



Practical Lab Manual of Organic Estimations and



Organic Qualitative Analysis



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CHAPTER I

INTRODUCTION OF THE LABORATORY SAFETY

Before you begin working in the chemistry laboratory, your instructor should review the safety rules and guidelines tell you what safety supplies, such as safety goggles and protective gloves you will need to use in the lab. In a first time working in a during laboratory period, the instructor will show you where safety equipment is located and tell you how to use it. As you locate each item, check it off the following list and make a note of its location and proper working, which is not expire:

1. Fire extinguishers.
2. Fire blanket.
3. Safety shower.
4. Eyewash fountain.
5. First aid supplies.
6. Spill cleanup supplies.

Every student and academician you should also learn the locations of chemicals, glassware consumable supplies (such as filter paper and boiling chips), waste containers, and various items of equipment such as balances and drying oven. If you find any glassware items with chips, cracks, or star fractures, you should have them replaced; they may cause cuts, break on heating, or shatter under stress. If necessary, clean up any dirty glassware and organize it neatly at this time.

Proper Working Efficiently

If all labs were geared to the slowest student, the objectives of the course could not be accomplished in the limited time available. Because of wide variations in individual working rates, it is usually not possible to schedule experiments so that everyone can finish in the allotted time. If you fall behind in the lab, you may need to put in extra hours outside your scheduled laboratory period in order to complete the course. The following suggestions should help you work more efficiently and finish each experiment on time:

Be prepared to start the experiment the moment you reach your work area

Don't waste precious minutes at the start of a laboratory period doing calculations, reading the experiment, washing glassware, or carrying out other activities that should have been done at the end of the previous period or during the intervening time. The first half hour of any lab period is the most important – if you use it to collect the necessary materials, set up the apparatus, and get the initial operation (reflux, distillation, etc.) under way, you should have no trouble completing the experiment on time.

Organize your time efficiently

Schedule a time each week to read the experiment and operation descriptions and to complete the pre-lab assignment - an hour before the lab period begins is too late. Plan ahead so that you know approximately what you will be doing at each stage of the experiment. A written experimental plan is invaluable for this purpose.

Organize your work area

Before performing any operation, arrange all of the equipment and supplies you will need during the operation neatly on your benchtop, in the approximate order in which they will be used. Place small objects and any items that might be

contaminated by contact with the benchtop on a paper towel, laboratory tissue, or mat. After you use each item, move it to an out-of-the way location where it can be cleaned and returned to its proper location when time permits; for example, put dirty glassware in a washing trough in the sink.

Getting along in the Laboratory

You will get along much better in the laboratory if you can maintain peace and harmony with your coworkers – or at least keep from aggravating them and stay on good terms with your instructor. Following these commonsense rules will help you do that:

Leave all chemicals where you can find them

You will understand the reason for this rule once you experience the frustration of hunting high and low for a reagent, only to find it at another student's station in a far corner of the lab.

Take only what you need

Whenever possible, liquids and solutions should be obtained using pipets, graduated cylinders, or other measuring devices so that it will take no more than you expect to use for a given operation.

Prevent contamination of chemicals

Do not use your own pipet or dropper to remove liquids directly from stock bottles, and don't return unused chemicals to stock bottles. Be sure to close all bottles tightly after use - particularly those that contain drying agents and other anhydrous chemicals.

If you must use a burner, inform your neighbors

Unless they are already using burners. This will allow them to cover any containers of flammable solvents and take other necessary precautions. In some

circumstances, you may have to use a different heat source, move your operation to a safe location (for instance under a fume hood), or find something else to do while flammable solvents are in use.

Return all community equipment to the designated locations

This may include ring stands, lab kits, clamps, condenser tubing, and other items. Because such items will be needed by students in other lab sections, they should always be returned to the proper storage area at the end of the period.

Clean up for the next person

Clean off the benchtop with a towel or wet sponge; remove condenser tubing, other supplies, and debris from the sink; and thoroughly wash any dirty glassware that is to be returned to the stockroom. Clean up any spills and broken glassware immediately. If you spill a corrosive or toxic chemical, such as sulfuric acid or aniline, inform the instructor before you attempt to clean it up. It is advised to maximize the labour and minimize the oratory while in the laboratory. This does not mean that all conversation must come to a halt. Quiet conversation during a lull in the experimental activity is okay, but a constant stream of chatter directed at a coworker who is performing a delicate operation is distracting and can lead to an accident. For the same reason, radios, CD or MP3 players and other audio devices must not be brought into the laboratory.

To Preventing Laboratory Accidents

Most of the organic chemistry lab courses are completed without incident, apart from minor cuts or burns, and serious accidents are rare. Nevertheless, the potential for a serious accident always exists. To reduce the likelihood of an accident. Additional safety rules or revisions of these rules may be provided by your instructor.

Always wear approved eye protection in the laboratory

Even when you aren't working with hazardous materials another student's actions could endanger your eyes, so never remove your safety goggles or safety glasses until you leave the lab. Do not wear contact lenses in the laboratory because chemicals splashed into an eye may get underneath a contact lens and cause damage before the lens can be removed. Properly determine the location of the eyewash fountain nearest to you during the first laboratory session and learn how to use it.

Never smoke in the laboratory or use open flames in operations that involve low boiling flammable solvents

Anyone found smoking in an organic chemistry laboratory is subject to immediate expulsion. Before you light a burner or even strike a match, inform your neighbors of your intention to use a flame. If anyone nearby is using flammable solvents, either wait until he or she is finished or move to a safer location, such as a fume hood. Diethyl ether and petroleum ether are extremely flammable, but other common solvents, such as acetone and ethanol, can be dangerous as well. When ventilation is inadequate, the vapours of diethyl ether and other highly volatile liquids can travel a long way; lighting a burner at one end of a lab bench that has an open bottle of ether at its other end has been known to start an ether fire.

