

**UNIT : 3**

**edexcel**   
advancing learning. changing lives

# LABORATORY CHEMISTRY

## PRACTICAL SKILLS I



  
**Chembase**  
School of Chemistry  
— Since 2005 —

<b>First Edition</b>	<b>Jan 2006</b>
<b>Second Edition</b>	<b>Jun 2009</b>
<b>Third Edition</b>	<b>Jun 2011</b>
<b>Fourth Edition</b>	<b>Jan 2019 (Revised New Syllabus)</b>

**All Rights Reserved.**

Unauthorized duplication contravenes applicable laws.

No part of this publication may be reproduced or utilized in any form or by electronic, mechanical, or other means, now known or hereafter invented including photocopying & recording, without the prior permission in writing from the copyright owner.

**Published by:**



**Copyright owner:**

**Imran Razeek** *I.Chem.C (PI), M.Ed, Ph.D (Reading)*  
*Lecturer in Chemistry cum Chairman*

**Chembase - School of Chemistry,**

 **34, 1/2, Galle Road, Dehiwela, Sri Lanka.**

 **(+94) 776 534 233 / (+94) 776 136 047**

 **www.chembase.lk**

 **info@chembase.lk**

**CAMBRIDGE**  
International Examinations

**WizIQ**  
education.online

**edexcel**  
advancing learning, changing lives

## Common Lab safety Symbols



**Irritant**  
Substances that can make your skin go red or blister – if they are dry powders, they can cause coughing  
*Examples:* copper carbonate, calcium chloride



**Highly inflammable**  
Substances that catch fire easily  
*Examples:* ethanol, hexane



**Oxidising**  
Substances that help others burn more strongly  
*Examples:* ammonium nitrate, potassium manganate(VII)



**Toxic**  
Substances that are poisonous and can kill you  
*Examples:* chlorine, methanol



**Harmful**  
Substances that may cause pain and discomfort  
*Examples:* copper sulphate, barium chloride



**Corrosive**  
Substances that will burn the skin and damage the eyes – they can damage wood and metal  
*Examples:* sulphuric acid, sodium hydroxide



**Harmful to the Environment**  
*Eg :* Potassium dichromate, potassium permanganate, silver nitrate, ammonia

## Common laboratory Glassware



Test tube



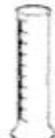
Boiling tube



Beaker



Conical flask



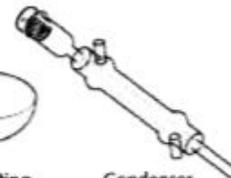
Measuring cylinder



Graduated beaker



Evaporating dish



Condenser



Bunsen burner



Tripod



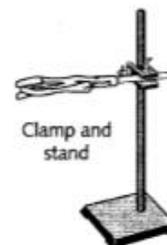
Gauze



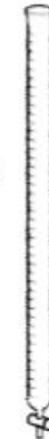
Filter funnel



Eye protection



Clamp and stand



Burette



Syringe



Dropping pipette



Stopclock



Balance



Mortar and pestle



Thermometer



Pipette



Spatula



Round bottom flask



Pear shaped flask



Separating funnel



Still head



Receiver adaptor



Volumetric / graduated flask



Bulb pipette



Pipette filler



Crucible with lid



Pipe clay triangle



Tongs

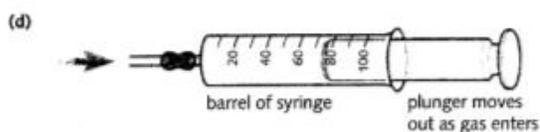
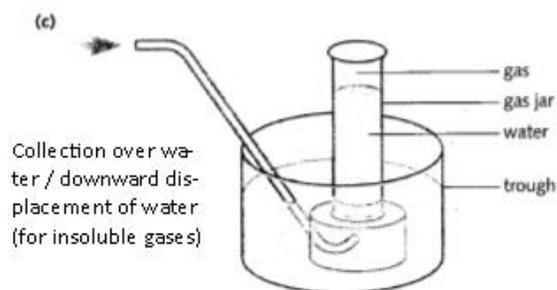
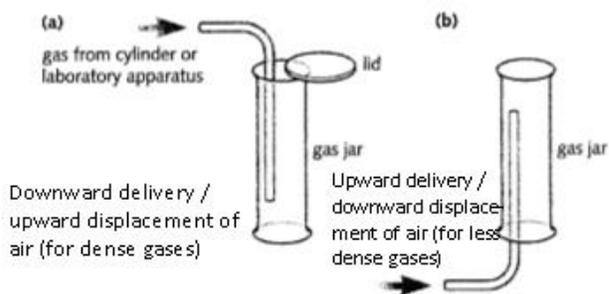


Test tube holder

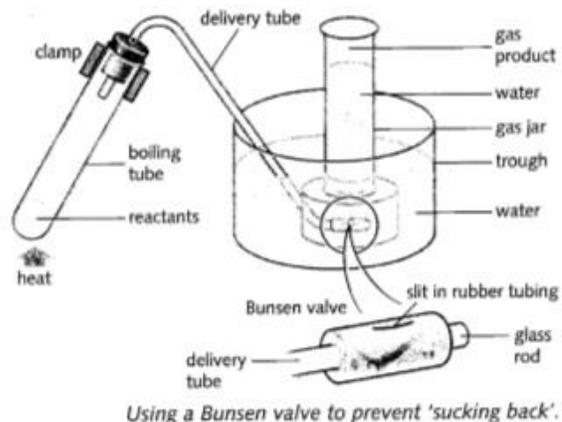


Graduated pipette

## Methods of collecting gases

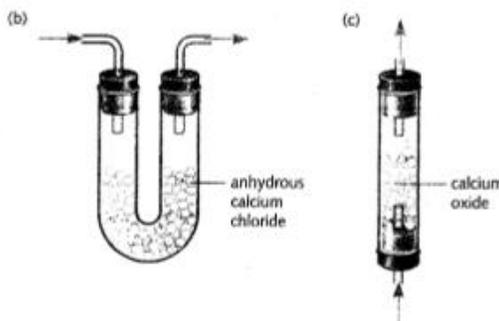
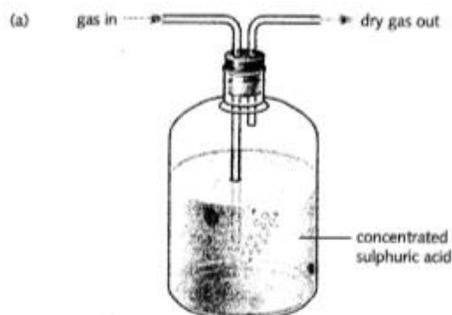


The different methods for collecting gases.



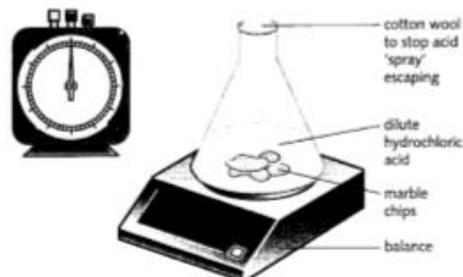
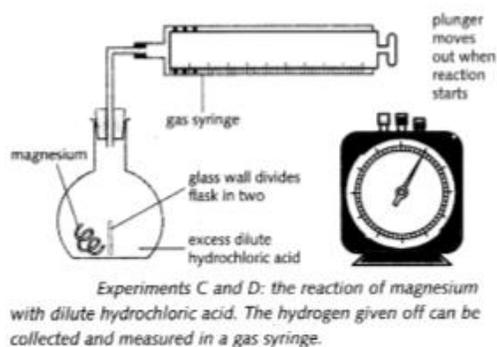
Making a solution of a very soluble gas.

## Methods of drying a gas

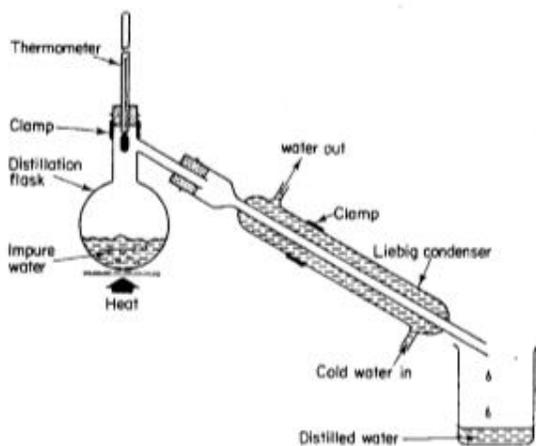


The different methods of drying gases.

