16 LAB SAFETY AND PRACTICAL SKILLS

Student Learning Outcomes [C-11-F-01 to C-11-F-21]

After studying this chapter, students will be able to:

- Identify the chemical hazards in the lab in the context of the experiment being conducted. **(Knowledge)**
- Test that the equipment is working properly without any potential risk of injury before conducting an experiment. **(Knowledge)**
- Ensure that work space for conducting the experiment is not crowded with apparatus as to be hazardous. **(Knowledge)**
- Ensure that safe distance space is kept at all times from other investigators who may handling lab apparatus. **(Knowledge)**
- Identify what bodily harm could occur from physical, chemical, biological and safety hazards in context of the experiment being conducted. **(Knowledge)**
- Recognize that it is always better to ask for help from the lab instructor when unaware of how to use new apparatus. **(Knowledge)**
- Identify the proper waste disposal system for chemicals being used. **(Knowledge)**
- Set up apparatus following instructions given in written or diagrammatic form. **(Understanding)**
- Use apparatus to collect an appropriate quantity of data. **(Understanding)**
- Make observations including subtle differences in colour, solubility or quantity of materials. **(Understanding)**
- Make measurements using pipettes, burettes, measuring cylinders, thermometers, and other common laboratory apparatus. **(Understanding)**
- Decide how many tests or observations to perform. **(Understanding)**
- Identify where repeated readings or observations are appropriate. **(Understanding)**
- Replicate readings or observations as necessary, including where an anomaly is suspected. **(Understanding)**
- Identify where confirmatory tests are appropriate and the nature of such tests. **(Understanding)**
- Select reagents to distinguish between given ions. **(Knowledge)**
- Carry out procedures using simple apparatus in situations where the method may not be familiar to the candidate. **(Application)**
- Describe acid base titration to include the use of a burette, volumetric pipette and suitable indicator. **(Understanding)**
- Describe how identify the end point of a titration using an indicator. **(Understanding)**
- Describe tests to identify the anions: (a) CO_3^{2-}, (b) Cl^{1-}, Br^{1-} and I^{1-} (c) NO_3^{1-}(d) SO_4^{2-} (e) SO_3^{2-} **(Understanding)**
- Describe tests using aqueous NaOH and aqueous NH_3 to identify the aqueous cations: (a) Al^{3+}(b) NH_4^+(c) Ca^{2+} (d) Cr^{3+} (e) Cu^{2+} (f) Fe^{2+}(g) Fe^{3+} and (h) Zn^{2+} **(Understanding)**

A chemistry laboratory is a chemist workshop. It is a place where a student is trained to observe the physical and chemical characteristics of substances by following definite procedures. Before starting the laboratory work, a student should get himself familiarized with the layout of the laboratory and various fittings provided on the laboratory table as well as the side shelves.

16.1. GENERAL INSTRUCTIONS TO THE STUDENTS

i. Students are expected to conduct themselves in a responsible manner at all times in the lab.

ii. They are advised not to work alone in the lab. Experiments should be performed in the presence of lab instructor and other laboratory staff.

iii. Students should always wear lab coat and safety goggles while working in the lab. Girls must tie up their scarves and hair before start working in the lab.

iv. Determine the potential hazards related to any equipment or the experiment before starting any work. Appropriate safety precautions must be observed.

v. There must not be any crowding in the lab and students should stick to their work places at a safe distance from each other.

vi. Don't bring any food items in the lab. Never taste or smell any compound or a gas.

vii. Any accident or breakage of glassware must be reported to the incharge of the laboratory immediately.

viii. If you cannot handle an instrument or an equipment properly then you must seek help from the instructor.

ix. Do not pour chemicals down the drains and do not utilize the sewer for chemical waste disposal.

x. Follow the warning sign displayed in the lab.

16.2 COMMON TYPES OF HAZARDS IN A LABORATORY

Most hazards which we might face while working in the laboratory fall into three categories, physical, chemical and biological hazards.