Consider all chemicals to be hazardous and minimize your exposure to them

No and never taste chemicals, do not inhale the vapours of volatile chemicals or the dust of finely divided solids, and prevent contact between chemicals and your skin, eyes and clothing. Many chemicals can cause poisoning by ingestion, inhalation, or absorption through the skin. Strong acids and bases, bromine, thionyl chloride, and other corrosive material scan produce severe burns and require special precautions, such as wearing gloves and lab coat. Some

chemicals cause severe allergic reactions, and others may be carcinogenic (tending to cause cancer) or teratogenic (tending to cause birth defects) by inhalation, ingestion (swallowing) or skin absorption. To prevent accidental ingestion of toxic chemicals, don't bring food or drink into the laboratory or use mouth suction for pipetting, and wash your hands thoroughly after handling any chemical. To prevent inhalation of toxic or carcinogenic chemicals, work under an efficient fume hood or use a gas trap to keep chemical fumes out of the laboratory atmosphere. To prevent contact with corrosive or toxic chemicals, wear appropriate gloves and a lab coat.

Basic Safety Rules

These basic rules provide behaviour, hygiene, and safety information to avoid accidents in the laboratory.

1. Know locations of laboratory safety showers, eyewash stations, and fire extinguishers. The safety equipment may be in the hallway near the laboratory entrance.
2. Know emergency exit routes.
3. Avoid skin and eye contact with all chemicals.
4. Minimize all chemical exposures.
5. No horseplay will be tolerated.
6. Assume that all chemicals of unknown toxicity are highly toxic.
7. Post warning signs when unusual hazards, hazardous materials, hazardous equipment, or other special conditions are present.
8. Avoid distracting or startling persons working in the laboratory.
9. Use equipment only for its designated purpose.
10. Combine reagents in their appropriate order, such as adding acid to water.

11. Avoid adding solids to hot liquids.
12. All laboratory personnel should place emphasis on safety and chemical hygiene at all times.
13. Never leave containers of chemicals open.
14. All containers must have appropriate labels. Unlabeled chemicals should never be used.
15. Do not taste or intentionally sniff chemicals.
16. Never consume and/or store food or beverages or apply cosmetics in areas where hazardous chemicals are used or stored.
17. Do not use mouth suction for pipetting or starting a siphon.
18. Wash exposed areas of the skin prior to leaving the laboratory.
19. Long hair and loose clothing must be pulled back and secured from entanglement or potential capture.
20. No contact lenses should be worn around hazardous chemicals – even when wearing safety glasses.
21. Laboratory safety glasses or goggles should be worn in any area where chemicals are used or stored. They should also be worn any time there is a chance of splashes or particulates to enter the eye. Closed toe shoes will always be worn in the laboratory. Perforated shoes or sandals are not appropriate.
22. Do not utilize fume hoods for evaporations and disposal of volatile solvents.
23. Perform work with hazardous chemicals in a properly working fumehood to reduce potential exposures.

24. Avoid working alone in a building. Do not work alone in a laboratory if the procedures being conducted are hazardous.
25. If an unknown chemical is produced in the laboratory, the material should be considered hazardous.
26. Do not pour chemicals down drains. Do not utilize the sewer for chemical waste disposal.
27. Keep all sink traps (including cup sink traps and floor drains) filled with water by running water down the drain at least monthly.
28. Access to laboratories and support areas such as stockrooms, specialized laboratories, etc. should be limited to approved personnel only.
29. All equipment should be regularly inspected for wear or deterioration.
30. Equipment should be maintained according to the manufacturer's requirements and records of certification, maintenance, or repairs should be maintained for the life of the equipment.
31. Designated and well-marked waste storage locations are necessary.

CHAPTER II

THE BASIC GLASSWARE USED IN THE LABORATORY

Beakers

Beakers are used to mix chemicals, dissolve solids into solutions, heat or cool solutions and measure liquid volume in mL. They have very poor accuracy and should only be used to estimate volumes.

Erlenmeyer flasks

Like beakers, Erlenmeyer flasks can be used to mix, dissolve solids into solutions, and heat or cool solutions. Unlike beakers, Erlenmeyer flasks were developed for swirling solutions with minimum loss and they can be plugged with stoppers and used to catch vapour or condensed liquid. Their graduations are not very accurate.

Graduated Cylinders

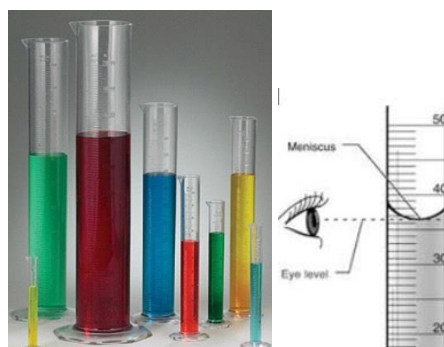
Graduated Cylinders are used to more accurately measure volumes of liquids in milliliters (mL). Measurement readings should be taken at eye level from the bottom of the meniscus, the concave curved surface formed at the top of the liquid.



Beaker



Erlenmeyer flasks



Graduated Cylinders

Volumetric Flask

A Volumetric Flask is used to accurately prepare solutions. A weighed amount of solid chemical is placed at the bottom, and the liquid solvent is added until the fill line is reached. The volumetric flask shown here is used to prepare a 250-mL solution at the fill line.