## Methods of determining the rate of a reaction

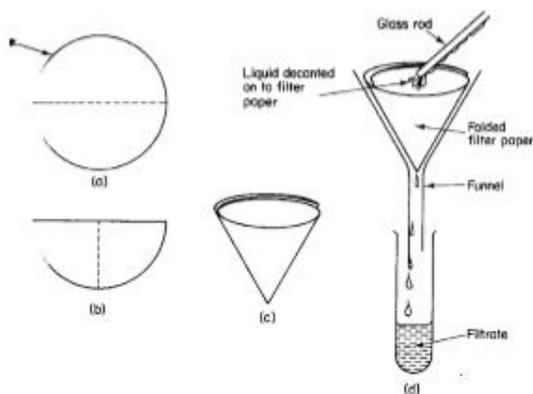
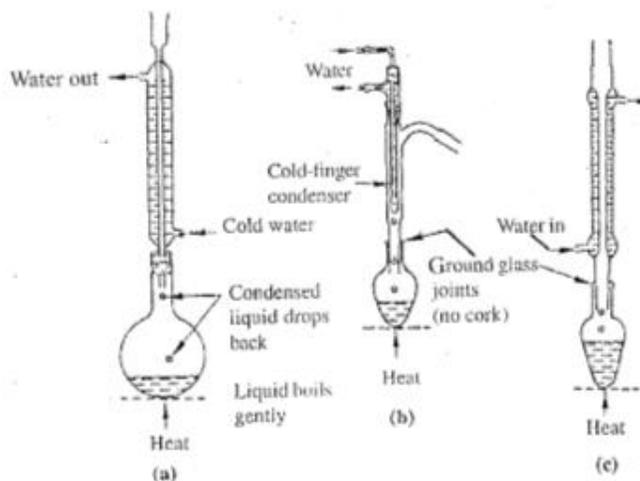


Experiments A and B: the reaction of marble chips with dilute hydrochloric acid. The loss of carbon dioxide from the flask produces a loss in mass. This is detected by the balance.

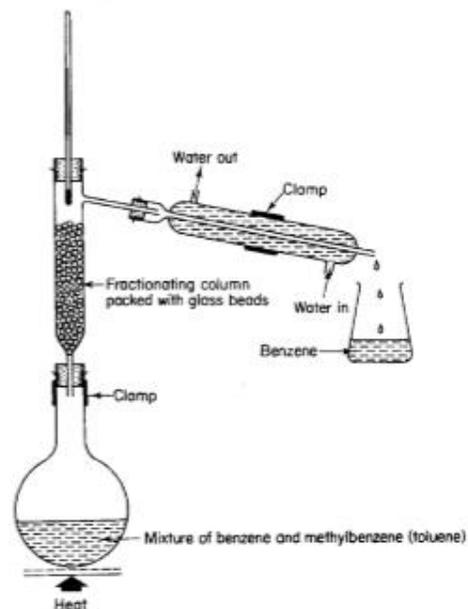


Laboratory apparatus for distillation

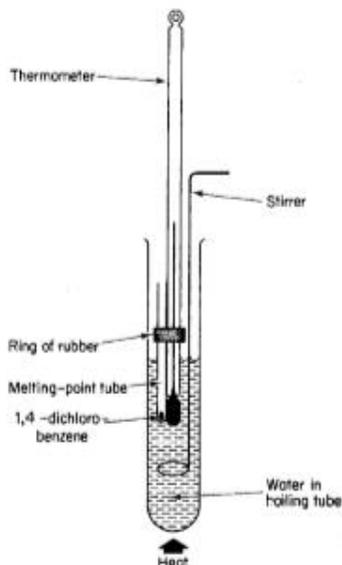
Heating under a reflux condenser



Filtration in the laboratory, using a funnel and filter paper



Separation of a benzene/methylbenzene (toluene) mixture fractional distillation



Apparatus used for the determination of melting points

- 1 The mixture of immiscible liquids settles into two layers, as the liquids do not mix
- 2 The tap is opened to let only the bottom layer run into the beaker
- 3 The tap is closed and the beaker is changed. The tap is opened to let the top layer run out



A separating funnel can be used to separate two immiscible liquids.

## 1. Test for aqueous positive ions (cations)

Positive ion (in solution)	Effect of adding sodium hydroxide	Effect of adding Ammonia solution
<b>Ammonium</b> (NH <sub>4</sub> <sup>+</sup> )	Ammonia gas produced on warming	-----
<b>Copper (II)</b> (Cu <sup>2+</sup> )	light blue precipitate of Cu <sup>2+</sup> (aq) + 2OH <sup>-</sup> (aq) → Cu(OH) <sub>2</sub> (s) Insoluble in excess NaOH	Blue precipitate with is soluble in excess ammonia solution resulting dark blue solution
<b>Iron (II)</b> (Fe <sup>2+</sup> )	Green precipitate Fe <sup>2+</sup> (aq) + 2OH <sup>-</sup> (aq) → Fe(OH) <sub>2</sub> (s) Insoluble in excess NaOH	Green precipitate Insoluble in excess
<b>Iron (III)</b> (Fe <sup>3+</sup> )	Red-brown precipitate Fe <sup>3+</sup> (aq) + 3OH <sup>-</sup> (aq) → Fe(OH) <sub>3</sub> (s) Insoluble in excess NaOH	Red-brown precipitate Insoluble in excess
<b>Calcium</b> (Ca <sup>2+</sup> )	White precipitate Ca <sup>2+</sup> (aq) + 2OH <sup>-</sup> (aq) → Ca(OH) <sub>2</sub> (s) Insoluble in excess NaOH	No precipitate
<b>Magnesium</b> (Mg <sup>2+</sup> )	White precipitate Mg <sup>2+</sup> (aq) + 2OH <sup>-</sup> (aq) → Mg(OH) <sub>2</sub> (s) Insoluble in excess NaOH	White precipitate Insoluble in excess
<b>Aluminium</b> (Al <sup>3+</sup> )	White precipitate Al <sup>3+</sup> (aq) + 3OH <sup>-</sup> (aq) → Al(OH) <sub>3</sub> (s) soluble in excess NaOH resulting a colourless solution	White precipitate Insoluble in excess
<b>Zinc</b> (Zn <sup>2+</sup> )	White precipitate Zn <sup>2+</sup> (aq) + 2OH <sup>-</sup> (aq) → Zn(OH) <sub>2</sub> (s) soluble in excess NaOH	White precipitate soluble in excess

## 2. Test for negative ions (Anions)

Negative ion	Test
<b>Carbonate</b> (CO <sub>3</sub> <sup>2-</sup> )	CO <sub>2</sub> liberated by dilute acids (CaCO <sub>3</sub> + HCl → CaCl <sub>2</sub> + CO <sub>2</sub> + H <sub>2</sub> O) CO <sub>2</sub> liberated by heating (except for group 1 other than Lithium carbonate) (CaCO <sub>3</sub> $\xrightarrow{\Delta}$ CaO + CO <sub>2</sub> )
<b>Hydrogencarbonate</b> (HCO <sub>3</sub> <sup>-</sup> )	CO <sub>2</sub> liberated by dilute acids (NaHCO <sub>3</sub> + HCl → NaCl + CO <sub>2</sub> + H <sub>2</sub> O) CO <sub>2</sub> liberated by heating (2NaHCO <sub>3</sub> $\xrightarrow{\Delta}$ Na <sub>2</sub> CO <sub>3</sub> + CO <sub>2</sub> + H <sub>2</sub> O)
<b>Chloride</b> (Cl <sup>-</sup> )	Acidify with dilute nitric acid, and then add aqueous silver nitrate. White precipitate will result (AgCl) The precipitate is soluble in dilute ammonia solution.
<b>Bromide</b> (Br <sup>-</sup> )	Acidify with dilute nitric acid, and then add aqueous silver nitrate. Cream precipitate will result (AgBr) The precipitate is partially soluble in dilute ammonia solution & completely soluble in concentrated ammonia solution.
<b>Iodide</b> (I <sup>-</sup> )	Acidify with dilute nitric acid, and then add aqueous silver nitrate. Yellow precipitate will result (AgI) The precipitate is insoluble in ammonia solution.
<b>Sulphate</b> (SO <sub>4</sub> <sup>2-</sup> )	Acidify with dilute nitric acid, and then add aqueous barium nitrate OR Acidify with dilute hydrochloric acid, and then add aqueous barium chloride White precipitate, which is insoluble in acids, will result. (BaSO <sub>4</sub> )
<b>Sulphite</b> (SO <sub>3</sub> <sup>2-</sup> )	SO <sub>2</sub> gas is liberated with dilute acids. Results white precipitate with aqueous Ba <sup>2+</sup> solutions, which is soluble in dilute/ strong acids
<b>Nitrate</b> (NO <sub>3</sub> <sup>-</sup> )	Ammonia gas liberated on heating with NaOH (or KOH) & Aluminium foil.