16.2.1 Physical Hazards

The most common physical hazards are slips and falls when working on wet floor. A worker must take all types of precautions to avoid accidents related to physical hazards such as slipping, pulling, falling etc.

A worker should wear cut-resistant gloves while handling the broken glassware to prevent cuts, abrasions and skin damage. It should be mandatory for the laboratory staff to dispose off broken glassware in special container to prevent injury.

16.2.2 Chemical Hazards

A laboratory worker must use the chemicals according to the standard procedures keeping in view the particular hazards and precautions required for the safe use.

Figure 16.1 Pictogram of chemical hazards

16.2.3 Biological Hazards

The most common biological hazards are allergens, microbes including virus and bacteria. These are transferred from animals, plants, water and air to human.

16.3 WASTE DISPOSAL SYSTEM FOR CHEMICALS

Chemical waste cannot be disposed off in bins or sewer system. Most chemical wastes must be disposed off keeping in view the following rules and regulation of Environmental Protection Agency (EPA):

1. Store chemical wastes in proper containers.
2. Label the chemical waste containers with the types of wastes, the date of waste and place of origin.
3. These containers are then transferred to the allocated site where these are appropriately treated to dispose them off.
4. Chemical treatment of wastes involves neutralization, precipitation, ion exchange, oxidation or reduction.

16.4 FIRST AID IN LABORATORY

Every laboratory must have a first aid box. Common accidents and their first aid treatments are given in the **Table 16.1** below:

Table 16.1 Accidents and their First Aid treatment

	Type of Accident	First Aid Treatment
1	**Cuts**	
	i. Minor cuts	i. Remove the glass piece if any, apply a little methylated spirit or tincture iodine with a piece of cotton. Both act as disinfectant.
	ii. Serious cuts	ii. Apply pressure on the cut for about 10 minutes to stop bleeding. Consult a doctor.
2	**Eye Injuries**	
	i. Acid in the eye	i. Wash thoroughly with water and then with 1% sodium bicarbonate solution.
	ii. Alkali in the eye	ii. Wash with water followed by 1% boric acid solution.
	iii. Foreign particle in the eye	iii. Do not rub the eyes. Remove the particle carefully with soft handkerchief then wash with water.
	iv. Soreness in the eye	iv. Put a drop of olive oil in the eyes and keep them closed for some time.
3	**Burns**	
	i. Burns with dry heat (flame, hot object)	i. Apply burnol or mustard oil.
	ii. Acid burns	ii. Wash freely with ice cold water. Then wash with a saturated solution of sodium bicarbonate and again with water.
	iii. Alkali burns	iii. Wash freely with water and then with 1% acetic acid solution and again with water, dry the skin and apply the burnol.
	iv. Bromine burns	iv. Wash fully with 2% ammonia solution and apply glycerin. Wipe off glycerin after sometime and apply burnol.
4	**Poisons**	
	i. Poisons swallowed	i. Spit immediately, wash mouth with water repeatedly.
	ii. Acid swallowed	ii. Drink a lot of water or lime water or milk of magnesia.
	iii. Caustic alkalies swallowed	iii. Drink a lot of water; drink a glass of lemon or orange juice.
	iv. Salt of heavy metals swallowed.	iv. Take milk or white part of the egg.

5	**Fire**	
	i. Clothes catch fire	i. Do not run. Wrap with blanket or with dry cotton cloth. Lie down on the floor.
	ii. Beaker containing inflammable liquid. catches fire.	ii. Cover the beaker with a duster or damp cloth. This will cut off the supply of oxygen.
	iii. Spirit or oil catches fire.	iii. Throw a mixture of sand and sodium bicarbonate. Do not throw water. It will simply spread the fire.
	iv. Electric parts catch fire.	iv. Switch off the electric supply immediately and throw sand. Do not throw water in such cases to extinguish fire.

