



MARRI LAXMAN REDDY INSTITUTE OF PHARMACY

(Approved by AICTE & PCI, New Delhi and Affiliated to J.N.T. U, Hyderabad)

Dundigal (M) Medchal (Dist) Hyderabad- 500043

PHARMACEUTICAL ORGANIC CHEMISTRY- I

LAB MANUAL

About MLRIP



To be an educational Institute of par excellence and produce competent pharmacy professionals to serve the community through research and the ever-increasing needs of Industry.



1. Imparting quality education and innovative research for various career opportunities.
2. Creating conducive academic environment to produce competent pharmacy professionals.
3. Indoctrination of students adorned with high human values and make them aware of their responsibility as health care professionals.

Program Educational Objectives

PEO 1: To produce graduates with sound theoretical knowledge and technical skills required for their career opportunities in various domains.

PEO 2: To incite the students towards research and to address the challenges with their innovative contributions for the benefit of the mankind.

PEO 3: To instill the essence of professionalism, ethical commitment to become a health care professional with sound integrity and adherence to the core human values in the service of the society.



PROGRAM OUTCOMES

1. **Pharmacy Knowledge:** Possess knowledge and comprehension of the core and basic knowledge associated with the profession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.
2. **Planning Abilities:** Demonstrate effective planning abilities including time management, resource management, delegation skills and organizational skills. Develop and implement plans and organize work to meet deadlines.
3. **Problem analysis:** Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, while solving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions.
4. **Modern tool usage:** Learn, select, and apply appropriate methods and procedures, resources, and modern pharmacy-related computing tools with an understanding of the limitations.
5. **Leadership skills:** Understand and consider the human reaction to change, motivation issues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and well-being.
6. **Professional Identity:** Understand, analyze and communicate the value of their professional roles in society (e.g. health care professionals, promoters of health, educators, managers, employers, employees).
7. **Pharmaceutical Ethics:** Honour personal values and apply ethical principles in professional and social contexts. Demonstrate behavior that recognizes cultural and personal variability in values, communication and lifestyles. Use ethical frameworks; apply ethical principles while making decisions and take responsibility for the outcomes associated with the decisions.
8. **Communication:** Communicate effectively with the pharmacy community and with society at large, such as, being able to comprehend and write effective reports, make effective presentations and documentation, and give and receive clear instructions.
9. **The Pharmacist and society:** Apply reasoning informed by the contextual knowledge to assess societal, health, safety and legal issues and the consequent responsibilities relevant to the professional pharmacy practice.
10. **Environment and sustainability:** Understand the impact of the professional pharmacy solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
11. **Life-long learning:** Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change. Self-assess and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

List of Experiments

I. Systematic qualitative analysis of unknown organic compounds like

1. Preliminary test: Color, odour, aliphatic/aromatic compounds, saturation and unsaturation, etc.
2. Detection of elements like Nitrogen, Sulphur and Halogen by Lassaigne's test
3. Solubility test
4. Functional group test like Phenols, Amides/ Urea, Carbohydrates, Amines, Carboxylic acids, Aldehydes and Ketones, Alcohols, Esters, Aromatic and Halogenated Hydrocarbons, Nitro compounds and Anilides.
5. Melting point/Boiling point of organic compounds
6. Identification of the unknown compound from the literature using melting point/ boiling point.
7. Preparation of the derivatives and confirmation of the unknown compound by melting point/ boiling point.
8. Minimum 5 unknown organic compounds to be analysed systematically.

2. Preparation of suitable solid derivatives from organic compounds

3. Construction of molecular models

INTRODUCTION

Good Laboratory Practices:

1. Wear laboratory apron while working.
2. Use clean glassware.
3. Use strong acid and alkali carefully.
4. Do not displace the reagents from their respective places.
5. Do not interchange pipettes from one reagent to another without thorough cleaning.
6. Carry out the reactions carefully without harming the neighboring student.
7. Use the gas whenever necessary. Close the gas tap when not required.
8. Prepare your own reagents when necessary for correct results.
9. Do not consult your friends for doubts. Consults books and teachers for your problems.
10. Take signature of your teachers for all your assignments.
11. Leave the laboratory well prepared for the experiment concerned.
12. Write the laboratory records regularly

APPARATUS AND GLASS WARES

Before starting an experiment cleaning of glassware, equipment and bench is necessary. The bench should be kept clean and remove any solid or liquid chemicals. Glass ware are first washed with chemicals and then rinsed with distilled water before use. The outer surface may be dried with a cloth or filter paper. All the apparatus used in particular experiment should be kept together on the bench to avoid confusion in determining the duplicate experiments. Excess of apparatus which are not in use should be removed from the bench. All the solutions, precipitates and filtrates should be labeled to avoid confusion and covered to prevent contamination of the contents. Reagent bottles must be returned on the reagent shelves immediately after use. Experiment observations must be recorded in a shift covered notebook containing index page and remaining one sided line pages. The record must be concluded with calculations and results.

Glassware:

The convenient unit to measure large volumes of liquid is “liter”. The liter and milliliter are sufficiently precise for the requirement of titrimetric analysis. The capacity of a glass vessel may vary with temperature. Therefore, the temperature of the experiment is noted.

Graduated apparatus:

Graduated flasks, burettes and pipettes are the most commonly used apparatus in volumetric analysis. All these glassware must be perfectly clean from grease to get exact results. Many detergents are available to clean glass apparatus. Saturated solution of powdered sodium and potassium dichromate in conc. sulphuric acid is also used for cleaning purpose.