### 3. Tests for Gases

Gas	Test and Test results
Ammonia (NH <sub>3</sub> )	Turns damp red litmus paper blue (NH <sub>3</sub> is an alkaline gas)
Carbon dioxide (CO <sub>2</sub> )	Gives a white precipitate with limewater. Precipitate dissolves with excess CO <sub>2</sub> due to HCO <sub>3</sub> <sup>-</sup> formation
Carbon monoxide (CO)	Burns with a blue flame when a burning splint is held
Chlorine (Cl <sub>2</sub> )	Bleaches damp litmus paper (turns red & then bleaches)
Bromine (Br <sub>2</sub> )	Bleaches damp litmus paper (turns red & then slowly bleaches)
Iodine (I <sub>2</sub> )	Moist starch paper turns blue-black
Hydrogen (H <sub>2</sub> )	"Pops" with a lighted splint
Oxygen (O <sub>2</sub> )	Relights a glowing splint
Sulphur dioxide (SO <sub>2</sub> )	Good reducing agent. Turns aqueous potassium dichromate (VI) from orange to green. OR Turns aqueous potassium manganate (VII) from purple to colourless (SO <sub>2</sub> is a reducing agent) OR Moist blue litmus turns red
Nitrogen dioxide (NO <sub>2</sub> )	Orange-brown gas, Strong oxidizing agent, it oxidizes a colourless solution of Potassium Iodide to produce yellow brown precipitate of Iodine, also dissolves in water to produce nitric acid. Moist blue litmus turns red.
Hydrogen halides	Moist blue litmus turns red OR When the gas is passed into silver nitrate solution the solution turns cloudy (white if HCl, Cream if HBr, Yellow if HI)

#### 4. Recognition of common gases.

GAS	OBSERVATIONS
Chlorine	Pale green gas
Bromine	Brown gas
Iodine	Purple vapour
Nitrogen dioxide	Brown gas
Hydrogen chloride	Steamy fumes on exposure to moist air, acidic
Water vapour	Turns blue cobalt paper pink

#### 5. Ignition.

You may be asked to heat an unknown substance in an ignition tube or a test tube. The colour changes or the identity of any gas evolved may provide evidence to identify the substance.

Gases or vapours may be evolved on heating the solid.

GAS OR VAPOUR	POSSIBLE SOURCE
Carbon dioxide	Carbonates of metals other than group 1; Hydrogen carbonates of group 1
Ammonia	Ammonium salts
Oxygen alone	Group 1 nitrates (other than Li) or halate of Na or K
Oxygen & nitrogen dioxide	Nitrates (other than Na or K)
Hydrogen chloride	Ammonium chloride
Water	Hydrated salts, hydrogen carbonates & other acid salts

#### 6. Action of dilute acids

When dilute sulphuric or hydrochloric acid is added to a substance a gas may be evolved or there may be a colour change in the solution.

ACTION OF ACID	CAUSE
Carbon dioxide evolved	Carbonate or hydrogen carbonates*
Sulphur dioxide evolved on warming	Sulphite
Nitrogen dioxide evolved	Nitrite

\*To distinguish between the carbonates & hydrogen carbonates of group 1, prepare an aqueous solution of the salt & proceed as follows

TEST	OBSERVATIONS	
	CARBONATE	HYDROGENCARBONATE
On aqueous solution		
pH	12-14	7-8
Boil	No change	CO <sub>2</sub> evolved
Add an aqueous Ca or Mg salt & boil the mixture	Immediate white precipitate at room temperature	A white precipitate only forms on boiling

## 7. Tests for oxidizing & reducing agents

Reducing agents usually decolorizes aqueous acidified potassium manganate (VII) & may also turn aqueous, acidified potassium dichromate (VI) from orange to green.

Reducing agents likely to be used in the practical examinations include iron (II) ions, nitrite ions, Sulphite ions, iodide ions & hydrogen peroxide.

Oxidizing agents usually liberate iodine as a brown solution or black solid from aqueous potassium iodide. Iodine solution gives a blue-black colour with starch.

Oxidizing agents likely to be used in the practical examinations include manganate (VII) ions, chromate (VI) ions, hydrogen peroxide, Copper (II) ions, aqueous chlorine & aqueous bromine.

## 8. Flame tests

FLAME COLOUR	INFERENCE
Intense, lasting yellow	Sodium ion
Lilac	Potassium ion
Orange red/ Brick red	Calcium ion
Bright Red	Lithium
Pale green / Apple green	Barium ion
Crimson Red	Strontium ion
Red/ purple	Rubidium ion
Blue / Violet	Cesium ion

## PRECIPITATES

### 9. Barium chloride solution

Aqueous  $\text{BaCl}_2$  forms precipitate of insoluble barium salts with a number of anions but is usually used as a test for the Sulphate ion ( $\text{SO}_4^{2-}$ ). Aqueous barium chloride is usually used with dilute hydrochloric acid.

Anion	Precipitate		Addition of dilute HCl
	Colour	Formula	
Sulphate	White	$\text{BaSO}_4$	Precipitate is insoluble
Sulphite	White	$\text{BaSO}_3$	Precipitate dissolves
Carbonates	White	$\text{BaCO}_3$	Precipitate dissolves with effervescence

### 10. Silver nitrate solution

Aqueous silver nitrate is commonly used to test for the presence of halide ions in solution. Anions, which would interfere with the test (eg. Carbonates) are first removed by adding dilute nitric acid before the aqueous silver nitrate. The identity of a halide may be confirmed by the addition of aqueous ammonia, both dilute & concentrated.

Anion	Precipitate		Addition of aqueous Ammonia	
	Colour	Formula	dilute	concentrated
Chloride	White	$\text{AgCl}$	Soluble	-----
Bromide	Cream	$\text{AgBr}$	Slightly soluble	Soluble
Iodide	Yellow	$\text{AgI}$	Insoluble	Insoluble

### 11. Concentrated sulfuric acid.

When a few drops of concentrated acid are added to a solid halide the observed reaction products may be used to identify the particular halide ion present. This is a potentially hazardous reaction. It must be carried out on a small scale & in a fume cupboard.

Halide	Observations on adding H <sub>2</sub> SO <sub>4</sub>	Observed products
Chloride	White steamy vapour	HCl
Bromide	White steamy vapour, Brown vapour	HBr, Br <sub>2</sub> , (SO <sub>2</sub> )
Iodide	White steamy vapour, black solid, purple vapour, yellow solid, (Rotten egg smell)	HI, I <sub>2</sub> , S, (H <sub>2</sub> S)

The products in brackets will not be observed since they are colourless gases. No attempt should ever be made to detect these gases by smell.