Pipet (Pipette)

Pipets are used to measure or transfer liquids. Mohr pipets (shown) are graduated pipets used for precise measurement. A pipet pump (top) or a pipet bulb (bottom) is attached to the end of the pipet to help draw up and release liquid.

Dropper

A dropper is a glass tube with a hollow rubber bulb at one end and a small opening at the other, for drawing in a liquid and expelling it in drops.

Buret (Burette)

A Buret is an extremely accurate device with a stopcock at the bottom used to measure volumes of reagents. Burets are often used to determine the concentration of a solution by dispensing a solution with a known concentration in a process known as titration. (Shown with Ring Stand and Double Buret Clamp)



**Volumetric
Flask**



Pipet (Pipette)



Dropper



**Buret
(Burette)**

Ring Stand

A ring stand is a metal stand consisting of a long upright rod attached to a heavy rectangular base that is used with rings and clamps for supporting laboratory apparatus.

Test Tube Rack

Test tube racks are used for holding test tubes upright when they contain chemicals, and also hold test tubes upside down to dry. They can be made of wood, metal, or plastic.

Test Tube

Test tubes are narrow glass cylinders, closed at one end, that are used for mixing, heating, and storing small quantities of chemicals. They are usually made of a type of glass that can withstand high temperatures. Test tubes come in many different sizes.

Test Tube Brush

A test tube brush is used for cleaning test tubes and narrow mouth laboratory glassware, such as beakers and flasks.



Ring Stand



Test Tube Rack



Test Tube



**Test Tube
Brush**

Funnel

Funnels are used to pour liquids into narrow mouth containers such as flasks and bottles.

Buchner funnels

Buchner funnels are special two-piece funnels for filtering mixtures through filter paper into a filtering flask.

Watch Glass

Watch glasses can be used to cover beakers, evaporate water from solutions, weigh out solid chemicals, or observe samples under a stereo microscope.

Bunsen Burner

A Bunsen burner is attached to a gas line and lit to provide heat for performing experiments.

Wire Gauze

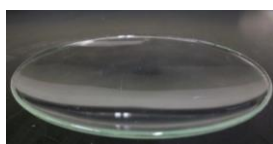
A Wire gauze is a sheet of thin metal that has net-like crosses or a wire mesh. The purpose of wire gauze is to be placed on the support ring that is attached to the ring stand between the Bunsen burner and the beakers to diffuse the heat and to support the beakers or other glassware or flasks during heating.



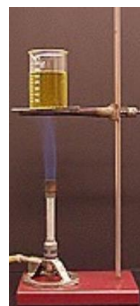
Funnel



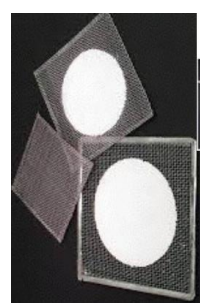
**Buchner
funnels**



Watch Glass



**Bunsen
Burner**



Wire Gauze

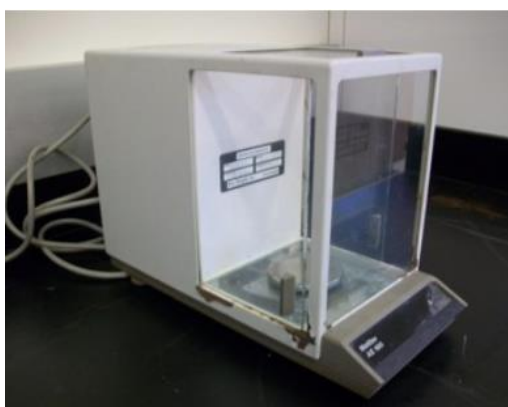
Analytical Balance

Analytical balances are much more precise and cost much more than toploading balances. They are sensitive to vibrations (be still and do not lean on the lab bench) and to air flow (close the glass doors).

Most of the analytical balances provided by AL Science in Motion can mass substances to 4 decimal places (0.0001g).

Wash Bottle

A wash bottle is used to store and dispense distilled or deionized water.



Analytical Balance



Wash Bottle

CHAPER III

ORGANIC ESTIMATION

S.No	Article to be weighed	Weights				Correct weight (gm)
		Gram	Milligram	Rider of		
				MD	SD	
1.	Crucible + $K_2Cr_2O_7$					
2.	Empty crucible					

250ml of $K_2Cr_2O_7$ contains =

1000ml of $K_2Cr_2O_7$ contains =

Strength of $K_2Cr_2O_7$ =

= _____ N.

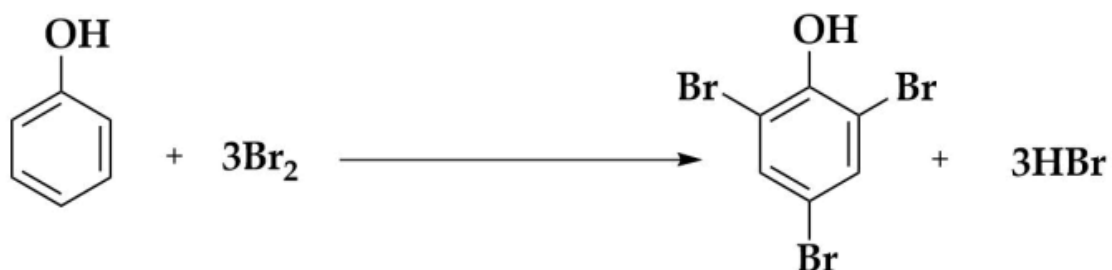
ESTIMATION OF PHENOL

AIM

To estimate the amount of Phenol present in the whole of the given solution.