Quick Check 16.1

a) Mention common types of hazards.

b) How chemical waste is disposed off?

c) How H_2SO_4 burn is treated in the lab?

d) What are different types of burns?

16.5 ACID-BASE TITRATION

Volumetric analysis is used to find the concentrations of solutions by means of a technique known as titration. In this technique a solution of unknown concentration is combined slowly and carefully with a known volume of a standard solution until a colour change shows the completion of the reaction. The substance which indicates the completion of reaction by the change in its colour is called an **indicator**. The moment at which the indicator changes colour is called the **end point**. Either acidic or basic solution can be taken in a burette with the other solution taken in a conical or a titration flask.

Did you know?

Phenolphthalein solution is prepared by adding one gram of the indicator in 500 cm^3 of 50% ethanol. Use only one to two drops in 10 cm^3 of the solution to be titrated. The color change is from pink to colorless as the pH decreases. The light pink color which marks the end point tends to fade gradually due to the interference of atmospheric carbon dioxide which slowly dissolves in the solution.

Materials Required

Burette, pipette, funnel, conical flask, HCI solution, NaOH solution, phenolphthalein.

Procedure of Titration

i. Rinse the pipette first with distilled water and then with the given NaOH solution.
ii. Rinse the conical flask with distilled water only.
iii. Pipette out 10 cm^3 of NaOH solution into a conical flask.
iv. Add one to two drops of phenolphthalein indicator into it. The solution turns pink.
v. Rinse the burette first with distilled water and then with the given HCl solution.

vi. Fix the burette on a clamp stand in an upright position.
vii. Fill it with the given HCl solution with the help of a funnel. Remove funnel from the burette.
viii. Using the tap at the base of the burette, allow the acid to flow into a beaker to remove any air bubble present in the nozzle.
ix. Note the burette reading as an initial reading using an anti-parallax card or a white paper.
x. During titration, place the conical flask on a white paper under the burette to see the color change of the indicator clearly. Check that the burette does not leak.
xi. Carry out a rough titration by adding hydrochloric acid solution from the burette drop wise to the conical flask.
xii. The contents of the flask must be swirled for thorough mixing.
xiii. Keep on adding acid till the colour of solution becomes light pink persistently. This will be the end point of the reaction.

Again, note down the burette reading, this will be the final reading. The difference of final and initial readings of burette will give the volume of the acid used.

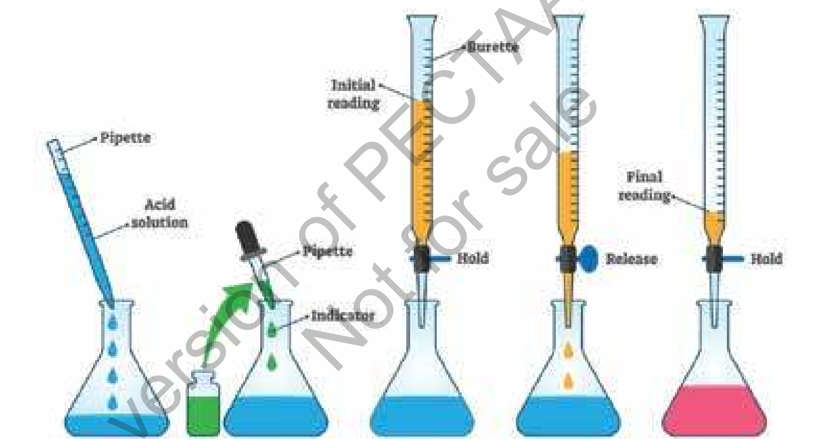


Figure 16.2 Procedure of titration

Repeat the titration to get accurate and concordant readings. Take at least three concordant readings which agree with one another within 0.1 cm^3.

