmatory Tests for Anions

Nitrates (NO_3^-)

To a nitrate solution in a test tube, a freshly prepared iron(II) sulphate is added followed by a careful addition of the concentrated H_2SO_4 on the side of a test tube. A brown ring is formed. The reactions involved are shown here below:

$$XNO_3(s) + H_2SO_4(aq) \longrightarrow XHSO_4(aq) + HNO_3(aq)$$

Nitrate solution

Where X represents a cation

The nitric acid formed is reduced by some of the iron(II) sulphate to nitrogen monoxide, (NO).

$$6FeSO_4(aq) + 2HNO_3(aq) + 3H_2SO_4(aq) \longrightarrow 3Fe_2(SO_4)_3 + 4H_2O(l) + 2NO(g)$$

The nitrogen monoxide then reacts with iron(II) sulphate to give a brown compound, $FeSO_4.NO$ which appears as a brown ring.

$$FeSO_4(aq) + NO(g) \longrightarrow FeSO_4.NO(aq)$$

Brown ring

Sulphates (SO_4^{2-})

To a solution of a sample, dilute HCl is added followed by $BaCl_2$ solution. A white precipitate insoluble in dilute HCl is formed.

$$BaCl_2(aq) + SO_4^{2-}(aq) \longrightarrow 2Cl^-(aq) + BaSO_4(s)$$

$$\text{i.e. } Ba^{2+}(aq) + SO_4^{2-}(aq) \longrightarrow BaSO_4(s)$$

Chlorides (Cl^-)

To a sample solution, $AgNO_3$ solution is added followed by dilute HNO_3 solution then excess ammonia solution. A white precipitate insoluble in dilute HNO_3 but soluble in ammonia solution is formed.

$$Ag^+(aq) + Cl^-(aq) \longrightarrow AgCl(s)$$

$$AgCl(s) + 2NH_3(aq) \longrightarrow [Ag(NH_3)_2]^+(aq) + Cl^-(aq)$$

Carbonates (CO_3^{2-}) and Hydrogen carbonates (HCO_3^-)

To a solid sample, dilute HCl is added. An effervescence and a colourless gas, which turns limewater milky is evolved. The gas is CO_2, indicating the presence of CO_3^{2-} or HCO_3^-. To a solution of a sample in water, $MgSO_4$ is added and then boiled, on cooling, a white precipitate is formed confirming the presence of CO_3^{2-}, if a white precipitate is formed after boiling, HCO_3^- is confirmed.

Confirmatory Test for Cations

Table 16: A Sample Presentation of Results

Cation	Procedure	Observation/inferences
Cu^{2+}	(a) To a sample solution, ammonia solution is added in excess. (b) To a sample solution, Potassium Ferrocyanide solution is added.	• Pale blue precipitate is formed soluble in excess ammonia to form deep blue solution. Cu^{2+} present and confirmed. • Reddish brown gelatinous precipitate is formed soluble in aqueous ammonia but insoluble in aqueous mineral acids. Cu^{2+} is present and confirmed.
NH_4^+	Add NaOH solution to a small amount of solid sample and warm it, pass moist litmus paper to the mouth of the test tube. 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^+ present and confirmed.
Zn^{2+}	(a) Add a small amount of $NaOH/NH_4OH$ solution. (b) Add two or three drops of 0.1 M Potassiumhexacyanoferrate(II) $K_4[Fe(CN)_6]$ and stir.	• White precipitate soluble in excess NaOH is formed. Zn^{2+} present and confirmed. • Bluish white precipitate is formed. Zn^{2+} present and confirmed.
Ca^{2+}	(a) Add ten drops of 2 M aqueous ammonia to a test solution. (b) Test the acidity of the mixture	• White precipitate is formed.

Cation	Procedure	Observation/inferences
	using the litmus paper. (c) Continue adding ammonia until the mixture is basic. (d) Add about ten drops of 0.2 M ammonium oxalate solution to the mixture.	- Ca^{2+} present and confirmed.
Fe^{2+}	Add a few drops of 0.1 M potassium ferricyanide solution.	- Dark blue precipitate is formed. - Fe^{2+} is confirmed.
Fe^{3+}	Add four drops of potassium thiocyanate solution.	- Deep blood-red precipitate is formed. - Fe^{3+} is confirmed.
Pb^{2+}	(a) Add small amount of 0.1 M potassium chromate (K_2CrO_4) solution. (b) Add a small amount of KI solution to the solution of the sample. Warm and cool the mixture.	- Yellow precipitate is formed. - Pb^{2+} ion is confirmed. - Yellow precipitate is formed, which disappears on warming and reappears on cooling. - Pb^{2+} present and confirmed.