### Tests on Organic Compounds

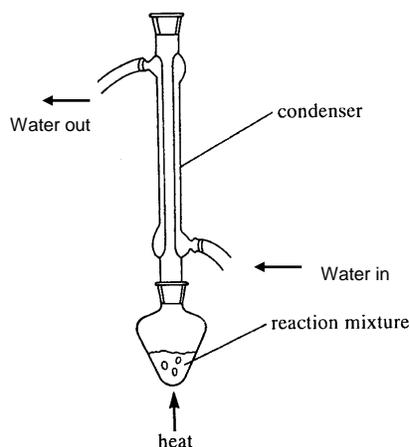
TEST	OBSERVATION	INFERENCE
Warm with acidified potassium dichromate (VI)	Orange to green solution	Primary or secondary alcohol; aldehyde
Warm with acidified potassium manganate (VII)	Purple to colourless solution	Primary or secondary alcohol; aldehyde
Shake with bromine solution	Orange brown solution is decolorized	Alkene
Warm with aqueous NaOH, acidify with dilute nitric acid then add aqueous AgNO <sub>3</sub>	White precipitate Cream precipitate Yellow precipitate	Haloalkane; C-Cl, C-Br, C-I
Phosphorus pentachloride	Effervescence, steamy fumes of HCl	-OH group in alcohols & carboxylic acid
Warm with Fehling's or Benedict's solution	Red Precipitate	Aldehyde group present.
Warm with Tollens reagent	Silver Mirror	Aldehyde group present.
Add sodium carbonate / sodium bicarbonate	Effervescence	Carboxylic Group present.

## Practical Techniques

### □ Heating under Reflux

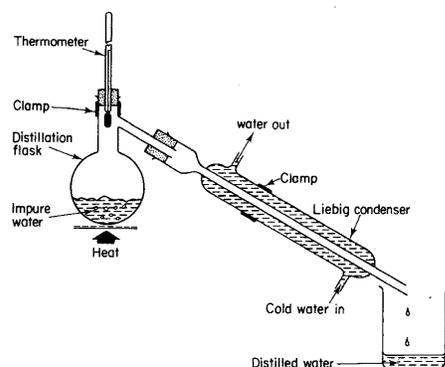
Heating under reflux enables a mixture including volatile materials to be heated for a long time without loss of solvent. The system is designed to keep materials in the flask, this is achieved by the vertical condenser attached to the reaction vessel so that the condensed vapour is returned back to the flask. Cooling water is circulated to condense escaping vapors. One should always use a boiling stone or a magnetic stirrer to keep the boiling solution from "bumping."

- The water sent in at the bottom of the condenser against the gravity to ensure proper cooling.
- There must not be a stopper in the top of the condenser - the apparatus must not be sealed in order to prevent the pressure build up.
- When heating flammable substances (eg:- ethanol) direct flame is not applied, instead a water bath (for substances having boiling point lower than that of water) or a oil bath (for substances having boiling point higher than that of water) or an electric heating mantle can be used.



### □ Simple Distillation

Simple distillation is designed to evaporate a volatile liquid from a solution of non-volatile substances; the vapour is then condensed in the water condenser and collected in the receiver. It is based on the principle that a pure liquid at its boiling point will have the same composition for the vapour & will distill at a constant temperature & that is the boiling point of the liquid.



*Laboratory apparatus for distillation*

The apparatus consists of a **round-bottomed distilling flask** connected to a **water condenser** (Liebig condenser). The thermometer bulb should be at the same level of the condenser to find the temperature of the exit vapour. Temperature begins to change when the next fraction enters the condenser, thus the receiver should be changed to collect the next fraction,

Water is sent against gravity to ensure proper cooling. The condenser has to be a water condenser if the boiling point of the liquid is below 100°C, if above 100°C an air condenser should be used.

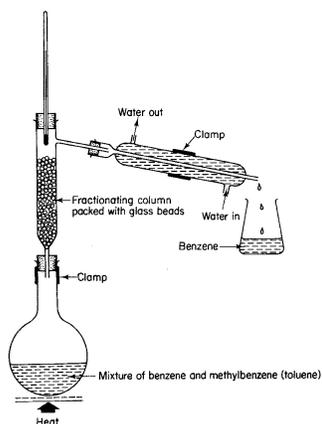
If a flammable substance is heated then it should be done by a water bath / oil bath / electrical heater.

This simple distillation is used when separating a liquid, which is mixed with solids. When there is a liquid mixture there should be significantly large difference in the boiling points of the liquids. Separation of mixtures with close boiling points is done by **Fractional distillation**.

### □ Fractional Distillation

Fractional distillation is used to separate two or more miscible liquids based on their different boiling points. This process is similar to the distillation except that a fractionating column is fitted between the distilling flask & the condenser.

The mixture is heated, & the liquid with the lowest boiling point will boil first. This liquid vaporizes & condenses before the other liquid. The Fractionating column attached consist of glass beads packed together. The fractionating column helps to make sure that the second liquid does not get into the condenser until the entire first one has.



Separation of a benzene/methylbenzene (toluene) mixture  
fractional distillation

The fractionating column helps to cool the vapours rising up the column. The length of the fractionating column can be varied based on the boiling points of the liquids. If the difference in boiling points is small then a long fractionating column should be used.

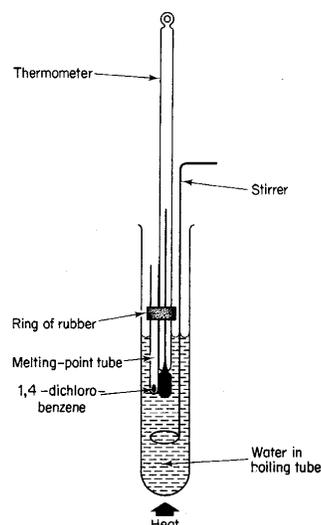
The temperature of the thermometer stays constant until the relevant solution distills off. The as the second solution starts to enter the temperature of the thermometer rises. By observing the temperature difference various fractions can be separated.

	Simple distillation	Fractional distillation
Advantages	<ul style="list-style-type: none"> <li>• Simpler setup than fractional distillation.</li> <li>• Faster distillation. Easy to setup.</li> <li>• consumes less energy than fractional distillation</li> </ul>	<ul style="list-style-type: none"> <li>• much better separation between liquids than simple distillation</li> <li>• purifies readily the complex mixtures than simple distillation</li> </ul>
Disadvantages	<ul style="list-style-type: none"> <li>• requires the liquids to have large boiling point differences (&gt;25°C)</li> <li>• poorer separation than fractional distillation</li> </ul>	<ul style="list-style-type: none"> <li>• more complicated setup than simple distillation</li> <li>• Takes longer time.</li> <li>• consumes more energy than simple distillation</li> </ul>

## □ Determination of melting point

The melting point is the temperature at which a solid is converted to liquid. This is an important property of solids. The melting point of solids, like the boiling point of liquids, is often used for the identification of substances.

Melting temperature is often used to check for the purity of a solid. The most common melting point apparatus consists of a closed end capillary tube which is attached to a thermometer dipped in a boiling tube which contains a solvent which has a higher boiling point than the melting point of the solid.



### Summary of melting points determination steps.

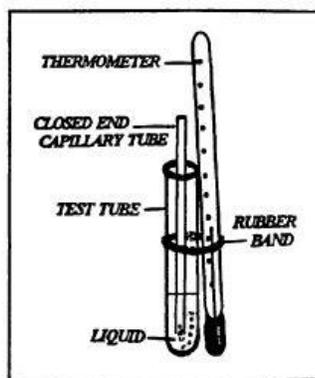
- Take a capillary tube and seal the end by holding the end corner to the Bunsen flame to make a seal. Push the open end of a capillary tube into the powdered sample.
- Move the powder to the closed end of the capillary tube by tapping it on the table. Repeat until the powdered sample occupies 1-2 mm of the capillary tube end.
- Attach the capillary tube to a thermometer with rubber bands and align the bulb of the thermometer with the closed end of the capillary tube.
- Place the thermometer/capillary tube assembly in the water bath so that the surface level of the powdered sample is beneath the surface level of the water bath/oil bath.
- Place the beaker on the burner stand and, stirring frequently to insure even heating, carefully heat the water bath/oil bath with your heat source.
- Note the temperature at which the solid melts.