PRINCIPLE

Phenol is readily brominated by a known excess of acidified bromide-bromate mixture to give 2,4,6-tribromophenol. The excess unreacted bromine liberates I_2 from KI and the liberated iodine is titrated against standard thiosulphate solution. From the quantity of bromine consumed, the amount of phenol present is calculated. For bromination, the bromide-bromate solution is preferred instead of bromine, because the latter loses its strength on keeping due to volatility of bromine.



PROCEDURE

PREPARATION OF STANDARD POTASSIUM DICHROMATE SOLUTION

About 1.25gm of potassium dichromate is accurately weighted in a chemical balance, dissolved in distilled water and made up to the mark in 250ml SMF.

STANDARDIZATION OF THE SODIUM THIO SULPHATE

STD $K_2Cr_2O_7$ Vs SODIUM THIO SULPHATE

$$\text{Strength of Standard } K_2Cr_2O_7 \text{ solution} = \frac{\text{Weight X 4}}{49.04}$$
$$= \text{_____ N}$$

S. NO	Volume of $K_2Cr_2O_7$	Burette Reading		Volume of thiosulphate(ml)	Indicator
		Initial (ml)	Final (ml)		
1.					
2.					
3.					

Volume of standard $K_2Cr_2O_7$ solution (V1) = 20 ml

Normality of standard $K_2Cr_2O_7$ solution (N1) = _____

Volume of Sodium Thio Sulphate solution (V2) = _____

Normality of Sodium Thio Sulphate solution (N2) = ?

$$V1 \times N1 = V2 \times N2$$

$$N2 = \frac{V1 \times N1}{V2}$$

$$N2 = \frac{20 \times N1}{(x) \text{ ml}}$$

$$N2 = \text{_____ N}$$

STANDARDISATION OF SODIUM THIO SULPHATE

The burette is filled with the Sodium Thio Sulphate solution upto the zero mark. 20ml of Potassium Dichromate solution is pipetted out into a clean conical flask. About 20ml of dil H_2SO_4 is added followed by the addition of 10ml of 10% KI solution. Now the liberated Iodine is titrated against Sodium Thio Sulphate solution in the burette. 1ml of starch indicator is added as the solution become straw yellow in colour. The end point is just disappearance of green colour. Titer values are noted. Titrations are repeated for concordant values. From this titer value, the strength of Sodium Thio Sulphate can be calculated.

ESTIMATION OF PHENOL

The burette is filled with the same solution of Sodium Thio Sulphate upto the mark, 20ml of Phenol is pipetted out into a clean Iodine flask. Then 5ml of analar HCl is added to the flask. Then the iodine flask is immediately closed by means of stopper and it is shaken well. Then it is left undisturbed for 10 minutes. Then about 10ml of 10% KI is added. The liberated Iodine is titrated against thio sulphate using starch as the indicator. The end point is the just disappearance of blue colour. Titrations are repeated for concordant values. From these values the strength of Phenol can be calculated.

A blank titration is also performed with 20ml of Winkler's solution.

PHENOL Vs STANDARD SODIUM THIO SULPHATE

S.No	Volume of Phenol (ml)	Burette reading		Volume of Sodium Thio Sulphate (ml)	Indicator
		Initial (ml)	Final (ml)		
1.					
2.					
3.					

Volume of Sodium Thio Sulphate =

WINKLER'S SOLUTION Vs STD SODIUM THIOSULPHATE

S.No	Volume of Winkler's solution (ml)	Burette reading		Volume of Sodium Thio Sulphate (ml)	Indicator
		Initial (ml)	Final (ml)		
1.					
2.					
3.					

Volume of Sodium Thio Sulphate =

BLANK TITRATION

The burette is filled with the same solution of Sodium Thio Sulphate. 20ml of brominating mixture is pipetted out into a clean iodine flask. About 5ml of analar Hydro Chloric acid is added. Then the mixture is immediately closed by the means of a stopper and it is shaken well. Then about 10ml of 10% Potassium Iodide is added. The liberated Iodine is immediately titrated against Sodium Thio Sulphate solution in the burette using Starch as the indicator. End point is the just disappearance of blue colour. The titrate values are noted. The titrations are repeated for concordant values.

CALCULATION

Volume of Sodium Thio Sulphate needed for 20ml Winkler's solution =

Volume of Sodium Thio Sulphate needed for 40ml Winkler's solution =

Volume of Sodium Thio Sulphate needed for excess of Winkler's solution =

Volume of Phenol V_2 =

Strength of Phenol N_2 =

Volume of thio equivalent for Aniline V_1 =

Strength of thio sulphate N_1 =

Strength of Phenol N_2 = $N_1 V_1 / V_2$

= _____ N

Amount of Phenol present in 1000ml =

Amount of Phenol present in 100ml = _____ g.

RESULT

The amount of Phenol present in the whole of the given solution = _____g.

S.No	Article to be weighed	Weights				Correct weight (gm)
		Gram	Milligram	Rider of		
				MD	SD	
1.	Crucible + $K_2Cr_2O_7$					
2.	Empty crucible					

250ml of $K_2Cr_2O_7$ contains =

1000ml of $K_2Cr_2O_7$ contains =

Strength of $K_2Cr_2O_7$ =

= _____ N.