Observations

Sr. No.	Initial reading (a)	Final reading (b)	Volume of acid used (cm^3) (b - a)
1			
2			
3			

Volume of HCl solution used = ________cm^3

Chemical Equation

$$NaOH_{(aq)} + HCl_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)}$$

Calculation

Find out the molarity of NaOH solution by using the following molarity equation:

$$\begin{array}{ccc} Acid & & Base \\ \dfrac{M_1V_1}{n_1} & = & \dfrac{M_2V_2}{n_2} \end{array}$$

M_1 = Molarity of acid solution
V_1 = Volume of the acid used
n_1 = No. of moles of the acid in the balanced chemical equation
M_2 = Molarity of base solution
V_2 = Volume of base solution used
n_2 = No. of moles of the base in the balanced chemical equation

$$M_2 = \frac{M_1V_1}{n_1} \times \frac{n_2}{V_2}$$

Molarity of the given base solution is ____M.

Quick Check 16.2

a) Define indicator and endpoint in a titration.
b) What is meant by concordant reading?
c) How the volume of the titrant used is calculated?

16.6 TESTS FOR IDENTIFICATION OF ANIONS

a) Identification of carbonate CO_3^{2-} radical

Experiment	Observation	Inference
Take about 2 g of solid carbonate sample in a clean test tube and then add about 5 cm^3 of dilute HCl solution. Pass the gas evolved in the above step through a solution of lime water.	Effervescence takes place during which a gas evolves briskly, that turns lime water turns milky.	Carbonate (CO_3^{2-}) is indicated.

Reactions involved in the above steps

$$CO^{2-}_{3(aq)} + 2H^+_{(aq)} \rightarrow H_2O_{(\ell)} + CO_{2(g)}$$

$$\underset{\text{Lime water}}{Ca(OH)_{2(aq)}} + CO_{2(g)} \rightarrow \underset{\text{(white ppt)}}{CaCO_{3(s)}} + H_2O_{(l)}$$

(b) Identification of chloride (Cl^{1-}), Bromide (Br^{1-}) and Iodide (I^{1-}) radicals

Experiment	Observation	Inference
Take 2 g of solid sample in a test tube and add 5 cm^3 of distilled water to make the aqueous solution. Add a few drops of dil. HNO_3 solution to acidify the solution of the salt, finally add about 5 cm^3 of aqueous $AgNO_3$.	A thick white precipitate is formed which dissolves in aq.NH_3.	Cl^{-1} indicated.
	A thick cream-yellow precipitate is formed.	Br^{1-} indicated.
	A bright yellow precipitate is formed.	I^{1-} indicated.

Reactions involved in the above tests

$$Ag^+_{(aq)} + Cl^-_{(aq)} \rightarrow \underset{\text{white ppt}}{AgCl_{(s)}}$$

$$Ag^+_{(aq)} + Br^-_{(aq)} \rightarrow \underset{\text{cream yellow ppt}}{AgBr_{(s)}}$$

$$Ag^+_{(aq)} + I^-_{(aq)} \rightarrow \underset{\text{bright yellow ppt}}{AgI_{(s)}}$$

Did you know?

In the above tests for the detection of halide ions, dilute nitric acid is added to prevent the precipitation of carbonate ions along-with halide ions.

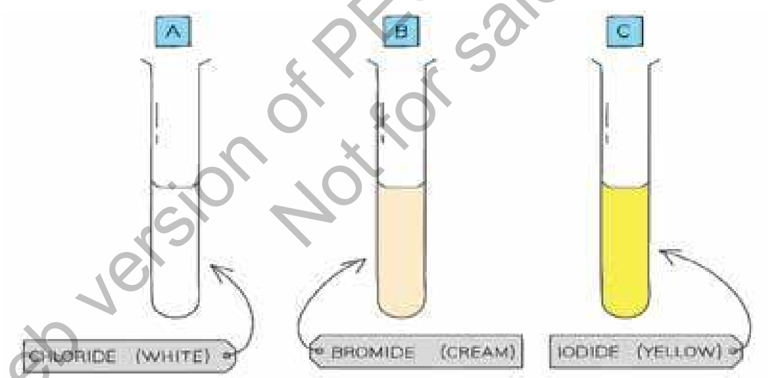
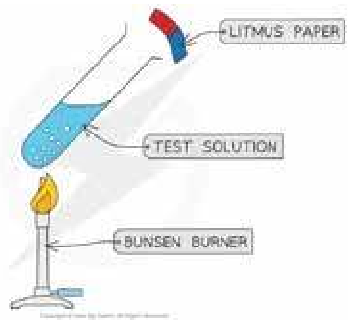