## □ Determination of boiling point

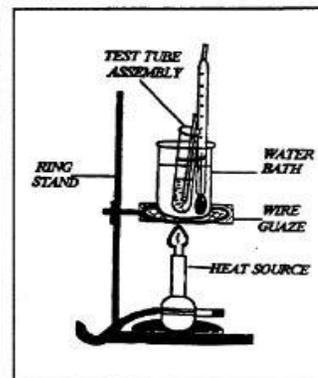
The best method to determine the boiling point is to carry out a distillation & observe the temperature when the distillate is produced. If the sample is small it can be done by setting up the following apparatus.

### Procedure:

- Place about 1 ml of liquid sample in the test tube.
- Using a small rubber band, attach a thermometer to the outside of the test tube. The thermometer bulb should be even with the test tube's bottom.



Test Tube Assembly ↑

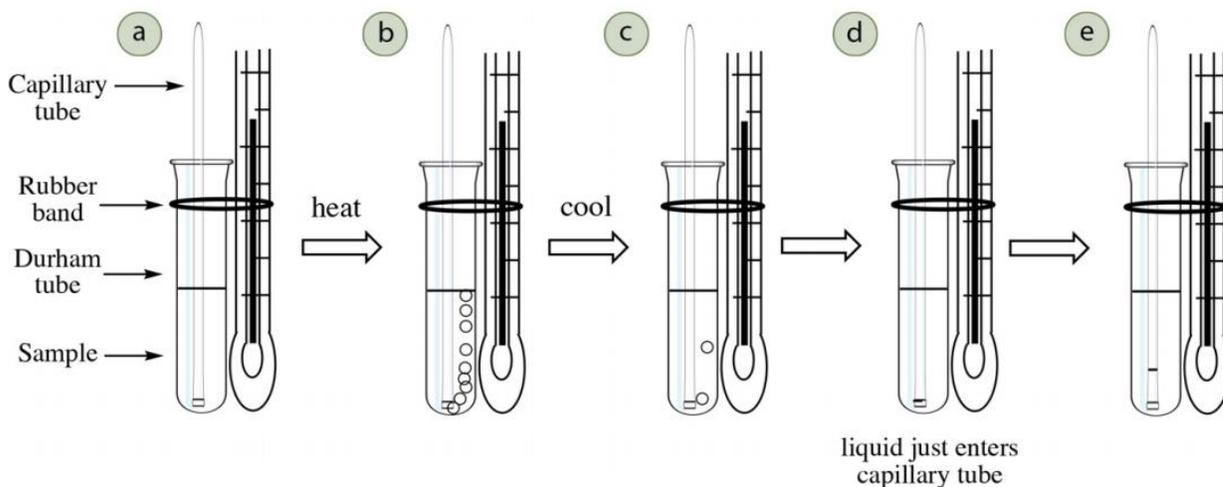


Heating Assembly ↑

- c. Insert an inverted closed end capillary tube into the test tube. (the open mouth of the narrow capillary tube should be dipped in the liquid & the sealed end exposed to air)
- d. Make a water bath assembly by using the following directions and illustration.
- e. Half fill a 100 ml or larger beaker with warm tap water. [Note: a water bath is used if the boiling point of the material is expected to be less than the boiling point of water; otherwise, an oil bath is needed.]
- f. Place the above test tube assembly in the water bath so that the surface level of the liquid in the test tube is beneath the surface level of the water bath.
- b. Place the beaker on the wire stand and, stirring frequently to insure even heating, carefully heat the water bath with your heat source until the water bath boils and a rapid stream of bubbles continuously emerges from the capillary tube. Remove the heat source and begin observing the stream of bubbles. (air slowly escapes from the small capillary tube until the boiling temperature of the sample where a rapid stream of bubbles occur.
- g. When the last bubble emerges from the capillary tube, record the temperature.
- h. Reheat the water bath and repeat the cooling process two more times. Record the temperature reading after each trial, and average all three trials.

This method utilizes the definition of boiling point: the temperature where the compound's vapor pressure equals the atmospheric pressure. The inverted capillary tube acts as a reservoir to trap the compound's vapors. As the apparatus is heated, the air initially trapped in the capillary tube expands and causes bubbles to emerge from the tube (Figure b). With further heating, the compound's vapors eventually displace all of the trapped air, which is why heat is applied until there is a continuous stream of bubbles.

When the apparatus is cooled, eventually the pressure inside the capillary tube will match the atmospheric pressure, at which point the bubbles will slow and liquid will be drawn into the tube. The temperature where this begins is the compound's boiling point (Figure d).



## Solvent Extraction

**Solvent extraction** also known as **Liquid-liquid extraction** and **partitioning**, is a method to separate compounds based on their relative solubilities in two different immiscible liquids, usually water and an organic solvent. It is an extraction of a substance from one liquid phase into another liquid phase. Liquid-liquid extraction is a basic technique in chemical laboratories, where it is performed using a separating funnel.

In other words, this is the separation of a substance from a mixture by preferentially dissolving that substance in a suitable solvent. By this process, a soluble compound is usually separated from an insoluble compound. Solvent extraction is used in the production of fine organic compounds, the processing of perfumes, and other industries.

### **Procedure:**

- After placing the reaction mixture & the solvent in the separating funnel, place the stopper invert and gently shake the contents for a while.
- Open the inverted funnel in-between mixing to release any build up pressure.
- Place the funnel in a ring and allow the layers to separate.
- Remove the stopper and open the tap and drain the lower layer out.
- Pour the upper layer from the top of the funnel (not from the tap) into a flask.

### Purification by Washing

If the product does not decompose at the boiling temperature of the reaction mixture, distillation is carried out to obtain the product. If the product is insoluble in water it can be washed with sodium carbonate solution in a separating funnel. This treatment removes any acidic impurities. Such impurities react with the carbonate to give off carbon dioxide gas. The separated funnel has to be inverted & tap should be opened to release the carbon dioxide so that the pressure is maintained. Washing should be repeated until no gas is produced.



The aqueous layer is discarded & the organic layer is washed with water. This is done to remove any unreacted sodium salts & any soluble organic substances, such as ethanol.

The new aqueous layer is discarded & the organic layer is dried with a suitable drying agent which shows no reaction with the product. The dried organic layer is decanted off the solid drying agent & distilled.

It's much efficient to use smaller volumes (i.e: four portions of 25cm<sup>3</sup> than one portion of 100cm<sup>3</sup>) as it results in more efficient extraction. It removes more desired product than the single extraction.

### □ Drying with an Anhydrous Salt.

Drying agent is an anhydrous salt such as sodium sulfate, magnesium sulfate & calcium sulfate. Main feature of a drying agent is that it should not react with the substance being dried.

These drying agents absorb water from the organic liquid and form hydrous salt, and water will be as the water of crystallization of the salt.

#### ▪ **Procedure:**

The anhydrous drying agent is added to the organic liquid which should be dried and is swirled. When the powdery drying agent absorbs water it appears to be crystalline. It should be left for some time until the solution becomes clear (Organic liquid with water would appear cloudy) upon further addition of the drying agent if it remains powdery in the solution is an indication that the liquid is dry. Remove the drying agent by decantation or filtration with the help of a cotton plug.

### □ **Crystallisation:**

This technique is used to separate a solid from a solution. The solution is heated over a water bath until the point of crystallization. (or point of saturation) The point of crystallization is the temperature in which the crystals begin to appear in the mixture upon cooling. In order to determine this point a glass rod is dipped from time to time and checked for crystals upon cooling.

Once the solution is heated up to the point of crystallization it is taken off the water bath & left to cool at room temperature. Crystals begin to appear gradually upon cooling. (As the solubility decreases with decrease in temperature) At this point filter off the crystals & dry it in an oven or a desiccator till a constant mass is obtained in order to ensure no water present.

#### **Apparatus:**

## ACID-ALKALI TITRATION

01. What is the purpose of carrying out an acid-alkali titration?

The Purpose of a titration is to find the concentration of a concentration unknown acid or alkali using a concentration known acid or alkali. As this method involves measuring volumes its also known as volumetric analysis.

02. Describe how an acid-base titration is carried out between  $0.1\text{mol dm}^{-3}$  NaOH & concentration unknown HCl.