3.4 Two Component Liquid Systems

If a substance is soluble in two liquids which are completely immiscible, it will distribute itself between the two liquids in such a way that the ratio of its concentration in the two liquids is constant. The distribution coefficient of the substance between two immiscible liquids is then calculated using the following relationship:

$$\text{Distribution coefficient} = \frac{\text{Concentration of a solute in aqueous layer}}{\text{Concentration of a solute in organic layer}}$$

For example, ammonia distributes itself between water and isobutyl alcohol until the ratio of concentration of NH_3 becomes constant.

$$\text{Distribution coefficient} = \frac{\text{Concentration of } NH_3 \text{ in aqueous layer}}{\text{Concentration of } NH_3 \text{ in isobutyl alcohol}}$$

The Specific Objective

The teacher, the tutor and the laboratory technician should be able to prepare a practical for candidates to determine the distribution coefficient of a solute when dissolved in a mixture of two immiscible liquids.

Key issues

(i) Apparatuses Needed by the Teacher, the Tutor and the Laboratory Technician

- A 500 cm^3 beaker
- A digital or chemical balance
- A stirring rod
- A filter funnel
- A volumetric flask (1000 cm^3 or 2000 cm^3)

(ii) Apparatuses needed by a candidate

- A separating funnel (250 or 300 cm^3)
- A burette (50 cm^3)
- A filter funnel
- 2 Conical flasks (250 cm^3)
- A white tile
- A retort stand and its accessories
- A pipette (10 or 20 or 25 cm^3)
- A pipette filler

(iii) Chemicals

- Ammonia solution
- Isobutanol or Chloroform
- Hydrochloric acid
- Indicator solution (POP or MO)

Note

(i) Organic solvents are used in the form they are bought.

(ii) Each candidate should be given a pipette filler. Sucking liquid chemicals with mouth is strongly discouraged.

The Preparation of Solutions

The concentration of solutions will be given in the practical advance instructions.

The Procedure

For example: If you are instructed to prepare 0.5 M NH_3 and 0.05 M HCl solutions, follow the procedure presented in section 3.1.1.1

A Sample Question

=You are provided with the following:
=AA: A solution of 0.5 M NH_4OH;
=BB: A solution of 0.1 M HCl;
=CC: Isobutyl alcohol; and
=DD: Methyl Orange (MO) indicator.

Procedure

(i) Using a measuring cylinder, measure 50 cm^3 of CC and transfer it into a separating funnel.
(ii) Using a measuring cylinder, measure 50 cm^3 of AA and pour it into a separating funnel containing CC.
(iii) Close the separating funnel and shake the mixture for about two minutes while relieving the pressure in the separating funnel by removing a stopper at interval of about 30 seconds. Remove the stopper and leave the mixture to settle.
(iv) Run the lower aqueous layer into a beaker.
(v) Pipette 20 or 25 cm^3 of this aqueous layer into a conical flask. Add two drops of MO indicator and note the colour of the mixture.
(vi) Titrate the aqueous layer in the flask against solution BB from the burette until there is a colour change. Note the volume of the acid used.
(vii) Repeat the titration experiment three times and record your results in a tabular form

The Assessment

Candidates will be assessed based on their ability to fill in a table of results, the accuracy of their practical data, the observation of 2 decimal points as well as the manipulation of experimental data to determine the distribution coefficient.

4.0 CONCLUSION

This guidelines present strategies to be followed for effective and efficient preparation of practical examinations. They emphasize to the users the importance of keeping the chemistry laboratory organized all the time as well as keeping records of the laboratory inventories. Proper keeping of the laboratory records will not only help to greatly cut the cost of buying chemicals and apparatuses which are already present in the laboratory, but also ensure economic use of chemicals. Moreover, the guidelines covered various preparation techniques to reduce the time of preparing practical examinations. The guidelines have further addressed common challenges facing teachers, tutors and laboratory technicians during preparation of practical examinations. Thus, it is expected that practical examinations will be prepared at acceptable standards and within the given time frame if these guidelines are fully followed.