Figure 16.3 Observation of reactions of halide ions with $AgNO_3$

c) Identification of Nitrate (NO_3^{1-}) radical

Experiment	Observation	Inference
Take 2 g of solid sample in a clean test tube and dissolve it in 5 cm^3 distilled water. Add to it 5 cm^3 of sodium hydroxide solution. Finally add 3 g of powdered aluminium metal.	A characteristic smell of NH_3 gas is felt near the mouth of test tube. This gas turns red litmus paper blue.	NO_3^{1-} indicated.

Aluminium metal reduces nitrate ions to ammonium ions which then react with aqueous NaOH to evolve ammonia gas.



$$3NO_{3(aq)}^{1-} + 8Al_{(s)} + 5\bar{O}H_{(aq)} + 18H_2O_{(\ell)} \rightarrow 3NH_{3(g)} + 8\left[Al(OH)_{4(aq)}\right]^{1-}$$

(d) Identification of Sulphate (SO_4^{2-}) radical

Experiment	Observation	Inference
Take 3 g of solid sample in a clean test tube. Dissolve it in 5 cm^3 of distilled water. Acidify the solution with a few drops of dil. HNO_3 and then add 5 cm^3 barium nitrate solution.	A heavy white precipitate of $BaSO_4$ is formed.	SO_4^{2-} indicated

$$SO_{4(aq)}^{2-} + Ba_{(aq)}^{2+} \longrightarrow \underset{\text{white ppt}}{BaSO_{4(s)}}$$

Did you know?

Dilute nitric acid is added to destroy any carbonate ions present in the solution as an impurity.

Quick Check 16.3

a) Distinguish carbonate (CO_3^{2-}) and bicarbonate (HCO_3^{1-}) radicals.
b) What is ring test? Give equation for this test.
c) Give names and formulas of some water insoluble sulphates.

16.7 TESTS FOR IDENTIFICATION OF BASIC RADICALS

a) Identification of Aluminium (Al^{3+}) radical

Take 4 g of solid sample in a clean test tube. Dissolve it in about 10 cm^3 distilled water. Divide this solution into two parts for further tests.

Experiment	Observation	Inference
To one part, add about 5 cm^3 NaOH solution.	A white gelatinous precipitate is formed.	Al^{3+} indicated.

To the second part of the above solution, add a few drops of aqueous ammonia.	A white precipitate is formed.	Al^{3+} indicated

$$Al^{3+}_{(aq)} + 3OH^{1-}_{(aq)} \rightarrow \underset{\text{white ppt}}{Al(OH)_{3(s)}}$$

b) Identification of Ammonium (NH_4^{1+}) radical

Experiment	Observation	Inference
Take 4 g of sample in a clean test tube. Dissolve it in 10 cm^3 distilled water. Add NaOH solution and gently heat it.	Ammonia gas is evolved with a distinct smell. Ammonia gas turns moist red litmus blue.	NH_4^{1+} indicated.

$$NH^{1+}_{4(aq)} + OH^{1-}_{(aq)} \rightarrow NH_{3(g)} + H_2O_{(\ell)}$$

c) Identification of Calcium (Ca^{2+}) radical

Take 4 g solid sample in a clean test tube. Dissolve it in 10 cm^3 distilled water.