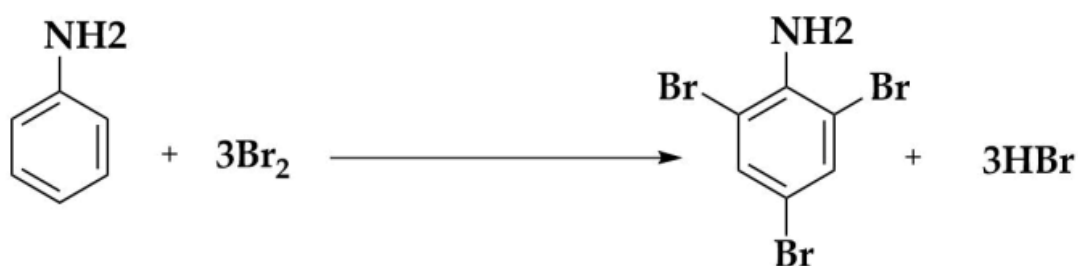
ESTIMATION OF ANILINE

AIM

To estimate the amount of Aniline present in the whole of the given solution.

PRINCIPLE

Like Phenol, Aniline also easily brominated by a known excess of acidified bromide-bromate mixture to give 2,4,6-tribromoaniline or s-tribromoaniline. The excess unreacted bromine liberates Iodine when Potassium Iodide solution is added and the liberated Iodine is titrated against standard thiosulphate solution.



PROCEDURE

PREPARATION OF STANDARD POTASSIUMDICHROMATE SOLUTION

About 1.25gm of Potassium Dichromate is accurately weighted in a chemical balance, dissolved in water and made up to the mark in a 250ml SMF.

STANDARDIZATION OF THE SODIUM THIO SULPHATE

STD $K_2Cr_2O_7$ Vs SODIUM THIO SULPHATE

$$\text{Strength of Standard } K_2Cr_2O_7 \text{ solution} = \frac{\text{Weight X 4}}{49.04}$$

$$= \text{_____ N}$$

S. NO	Volume of $K_2Cr_2O_7$	Burette Reading		Volume of thiosulphate(ml)	Indicator
		Initial (ml)	Final (ml)		
1.					
2.					
3.					

Volume of standard $K_2Cr_2O_7$ solution (V1) = 20 ml

Strength of standard $K_2Cr_2O_7$ solution (N1) = _____

Volume of Sodium Thio Sulphate solution (V2) = _____

Strength of Sodium Thio Sulphate solution (N2) = ?

$$V1 \times N1 = V2 \times N2$$

$$N2 = \frac{V1 \times N1}{V2}$$

$$N2 = \frac{20 \times N1}{(x) \text{ ml}}$$

$$N2 = \text{_____ N}$$

STANDARDISATION OF SODIUM THIO SULPHATE

The burette is filled with the Sodium Thio Sulphate solution upto the mark. 20ml of Potassium Dichromate solution is pipetted out into clean conical flask. About 20ml of dil H_2SO_4 is added followed by the addition of 10ml of 10% KI solution. Now the liberated Iodine is titrated against Sodium Thio Sulphate solution in the burette. 1 ml of starch indicator is added as the solution become straw yellow in colour. The end point is just appearance of blue colour. Titrated values are noted. Titrations are repeated for concordant values. From this titrated value, the strength of Sodium Thio Sulphate can be calculated.

ESTIMATION OF ANILINE

The burette is filled with the same solution of Sodium Thio Sulphate upto the mark, 20ml of Aniline is pipetted out into a clean Iodine flask. 40ml of brominating mixture is pipetted out into the same iodine flask, then 5ml of analar HCl is added to the flask. Then the iodine flask is immediately closed by means of stopper and it is shaken well and then it left undisturbed to 10 minutes. Then about 10ml of 10% KI is added. The liberated iodine is titrated against thio sulphate using starch as the indicator. The end point is the just disappearance of blue colour. Titrations are repeated for concordant values. From these values the strength of aniline can be calculated.

A blank titration is also performed with 20ml of Winkler's solution.

ANILINE Vs STANDARD SODIUM THIO SULPHATE

S.No	Volume of Aniline (ml)	Burette reading		Volume of Sodium Thio Sulphate (ml)	Indicator
		Initial (ml)	Final (ml)		
1.					
2.					
3.					

Volume of Sodium Thio Sulphate =

WINKLER'S SOLUTION Vs STD SODIUM THIOSULPHATE

S.No	Volume of Winkler's solution (ml)	Burette reading		Volume of Sodium Thio Sulphate (ml)	Indicator
		Initial (ml)	Final (ml)		
1.					
2.					
3.					

Volume of Sodium Thio Sulphate =

BLANK TITRATION

The burette is filled with the same solution of Sodium Thio Sulphate. 20ml of brominating mixture is pipetted out into a clean Iodine flask. About 5ml of analar Hydro Chloric acid is added. Then the iodine flask is immediately closed by the means of a stopper and it is shaken well. Then about 10ml of 10% Potassium Iodide is added. The liberated iodine is immediately titrated against Sodium Thio Sulphate solution taken in the burette using starch as the indicator. End point is the just disappearance of blue colour. The titrate values are noted. The titrations are repeated for concordant values.

CALCULATION

Volume of Sodium Thio Sulphate needed for 20ml Winkler's solution =

Volume of Sodium Thio Sulphate needed for 40ml Winkler's solution =

Volume of Sodium Thio Sulphate needed for excess of Winkler's solution =

Volume of Aniline V_2 =

Strength of Aniline N_2 =

Volume of thio equivalent for Aniline V_1 =

Strength of thio sulphate N_1 =

Strength of Aniline N_2 = $N_1 V_1 / V_2$

= _____ N

Amount of Aniline present in 1000ml =

Amount of Aniline present in 100ml = _____ g.