- The burette should be first washed with distilled water & then rinsed with the solution which is added to it. (let us assume as acid is added)
- Clamp the burette vertically on to a burette stand & fill the acid above the zero mark. Open the tap & let the acid out drop wise to a beaker until the volume can be read to the meniscus level. (it is not a must to start with  $0.00\text{cm}^3$ ) This is done to ensure the jet (space between tap and the tip) is filled, if not as tap is open during titration some of the liquid will fill this space and will not enter the conical flask.
- Wash the conical flask with distilled water (do not rinse with the alkali as this will increase the number of moles) and place it below the burette above a white tile.
- Wash the pipette with distilled water & rinse with the alkali. With the help of a pipette filler pipette out  $25.00\text{cm}^3$  of the alkali to the conical flask.
- Add 2-3 drops of the acid-alkali indicator to the conical flask. . An indicator is a weak acid or a weak base. Use of an excessive amount of indicator will affect the titre value.
- The solution from the burette is added rapidly while swirling of the flask, until the indicator changes colour. This is called the rough titration. (Titre) This titre gives the rough idea of the volume at the end point. (**Titre** is the volume added from the burette.)
- The titration is repeated with a fresh solution on the conical flask, the acid from the burette is added quickly until about  $2\text{cm}^3$  less than the approximate titre.
- The solution is then added slowly dropwise, swirling the flask constantly, until the indicator changes the colour permanently. This is the **equivalence point** of the titration & the **end point** of the indicator. The volume is recorded using the lowest part of the **meniscus**. The volume of the titre is recorded.
- The titration is repeated until at least two **concordant titres** are obtained. Concordant means that two titres are within  $0.2\text{cm}^3$  of each other.
- The concordant titres are then averaged to give the mean titre.

03.

i. What is a standard solution?

A solution with an accurately known concentration.

ii. What is a primary standard? What are its Characteristics?

A primary standard is a substance which can be weighed out accurately to make up a standard solution. Substance to be a primary standards should meet following criteria.

- Does not decompose / react with air
- Does not absorb moisture
- Higher degree of purity
- Has a relatively high molar mass so that weighing errors are minimized

Examples for primary standards: Anhydrous  $\text{Na}_2\text{CO}_3$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$ , KI

04. How do you tabulate your readings?

Initial volume of Titre / $\text{cm}^3$	Final volume of titre / $\text{cm}^3$	Volume of titre added / $\text{cm}^3$	Mean titre / $\text{cm}^3$

05. Common indicators & their colour changes

Indicator	Acid colour	Neutral colour	Alkaline colour
Phenolphthalein	Colourless	Colourless	Pink
Methyl orange	Red	Orange	Yellow
Bromothymol blue	Yellow	Green	Blue
Methyl Red	Red	Orange	Yellow

06. Explain why the burette should be rinsed with the solution before the titration

If it is not rinsed with the solution after washing with water, the moisture inside the burette will dilute the solution added.

07. How do you prepare a standard solution if a solid mass is given in order to titrate?

Preparation of standard solution is only appropriate with a chemical that is very pure, does not gain or lose mass when in air, & has a relatively high molar mass so that weighing errors are minimized. The chemicals meet these criteria are primary standards.

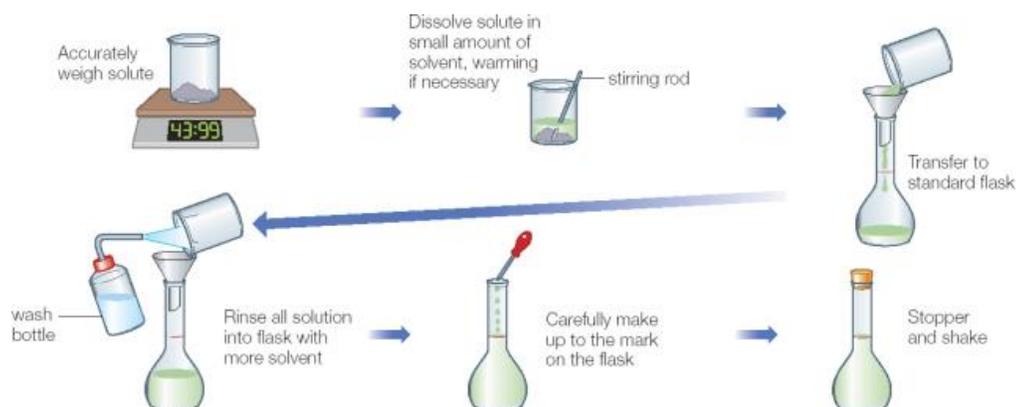
**Procedure:**

Weigh accurately known mass of primary standard into a beaker. Add about 100 cm<sup>3</sup> of deionized water and stir to dissolve the sample. Transfer all of the solution into a 250 cm<sup>3</sup> volumetric flask.

Place a large funnel in the neck of the volumetric flask, and pour the solution into the flask. Rinse the beaker, glass rod & funnel with deionized water and add the washings to the volumetric flask.

Make up the volumetric flask to the mark, using a dropper to add the final amounts of deionized water. Stopper the flask and invert several times. This is done to ensure a homogeneous solution.

The following figure shows the procedure for preparing a standard solution from a primary standard.



08. How to dilute a solution quantitatively?

Dilution is to make a solution with a lesser concentration by diluting a standard solution. The procedure for dilution is to take a measured volume of the highly concentrated solution with a pipette (or burette) & run it into a graduated flask. The flask is then carefully filled to the mark with distilled water.

The key to calculate the volume to use when diluting a solution is to remember that the amount in moles of the chemical dissolved in the diluted solution must be equal to the amount in moles of the sample taken from the concentrated solution.

The amount of moles of the chemical in the measured volume of the concentrated solution =  $C_1V_1$

The amount of moles of the chemical in the diluted solution =  $C_2V_2$

As above two amounts are same  $C_1V_1 = C_2V_2$

## Back Titration

A back titration used for substance which cannot be titrated directly, such as insoluble solid.

The method is as follows:

- Weigh a sample of the substance.
- Add excess of a standard solution, an acid or a base.
- Either titrate the excess or make up the solution to  $250\text{ cm}^3$  & titrate portions of the dilute solution that contain the excess.

Eg I: Determination of the purity of chalk (impure  $\text{CaCO}_3$ )

The procedure: weigh the impure chalk & place it in a beaker.

Add  $50\text{ cm}^3$  of  $1\text{M HCl}$  in excess to make sure all chalk reacts.

Transfer to a  $250\text{ cm}^3$  volumetric flask. Wash the beaker & the funnel into the flask & make up to the mark with distilled water. Shake the flask well.

Titrate  $25\text{ cm}^3$  portions of this solution against concentration known alkali.

The calculation:

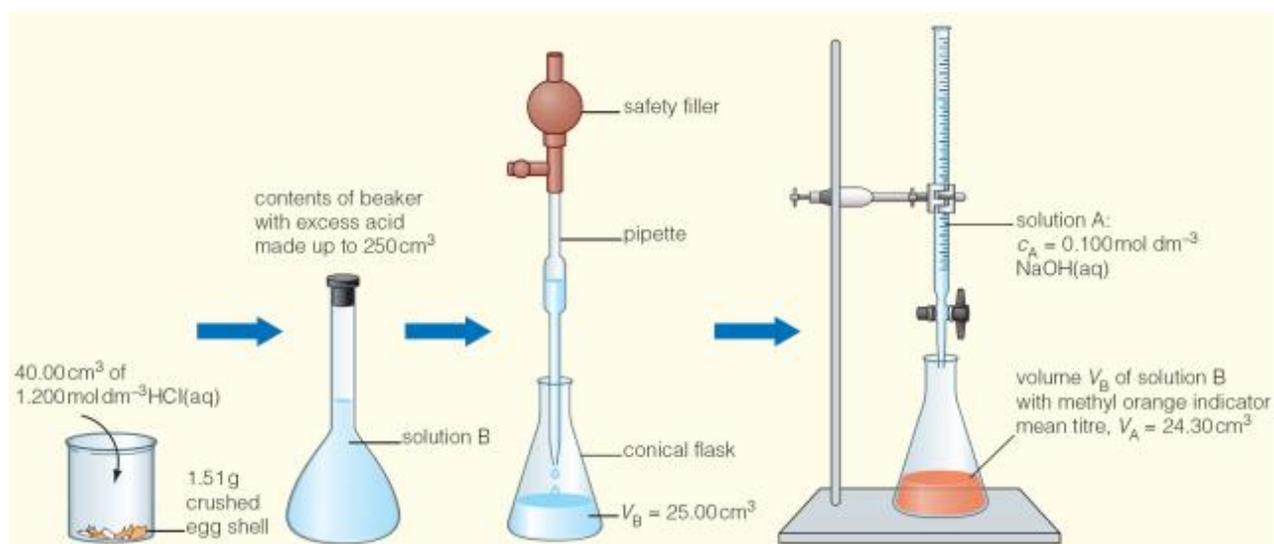
Mean titre  $\rightarrow$  moles of alkali  $\rightarrow$  moles of excess  $\text{HCl}$  in  $25\text{ cm}^3 \rightarrow$  total moles in excess  $\text{HCl}$