Make two parts of this solution for further tests.

Experiment	Observation	Inference
To one part, add NaOH solution.	White precipitate is formed which does not dissolve in excess of NaOH solution.	Ca^{2+} indicated.
To the second part of the above solution, add aqueous ammonia.	Only a slight turbidity appears or No precipitate is formed.	Ca^{2+} indicated.

$$Ca^{2+}_{(aq)} + 2OH^{-}_{(aq)} \rightarrow \underset{\text{white ppt}}{Ca(OH)_{2(s)}}$$

d) Identification of Chromium radical (Cr^{3+})

Add 4 g sample in a clean test tube. Dissolve it in 10 cm^3 distilled water. Make two parts of the above solution for further tests.

Experiment	Observation	Inference
To one part, add NaOH solution.	A green precipitate appears which turns into green solution when excess of NaOH is added	Cr^{3+} indicated.

To the second part of the above solution, add aqueous ammonia.	Green precipitate appears which is insoluble in excess of aqueous ammonia.	Cr^{3+} indicated.

$$Cr^{3+}_{(aq)} + 3OH^{-}_{(aq)} \rightarrow \underset{\text{Green ppt}}{Cr(OH)_{3(s)}}$$

e) Identification of copper radical (Cu^{2+})

Add 4g solid sample in a clean test tube. Dissolve it in 10cm^3 of distilled water. Make two parts of the above solution for further tests.

Experiment	Observation	Inference
To first part, add NaOH solution.	Light-blue precipitate is formed.	Cu^{2+} indicated.
To the second part of the above solution, add aqueous ammonia.	Deep blue solution is formed.	Cu^{2+} indicated.

$$Cu^{2+}_{(aq)} + 2OH^{-}_{(aq)} \rightarrow \underset{\text{Blue ppt}}{Cu(OH)_{2(s)}}$$

f) Identification of iron (II) radical (Fe^{2+})

Take 4 g sample in a clean test tube. Dissolve it in 10 cm^3 of distilled water. Make two portions of the solution for further tests.

Experiment	Observation	Inference
To one portion, add NaOH solution.	Green precipitate is formed which is turned into orange brown precipitate after some time.	Fe^{2+} indicated.
To second portion of the above solution, add aqueous ammonia.	White gelatinous precipitate of $Fe(OH)_2$ is formed which quickly oxidizes to form red brown precipitate of $Fe(OH)_3$.	Fe^{2+} indicated.

$$Fe^{2+}_{(aq)} + 2OH^{-}_{(aq)} \rightarrow \underset{\text{Green ppt}}{Fe(OH)_2}$$

g) Identification of Fe(III) radical (Fe^{3+})

Take 2 g $FeCl_3$ in a clean test tube. Dissolve it in 10 cm^3 of distilled water. Make two portions of the solution for further tests.

Experiment	Observation	Inference
To one portion, add NaOH solution.	Orange brown precipitate is formed.	Fe^{3+} indicated.
To the second portion of the above solution, add aqueous ammonia.	Orange brown precipitate is formed.	Fe^{3+} indicated.

$$Fe^{3+}_{(aq)} + 3OH^{-}_{(aq)} \rightarrow \underset{\text{Orange brown ppt}}{Fe(OH)_{3(s)}}$$

h) Identification of Zinc radical (Zn^{2+})

Take 4 g solid sample in a clean test tube. Dissolve it in 10 cm^3 of distilled water. Make two portions of the above solution for further tests.

Experiment	Observation	Inference
To one portion add NaOH solution.	White precipitate is formed which is soluble in excess of NaOH solution.	Zn^{2+} indicated.
To second portion add aqueous ammonia.	White precipitate is formed which dissolves in excess of ammonia.	Zn^{2+} indicated.

$$Zn^{2+}_{(aq)} + 2OH^{-}_{(aq)} \rightarrow \underset{\text{white ppt}}{Zn(OH)_{2(s)}}$$

Quick Check 16.4

a) What is lake test? Give equation.

b) How ammonium ion is indicated?

c) How will you distinguish ferrous (Fe^{2+}) and ferric (Fe^{3+}) radicals?

d) What happens when aq. NH_3 is added to Cu^{2+} solution in limited and excess quantities?