RESULT

The amount of Aniline present in the whole of the given solution = _____g.

ORGANIC QUALITATIVE ANALYSIS

CHAPTER IV

ORGANIC QUALITATIVE ANALYSIS

Introduction

The aim of organic qualitative analysis is to identify a given organic substance and to confirm its nature by performing a set of known standard reactions with it. The entire process of this analysis is based on the two important theoretical concepts, namely, Homologous series and Functional group.

Millions of Known organic compounds are easily and effectively classed into a limited number of (25 or 30) groups depending on their nature of functional group present. A compound belonging to a particular class will exhibit characteristic reactions of the atoms or group of atoms present in it.

When compounds of a class are arranged in the increasing order of their molecular weight, a homologous series result. Compounds of a particular homologous series show a similarity in chemical reactions and gradation in physical properties. Thus, a compound's position in a homologous series reveals its physical characteristics and chemical nature.

In general, organic compounds are considered to be derivatives of hydrocarbons (aliphatic or aromatic). The substituents or linking atoms or groups in an organic molecule are invariably inorganic atoms or groups such as halogens, oxygen and hydrogen, nitrogen and hydrogen, sulphur, etc., which lead to the formation of halogen compounds, alcohols, aldehydes, ketones, esters, amines, amides, thiols, etc., A good understanding of the reactions of this simple class of organic compounds is an essential requirement before attempting to take up qualitative analysis of substance.

S.No	EXPERIMENT	OBSERVATION	INFERENCE
1.	<u>COLOUR:</u> Note the colour of the given substance.	Yellow Dark brown Colourless	Presence of aromatic nitro group May be phenols and aromatic amines May be carbohydrates, amides, acids etc.
2.	<u>ODOUR:</u> Odour of the substance is noted.	Fishy smell Phenolic odour Bitter almond smell	May be Amines. May be Phenols. Benzaldehyde (or) Nitrobenzene.
3.	<u>LITMUS TEST:</u> Add 5ml of water to 10mg of compound and shake well. Test the solution with litmus paper. (If the given compound exists in liquid phase, place one drop of it on the litmus paper.)	Blue litmus turns red Red litmus turns blue	Carboxylic acid (or) Phenol. Amines.
4.	<u>SOLUBILITY:</u>		
a.	To 100mg of the given compound add 50ml of water and shake well.	Soluble in water	May be carbohydrates (or) Diamide.
b.	Add 2ml of saturated solution of sodium bicarbonate to 100mg of the given compound.	Substances dissolves with effervescence	May be carboxylic acids.
c.	Add 2ml of 10% sodium hydroxide solution to 100mg of the given substance.	Substance dissolves and regenerated by the addition of concentrated Hydrochloric acid	May be phenols.
d.	Add 2ml of dilute hydrochloric acid to the given compound.	Substance dissolves and regenerated by the addition of aqueous sodium hydroxide	May be amines.

S.No	EXPERIMENT	OBSERVATION	INFERENCE
5.	<u>TEST FOR ALIPHATIC AROMATIC NATURE:</u> Take 100mg [5 drops if liquid] of the given organic compound in nickel spatula. Ignite in non-luminous Bunsen flame.	Substance burns with sooty flame	Presence of Aromatic compound.
a.		Substance burns with non-sooty flame	Presence of Aliphatic compound.
b.	<u>NITRATION TEST</u> Take 100mg [5 drops if liquid] of a given organic compound. Add 1ml of con.Nitric acid and 1ml of con.H ₂ SO ₄ and heat it for 10 minutes, cool and pour the reaction mixture into a beaker containing water.	Yellow precipitate (or) yellow solution	Presence of Aromatic compound.
		No yellow precipitate (or) no yellow solution	Presence of Aliphatic compound.
6.	<u>TEST FOR SATURATION-UNSATURATION:</u> Place 100mg of the given substance in a test tube. Add 2ml of water and shake well. Introduce 3 drops of bromine water and shake well.	Brown colour of bromine disappears, with the formation of white precipitate	Saturated compound [aromatic primary amine (or)phenol].
a.		Brown colour of bromine remains as such	Saturated compound.
b.	To 100mg of the given substance in a test tube. Add 2ml of Carbon tetrachloride and shake well. Add 3 drops of bromine in CCl ₄ and shake well.	Brown colour of bromine vanishes	Unsaturated compound.
		Brown colour of bromine remains same	Saturated compound.
c.	Add 5ml of water to 100mg of the given compound and introduce 2 drops of 1% KMnO ₄ solution.	Pink colour disappears	Unsaturated compound.
		Pink colour persists	Saturated compound.

TEST FOR ELEMENTS:

PREPARATION OF SODIUM FUSION EXTRACT [LASSAIGNE'S TEST]:

Place a small piece of clean, dry sodium metal in a fusion tube. Heat well until the sodium metal melts and allow to cool. Add carefully 50mg of the given compound in the fusion tube. Heat well until the fusion tube becomes red hot. Immerse the red hot end of the fusion tube into 5ml of distilled water taken in a mortar. Break the bottom of the tube with the pestle and grind well. Transfer the contents of the mortar to a china dish and heat for 5 minutes. Filter and collect the filtrate. The clear filtrate is known as "Sodium Fusion Extract".