Original volume of  $\text{HCl} \rightarrow$  moles of  $\text{HCl}$  taken

Moles of  $\text{HCl}$  reacted with  $\text{CaCO}_3$  (original  $\text{HCl}$  moles – total moles of excess  $\text{HCl}$ )

Moles of  $\text{CaCO}_3 \rightarrow$  Mass of  $\text{CaCO}_3$  in the chalk sample  $\rightarrow$  % purity

Eg II: Finding the percentage of  $\text{CaCO}_3$  in egg shell.



## Redox Titrations

### □ Potassium manganate (VII) titrations:-

KMnO<sub>4</sub> can be used as a self indicator due to the intense purple colour and gives a pink colour even in very dilute solution. In titrations involving KMnO<sub>4</sub> it is usually held in the burette, so the pink solution can be easily observed at the end point.

The half reaction of KMnO<sub>4</sub> in acidic solution is given as:-

KMnO<sub>4</sub> oxidizes organic compounds as it is a strong oxidizing agent.

KMnO<sub>4</sub> cannot be obtained in a state of high purity. Therefore solutions of KMnO<sub>4</sub> should be standardized to know its accurate concentrations. Usually Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> (Sodium ethanedioate) is commonly used as a standard to standardize KMnO<sub>4</sub> solution.

The reaction after the oxidation of ethanedioate ion is

Therefore the overall reaction:

The reaction initially is quite slow. The product Mn<sup>2+</sup> catalyses the reaction hence makes it much faster. A reaction where the product catalyses the reaction is called an autocatalytic.

### □ Sodium thiosulphate titrations:-

The thiosulphate ions are oxidized to tetrathionate ions (S<sub>4</sub>O<sub>6</sub><sup>2-</sup>) at the presence of the oxidizing agent I<sub>2</sub>, where I<sub>2</sub> reduces itself to I<sup>-</sup> ions. The yellow brown colour of I<sub>2</sub> gradually disappears as the reaction proceeds.



Titration of I<sub>2</sub> with S<sub>2</sub>O<sub>3</sub><sup>2-</sup> the solution turns from yellow brown to colourless at the end point. As the colour difference is difficult to observe, freshly prepared starch solution is added to the conical flask at closer to the end point. When starch is added the solution turns deep blue due to the formation of starch iodine complex which turns colourless at the end point.

The above titration can be used to determine the amount of copper present in brass, or the number of water molecules of water of crystallization in hydrated CuSO<sub>4</sub>



In the above reaction I<sup>-</sup> ions are oxidized to I<sub>2</sub> therefore the liberated I<sub>2</sub> can be titrated against sodium thiosulphate solution to find the amount of I<sub>2</sub>, thus the amount of Cu<sup>2+</sup> can be found.

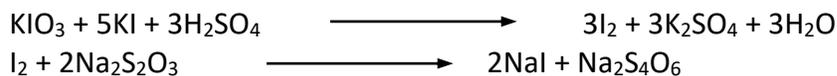
### Determination of purity of Potassium Iodate (V) (KIO<sub>3</sub>)

1.00 g of an impure sample of KIO<sub>3</sub> was dissolved in distilled water & the solution was made up to 250cm<sup>3</sup>.

25.0 cm<sup>3</sup> portions of this solution were taken & about 25 cm<sup>3</sup> of dilute sulfuric acid & excess KI were added. Each was titrated with standard 0.1 moldm<sup>-3</sup> sodium thiosulfate solution. The mean titre was 23.75 cm<sup>3</sup>.

Calculate the purity of the Potassium Iodate (V)

The equations for the reactions are:



Answer: (84.7%)

What is the concentration in ppm of a solution containing 45ng (nano grams) of a toxic chemical in 100 cm<sup>3</sup> of water?

There are 45 in 100g of water, which is 4.5 X 10<sup>-8</sup>g in 100g of water.

$$\text{ppm} = \frac{4.5 \times 10^{-8} \times 10^6}{100} = 0.00045 \text{ ppm}$$

## Determination of moles of water of crystallization by titration

Determination of the amount of water of crystallisation in hydrated sodium carbonate

Hydrated sodium carbonate has the formula  $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$ , where  $x$  is the number of molecules of water of crystallisation present. In this experiment,  $x$  is determined by titration of a solution made using hydrated sodium carbonate with a standard solution of hydrochloric acid.

### **Procedure:**

Weigh accurately known mass of hydrated sodium carbonate into a beaker. Add about 50  $\text{cm}^3$  of deionized water and stir to dissolve the sample. Transfer all of the solution into a 250  $\text{cm}^3$  volumetric flask. Rinse the beaker with deionized water and add the washings to the volumetric flask. Make up the volumetric flask to the mark, using a dropper to add the final amounts of deionized water. Stopper the flask and invert several times. This is done to ensure a homogeneous solution.

Rinse the burette, pipette and conical flask respectively with deionized water. Rinse the burette with hydrochloric acid solution, and rinse the pipette with sodium carbonate solution. Titrate the sodium carbonate solution against 0.1 M hydrochloric acid, using 25  $\text{cm}^3$  of sodium carbonate solution in the conical flask and 2-3 drops methyl orange as indicator. An indicator is a weak acid or a weak base. Use of an excessive amount of indicator will affect the titre value. Repeat the titrations until concordant results are obtained. Calculate the concentration of the sodium carbonate solution.

The formula of hydrated sodium carbonate can be determined by calculating the mass of sodium carbonate reacted with HCl. Subtract this mass which is the mass of the anhydrous  $\text{Na}_2\text{CO}_3$  from the initial mass to find the mass of water. Convert this into moles and by working out the simplest ratio between  $\text{Na}_2\text{CO}_3 : \text{H}_2\text{O}$  number of water moles per mole of  $\text{Na}_2\text{CO}_3$  can be found.

- Determine the formula of hydrated sodium carbonate. Sodium Carbonate solution was made by dissolving 1.57g of hydrated  $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$  in 250 $\text{cm}^3$  of distilled water. 25 $\text{cm}^3$  portions of this solution was titrated with 0.1M HCl. The mean titre was 12.55 $\text{cm}^3$

## MISTAKES, ERRORS, ACCURACY & PRECISION.

### Mistakes, Errors and uncertainty in volumetric Analysis

#### Types of uncertainty in measurements

Errors in measurements are not mistakes; and mistakes are not errors. Mistakes are done due to negligence and even skilled scientist will find it difficult to eliminate errors.

Eg: Forgetting to tare the balance, Forgets to remove the funnel from the burette, taking wrong readings.

Errors are unavoidable differences between measured values and true values. Often the true value of a particular measurement is not known, so scientists have to assess the degree of uncertainty in their measurements. 'Measurement uncertainty' and 'experimental error' often mean the same thing, but the term 'measurement uncertainty' is preferable. These are not errors or mistakes on the part of the experimenter, but unavoidable variations in results.

There are three kinds of uncertainty in measurements – random errors, systematic errors and zero errors.

**Random errors:** Caused by unpredictable conditions like temperature / pressure and are the uncertainties which arise when a measurement is repeated many times, giving values distributed equally on both sides of the true value.

Random error can be reduced by taking several measurements and calculating an average value.

Eg: Collecting volumes of gases at different temperatures / pressure / humidity during the day.

**Systematic errors:** Caused by affect all the measurements using a particular instrument or technique in the same way, making them all higher or all lower than the true value. These values will be precise but not accurate.