EXERCISE

MULTIPLE CHOICE QUESTIONS

Q.1 Four choices are given for each question. Select the correct choice.

I. While taking a reading with a burette, why it is always advisable to read the lower meniscus for the colorless liquids and the upper meniscus for the coloured liquids?

a) because it is more convenient
b) because colorless liquids have more surface tension than colored liquids
c) because lower meniscus does not exist for colored liquids
d) because of the parallax effect

II. Why phenolphthalein indicator is more appropriate to use during the titrations which involve a strong acid and a strong base?

a) because it is itself weakly acidic
b) because the pH at the equivalence point as well as the pH over where the colour of phenolphthalein changes match each-other.
c) because the solution at the end of titration is acidic.
d) because the solution at the end of titration is basic.

III. Which cation gives a white gelatinous precipitate upon the addition of aqueous ammonia?

a) Cr^{3+} b) Cr^{2+}
c) Zn^{2+} d) Al^{3+}

IV. Addition of NH_4OH to an aqueous solution of a cation gives a green precipitate which turns brown upon standing. Which basic radical is indicated?

a) Cu^{2+} b) Cr^{3+}
c) Fe^{2+} d) Fe^{3+}

V. On dry heating test for salt analysis, the evolution of a colorless, odorless gas that turns lime water milky suggests the presence of:

a) Chloride ion (Cl^-) b) Sulfate ion (SO_4^{2-})
c) Carbonate ion (CO_3^{2-}) d) Nitrate ion (NO_3^-)

VI. The chromyl chloride test is a specific confirmatory test for:

a) Bromide ions (Br^-) b) Iodide ions (I^-)
c) Chloride ions (Cl^-) d) Sulfate ions (SO_4^{2-})

VII. The brown ring test is a confirmatory test for which acid radical?

a) Chloride (Cl^-)
b) Nitrate (NO_3^-)
c) Sulfate (SO_4^{2-})
d) Carbonate (CO_3^{2-})

SHORT ANSWER QUESTIONS

Q.2 Attempt the following short-answer questions:

a. For which type of titration, methyl orange is used as an indicator?

b. Explain why phenolphthalein is a suitable indicator for the titration of a weak acid with a strong base but not for the titration of a strong acid with a weak base.

c. Explain why different indicators change color over different pH ranges.

d. It is always advisable to use dilute solutions while performing experiments in volumetric analysis? Give a reason.

e. White precipitates are formed when Ca^{2+}, Al^{3+} and Zn^{2+} all react separately with NaOH solution. How will you detect which basic radical is present?

f. How Fe^{2+} can be distinguished from Fe^{3+} chemically?

g. Why does Ca^{2+} not give precipitate with aqueous ammonia?

h. How will you find out the concentration of acetic acid in vinegar solution?

i. What precautions you need to observe while diluting a concentrated acid?

j. Why does an aqueous solution of Na_2CO_3 behave like a base?

k. If an aqueous solution of NaOH is kept in an open container, what changes do you expect to take place with the passage of time?

DESCRIPTIVE QUESTIONS

Q.3 Describe common types of the Chemistry lab hazards with two examples in each case.

Q.4 What are common accidents in the Chemistry lab? How they are managed in first aid treatment.

Q.5 How the following acid radicals are indicated and confirmed in salt analysis:

i) CO_3^{2-} ii) Cl^- iii) NO_3^- iv) SO_4^{2-}

Q.6 How the following basic radicals are indicated and confirmed in salt analysis:

i) Cu^{2+} ii) Al^{3+} iii) Fe^{3+} iv) Zn^{2+}