S.No	EXPERIMENT	OBSERVATION	INFERENCE
8.	<u>TEST FOR NITROGEN:</u> To 5 drops of the extract, 1ml of freshly prepared ferrous sulphate solution is added, heat, cooled and then acidified with dilute HCl.	Green (or) Prussian blue precipitate (or) solution.	Presence of Nitrogen.
9.	<u>TEST FOR SULPHUR:</u>		
a.	To 5 drops of the extract, add 1ml of acetic acid and 1ml of lead acetate solution.	Black precipitate	Presence of sulphur.
b.	To 5 drops of the extract add 2ml of sodium nitroprusside	Purple colour solution	Presence of sulphur is confirmed.
10.	<u>TEST FOR HALOGENS:</u>		
a.	Add 1ml of dilute nitric acid to 5 drops of the extract. To this add 1ml of silver nitrate solution. Yellow precipitate insoluble in aqueous ammonia.	Curdy white precipitate soluble in excess of aqueous ammonia	Presence of Chlorine. Presence of Bromine (or) Iodine.
b.	To 5 drops of extract, add 1ml of dil H_2SO_4 and 1ml of CCl_4 to this solution, then add 1ml of bromine water shake well and allow to stand for 5 minutes.	Reddish brown bottom layer Violet bottom layer	Presence of Bromine is confirmed. Presence of Iodine is confirmed.
11.	<u>NEUTRAL FERRIC CHLORIDE TEST:</u> Add 1ml of neutral ferric chloride solution to 100mg of the given substance.	Violet (or) blue or green colour.	Presence of Phenols.

S.No	EXPERIMENT	OBSERVATION	INFERENCE
12.	<u>ACTION OF CONCENTRATED SULPHURIC ACID:</u> Add 1ml of concentrated sulphuric acid to 5mg of the substance and then warmed well.	Substance chars	Presence of Carbohydrates.
13.	<u>TOLLEN'S REAGENT TEST:</u> Add 1ml of Tollen's reagent to 100 mg of the given compound and heat it on a water bath 10 minutes.	A bright silver mirror or black deposit	Presence of reducing sugars such as Carbohydrates(or) Aldehydes.
14.	<u>FEHLINGS REAGENT TEST:</u> Add 1ml of Fehling's A and B solutions to 100mg of the given compound and heat it on a water bath for 10 minutes.	Reddish brown precipitate	Presence of reducing sugars of Carbohydrates
15.	<u>LIBERMANN'S TEST:</u> To the substance, add sodium nitrate solution and con.H ₂ SO ₄ and heat to boil and pour into a beaker containing water and add alkaline sodium hydroxide solution.	Bluish green colour	Presence of Phenols.
16.	<u>TEST FOR α-NAPHTHOL:</u> To the substances, add few drops of dilute sodium hydroxide, glucose and con.H ₂ SO ₄ along the sides of the tube.	Violet ring	Presence of α -Naphthol.
17.	<u>TEST FOR β-NAPHTHOL:</u> To the substance, add dilute sodium hydroxide and aniline. To this mixture dilute Hydrochloric acid and Sodium nitrate are added and heated the mixture.	Scarlet red dye	Presence of β -Naphthol

S.No	EXPERIMENT	OBSERVATION	INFERENCE
1.	<u>TEST FOR FUNCTIONAL GROUP:</u> <u>TEST FOR CARBOXYLIC ACID:</u> The substance is treated with saturated sodium bicarbonate. To 100mg of the given compound, add 2ml of methyl alcohol. Then add 2 drops of con.H ₂ SO ₄ and heat the mixture on a water bath for 15 min. Then pour the mixture into a beaker containing sodium carbonate solution.	Brisk effervescence is obtained Sweet smell or fruity smell	Presence of Carboxylic acid. Presence of Carboxylic acid.
2.	<u>TEST FOR DICARBOXYLIC ACID:</u> <u>FLUORESCENCE TEST:</u> The substance is treated with equal amount of Resorcinol and few drops of con.H ₂ SO ₄ . The mixture is heated in a water bath gently until a deep red solution is formed and then allowed to cool. Pour the mixture into a beaker containing 5ml of dilute sodium hydroxide solution.	Greenish yellow fluorescence is obtained	Presence of Dicarboxylic acid.
3.	<u>TEST FOR ALDEHYDES & KETONES:</u> a. The substance in alcohol is heated with Broche's reagent [2,4 dinitro phenyl hydrazine] and few drops of concentrated hydrochloric acid is added and cooled. The mixture is shaken with Schiff's base reagent. b. The substance is warmed with Fehling's solution A&B on a water bath for 10 minutes.	Yellow or orange red precipitate is formed Pink colour appears in cold condition rapidly Reddish brown precipitate is obtained.	Presence of Aldehydes or Ketones. Presence of Aliphatic Aldehydes or reducing sugar. Presence of aliphatic Aldehydes or Reducing sugar.

S.No	EXPERIMENT	OBSERVATION	INFERENCE
c.	The substance is heated with Tollen's reagent and heat the mixture in water bath for 10 minutes.	A bright silver mirror or black precipitate is formed.	Presence of Aldehydes or Ketones.
d.	The substance is added with saturated solution of sodium bisulphate and shaken well.	White crystalline precipitate is formed.	Presence of Aldehydes or Ketones.
e.	The substance is added with semicarbazide, con.HCl, sodium acetate and alcohol. Then it is heated and cool.	Coloured crystals of semicarbazone is obtained.	Presence of Aldehydes & Ketones.
4.	<u>TEST FOR ESTER - HYDROXIMIC TEST:</u> The substance is mixed with methanoic solution of hydroxyl amine and hydrochloric acid. Then to the mixture sodium hydroxide, solution is added. Heated to boil and cool & add few drops of HCl to it. Then neutralize with ferricchloride solution.	Violet colour.	Presence of Esters.
5.	<u>TEST FOR CARBOHYDRATES:</u> To the substance add Tollen's reagent and heat it on a waterbath. NOTE:[Preparation of Tollen's reagent: 2ml of AgNO_3 + 1 to 2 drops of NaOH solution – a turbidity will appears, just dissolve this turbidity by adding ammonium hydroxide solution drop wise.]	Bright silver mirror is obtained.	Presence of Carbohydrate.
a.			
b.	To the substance add Fehling's[A+B] solution mixture. Then heat it on a water bath.	Red precipitate.	Presence of Monosaccharides(Glucose or Fructose)