Systematic errors are not reduced by taking repeated measurements and then averaging. However, systematic errors can be corrected by calibration with an accurate instrument or by comparison with a more reliable technique.

**Zero errors:** are a particular form of systematic error caused by measuring instruments that have a false zero. Examples are where the needle on an ammeter fails to register zero when no current flows, or a centigrade thermometer fails to show zero when the temperature is 0°C.

The errors in titration are associated in:

- Electronic digital balance
- Pipettes
- Volumetric flask
- Burettes
- Determining the end point of the titration.

It is possible to quantify the error associated with laboratory equipment so that the overall error of a titration can be found.

### Measurement of Uncertainty.

Percentage uncertainty depends on the value which is measured (how big / small) and the uncertainty/ error of the apparatus. Also depends on the number of recording taken each time.

- **How to find the error / uncertainty of an instrument?**

This is done by dividing the smallest reading on the apparatus by 2.

Eg: Burette which reads to minimum 0.01ml, the error would be +/- 0.005ml, Thermometer reads to 1°C error would be +/- 0.5 °C

- **How to calculate the total uncertainty / error?**

This depends on number of reading taken each time. If two readings taken error should be multiplied by 2 in order to obtain the total error.

Eg. of apparatus where error is multiplied by 2 : **Digital scale, burette, thermometer**

Eg. of apparatus where error is not multiplied by 2 : **Pipette, measuring cylinder, Volumetric flask**

- **How to calculate the percentage error / percentage uncertainty ?**

$$\% \text{ error} = \frac{\text{Total error}}{\text{measured value}} \times 100$$

% error can be minimized by taking larger measured values.

- **Electronic digital balance:**

Percentage error of a balance depends on the precision & the mass being weighed. A balance which weighs to 2 decimal places is more precise than the one which measures to one decimal place. Thus measurement of uncertainty involving 2 d.p balance is lower than 1 d.p balance. A typical digital balance in the laboratory would read to 0.01g. The error associated with a balance would be half of the smallest reading which is half of 0.01g = +/- 0.005g Percentage error depends on the mass being measured on it, greater the mass lesser would be the percentage uncertainty / error, The error of the balance should be multiplied by 2 in order to find the total error as we take two mass readings. On when calibrating the balance to zero and the other being the error when weighing. % error can be calculated as:

$$\% \text{ error} = \frac{0.005\text{g} \times 2}{\text{Mass measured (g)}} \times 100$$

Eg: Experiment where 10 g of a solid was being measured the error would be:

$$\% \text{ error} = \frac{0.005 \text{ g} \times 2}{10 \text{ g}} \times 100 = 0.1 \%$$

**NOTE : If the balance is used twice, the error should be multiplied 4 times. (Twice each)**

Eg: When mass of the weighing bottle + solid & mass of the empty weighing bottle taken separately.

○ **Pipettes:**

Pipettes are manufactured with different precisions. Combined estimate for a typical pipette is +/- 0.06 cm<sup>3</sup>. The error due to the use of a pipette would depend on its volume.

Eg: The error associated with using a 25 cm<sup>3</sup> pipette will be:

$$\% \text{ error} = \frac{0.06 \text{ cm}^3}{25 \text{ cm}^3} \times 100 = 0.24 \%$$

○ **Burettes :**

Burettes read to 0.05 cm<sup>3</sup>. The error is associated with the amount of volume measured. The smaller the volume larger the error. This is why it is usual to obtain the end-point when approximately 25cm<sup>3</sup> of solution have been titrated. The error allowing for the uncertainties of two scale readings +/- 0.10 cm<sup>3</sup>

$$\% \text{ error} = \frac{0.1 \text{ cm}^3}{25 \text{ cm}^3} \times 100 = 0.4\%$$

○ **Volumetric flask:**

Error depends on the size of the flask used. If a 250cm<sup>3</sup> flask is accurate to +/- 0.3cm<sup>3</sup> the error would be:

$$\% \text{ error} = \frac{0.3 \text{ cm}^3}{250 \text{ cm}^3} \times 100 = 0.12\%$$

**The overall error** is found by adding together the above individual errors of each apparatus.

**Calculating and combining uncertainties**

In most experiments the final results are calculated from a number of measurements. The total uncertainty is determined by combining the individual uncertainties.

**Uncertainties when measurements are added or subtracted**

When measurements are added or subtracted in calculating a result, the measurement uncertainties can be added to obtain the total uncertainty and percentage uncertainty.

*Example*

In a thermochemistry experiment, a 0–50°C thermometer graduated every 0.2 degrees were used to take the temperature. The readings before and after a reaction were 19.3°C and 27.8°C.

The uncertainty in each of these readings is  $\pm 0.1^\circ\text{C}$ , so the temperature rise is  $8.5^\circ\text{C}$  and the total uncertainty is  $\pm 0.2^\circ\text{C}$ .

$\therefore$  Temperature rise =  $8.5 \pm 0.2^\circ\text{C}$

The percentage uncertainty is given by:

$$\text{Percentage uncertainty} = \frac{\text{Uncertainty in the value} \times 100\%}{\text{Value}}$$

For the temperature rise, the percentage uncertainty is therefore given by:

$$\text{Percentage uncertainty in temperature rise} = \frac{0.2}{8.5} \times 100\% = 2.4\%$$

The percentage uncertainty in the experiment could also be reduced by planning to have a greater temperature rise. If, for example, the temperature rise had been  $15^\circ\text{C}$  rather than  $8.5^\circ\text{C}$ , then:

$$\text{Percentage uncertainty in temperature rise} = \frac{0.2}{15} \times 100\% = 1.3\%$$

---

---

## **Significant Figures**

Rules for assigning significant figures:

- For whole numbers & decimals of numbers greater than 1, all the figures, including any zeros at the end, are counted. Eg:  
503 is written to 3 significant figures (s.f)      2.03 is written to 3 s.f,  
1.020 is written to 4 s.f,                              200 is written to 3 s.f.
  - For decimals in numbers less than 1, count all the numbers, except the zeros before & immediately after the decimal point. Eg:  
0.402 is written to 3 s,f                              0.2020 is to 4 s,f  
0.00308 is written to 3 s,f                              0.02200 is written to 4 s,f
  - For values written in scientific notation, all the numbers before & after the decimal place are counted as significant figures. The power of 10 is not counted. Eg:  
 $6.02 \times 10^{-3}$  is written to 3 s,f
- 
-

## 1. The difference between Hazard & Risk

- Hazard is the potential of a substance to cause damage.
- Risk is a measure of the probability of that an adverse effect may occur when exposed.

A substance will have the same hazard in all situations due to its chemical properties. However differences may exist in the risk depending on how the substance is handled, protective measures used & other conditions that result in limited exposure.

Risk assessment is an action or a series of actions taken to recognize or identify hazards & to measure the risk or probability that something will happen because of that hazard.

Risk assessment provide us with the information we need to keep people safe laboratory. This also helps us to minimize any damage that would occur, when handling potentially hazardous chemicals.

## 2. Suggest ways by which risks can be reduced & reactions can be carried out safely

1) Working on a smaller scale:

Risk of a hazardous substance can be reduced or minimized by using them in smaller scale when carrying out reactions.

2) Taking specific precautions or using alternative techniques depending on the properties of the substance involved.

E.g.:

- I. When heating highly flammable substances (like ethanol) indirect heat such as water bath, oil bath or hot plate may be used.
- II. The use of fume hood. In reaction where toxic gases are evolved ( $\text{Cl}_2$ ,  $\text{Br}_2$ ,  $\text{SO}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{HCl}$ )
- III. Use of eye protection, gloves & lab coat when carrying out reactions.
- IV. Always add acid to water in dilution this prevents the acid from spattering.
- V. Point heating test tubes away from others & yourself & heating them gradually.

3) Carrying out the reactions using an alternative method, which involves less hazardous substance.

E.g. in the addition of HCN (hydrogen cyanide which is highly toxic) to carbonyl compounds use of  $\text{H}_2\text{SO}_4$  & KCN instead of HCN.  $\text{H}_2\text{SO}_4$  & KCN will react to form HCN safely in the reaction vessel hence no need to handle HCN.