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c.	<u>BARFOED'S TEST:</u> The substance in water add Barfoed's reagent and heat it on a water bath. (or) Barfoed's reagent [dil.copper acetate in acetic acid] is added to an aqueous solution of given substance and warmed on a water bath for 5 minutes.	Red precipitate. Red cuprous oxide is precipitated.	Presence of Carbohydrate. Presence of monosaccharide.
d.	<u>MOLISCH'S TEST:</u> To the substance in water add two drops of α -Naphthol and con.H ₂ SO ₄ is added along the sides of the test tube. (or) 2-3 drops of alcoholic solution of α -Naphthol is added to the substance in water, two drops of con.H ₂ SO ₄ is added slowly through the sides of the test tube without shaking.	Dark violet colour. A deep violet colouration is formed.	Presence of Carbohydrate. Presence of sugars.
6.	<u>TEST FOR PHENOLIC COMPOUNDS:</u> Substance in water or in alcohol add neutral ferric chloride solution.	Violet or brick or blue colour.	Presence of Phenol.
a.	<u>PHTHALEIN TEST:</u> To the substance add phthalic anhydride and con.H ₂ SO ₄ . The mixture is heated, cooled and poured into NaOH solution.	Pink colour Intense green fluorescence is seen.	Presence of Phenol. Presence of Dihydric Phenollike Resorcinol.
b.	<u>DYE TEST:</u> To aniline add con.HCl and sodium nitrate. Then the mixture is cooled to 0°C to the cooled mixture add the substance dissolved in NaOH.	A scarlet red dye is obtained.	Presence of Dihydric Phenol.

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c.	<u>LIBERMANN'S TEST:</u> The substance is warmed with sodium nitrate solution. Cooled and add con.H ₂ SO ₄ . It is then poured into 100ml beaker containing water and NaOH solution.	Blue or Green colour solution is obtained.	Presence of Phenol.
1. a.	<u>TEST FOR FUNCTIONAL GROUPS CONTAINING NITROGEN ATOM:</u> <u>TEST FOR AMINES:</u> <u>DYE TEST:</u> The substance is dissolve in dil.HCl. It is cooled to 0°C to 5°C. Add an ice cooled solution of sodium nitrate. To the above mixture add β-Naphthol solution in NaOH.	A scarlet red dye	Presence of Aromatic primaryamine.
b.	<u>LIEBERMANN'S TEST:</u> 1) Substance in dil.HCl, cool & add sodium nitrate solution. 2) To the yellow oily liquid add phenol add con.HNO ₃ then warm, cool and dilute with water. 3) The substance in dil.HCl sodium nitrate solution is added.	Yellow oily liquid separates. Blue colour is obtained It turns to red with NaOH solution. Brown orange precipitate. It turns to dark green with NaOH solution.	Presence of secondary amines. Presence of secondary amines. Presence of aromatic tertiaryamine.
c.	<u>CARBYLAMINE TEST:</u> To the substance add alcoholic potash add chloroform, Then heated.	An unpleasant smell of isocyanide is obtained.	Presence of primary amine.
2. a.	<u>TEST FOR NITRO COMPOUNDS:</u> <u>DYE TEST:</u> To the substance add con.HCl and a piece of tin metal. It is boiled and filtered. Cool the filtrate and to the filtrate add alkaline β-Naphthol solution.	Scarlet red dye is obtained.	Presence of Nitro compounds.

S.No	EXPERIMENT	OBSERVATION	INFERENCE
b.	<u>MULLIKEN'S TEST:</u> 1) The substance in alcohol add zinc dust and solid ammonium chloride and 10 drops of ammonium chloride. It is boiled and filtered. To this filtrate add Tollen's reagent and heat it again. 2) The above filtrate is heated with fehling's mixture [A+B]. 3) Substance in acetone, NaOH is added.	Bright silver mirror or black precipitate is obtained. Red precipitate is obtained. Violet colour appears, it changes to red on adding acetic acid.	Presence of nitro compounds. Presence of nitro compounds. Presence of polynitro compounds like meta-dinitro benzene.
3. a.	<u>TEST FOR AMIDES:</u> To the substance 10% NaOH is added and heated strongly.	Ammonia gas is liberated. White precipitate.	Presence of amide or diamide. Presence of aromatic amide.
b.	The above solution is cooled and con.HCl is added.	No precipitate.	May be an aliphatic amide.
	<u>BIURETE TEST</u> <u>TEST FOR DIAMIDE</u> <u>[UREA]:</u> Substance is heated till it melts. Ammonia is evolved, cool and add 2ml of water, dil.copper sulphate and 2ml of NaOH.	Violet colour appears.	Presence of diamide like urea.
4.	<u>TEST FOR ANILIDES:</u> 1) The substance in con.HCl is refluxed and cooled with sodium nitrate. This solution is coupled with alkaline β -Naphthol solution. 2) The substance with 10% NaOH solution is refluxed and cooled. Then add alcoholic potash and chloroform.	Scarlet red dye Unpleasant smell of isocyanide is liberated.	Presence of Anilide. Presence of Anilide.