Volumetric flask:

A volumetric flask is a flat-bottomed, pear-shaped apparatus with a long-narrowed neck. A thin line mark around the neck indicates the volume that it holds at a certain definite temperature,

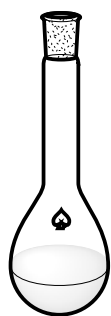
Usually, 20 degrees the capacity and the temperature are clearly marked on the flask. Both the front and the back of the mark should be seen as a single line to avoid the errors as parallax, when making the final adjustments. The lower edge of the meniscus should be tangential to the graduation mark. A small change in the volume is easily detected in a long narrow neck. Volumetric flasks are available in capacities of 1, 2, 5, 10, 25, 50, 100, 500, 1000, 2000 and 5000 cm³.

Pipette: Pipettes are of two types:

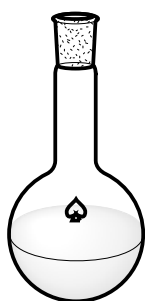
1. **Transfer/bulb/volumetric pipette:** which have one mark and withdraw a small and constant volume of solution.
2. **Measuring/graduated pipettes:** which are graduated and used to deliver various small volumes.

Filtration Apparatus:

- A conical flask and funnel fitted with a filter paper is usually used for filtration. The funnel should have angle nearby 60 degrees and a long stem.
- Sintered glass crucibles are made up of resistance glass and have porous disk fused into the body of crucible. The filtered disc is made up of various pore diameters as indicated by the numbers from 0-5.
- Buchner funnel is used to filter large quantities of materials. A good quality of filter paper is placed on a paper with sintered glass.



Narrow neck RBF



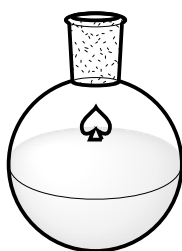
Long neck RBF flask



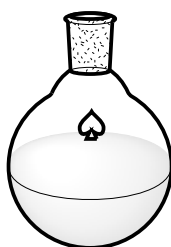
Pear shaped RBF flask



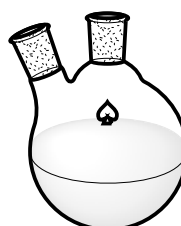
Volumetric Flask



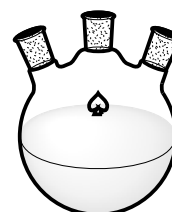
Round Bottomed Flask



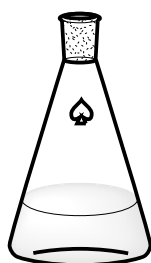
Round Bottomed Flask



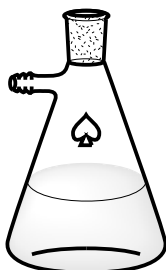
Two neck R.B Flask



Three neck R.B Flask



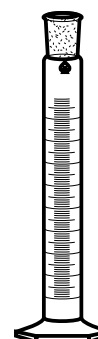
Erlenmeyer flask



Buchners Flask



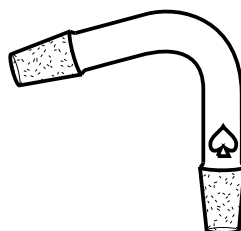
Beaker



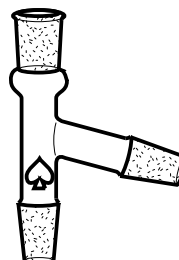
Measuring Cylinder



Thermometer adapter



Plain bend



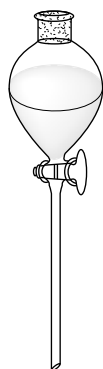
Distillation adapter



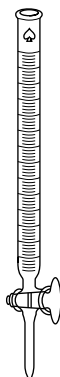
Vacuum adapter



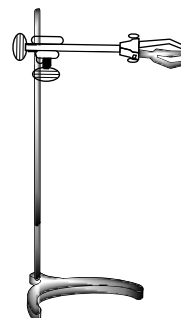
**Pear shaped
seperating funnel**



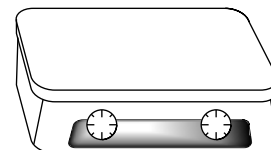
**globe shaped
seperating funnel**



titration burette



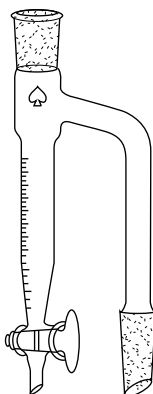
**burette
stand with clamp**



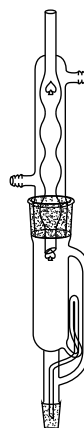
Heating Mantle



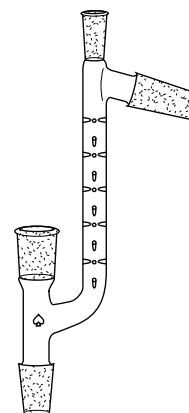
Liebig condenser



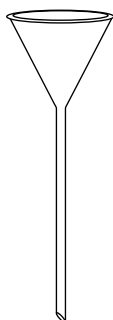
**clavengers apparatus
Condenser**



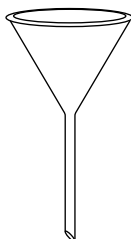
**Soxhlet with
Allihn condenser set**



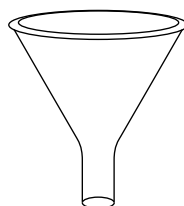
**claisen adapter
condenser**



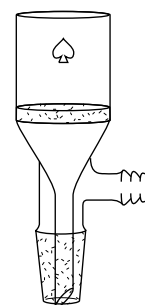
Long Stem Funnel



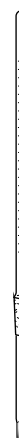
Short Stem Funnel



Cone powder funnel



Buchners Funnel



Thermometer

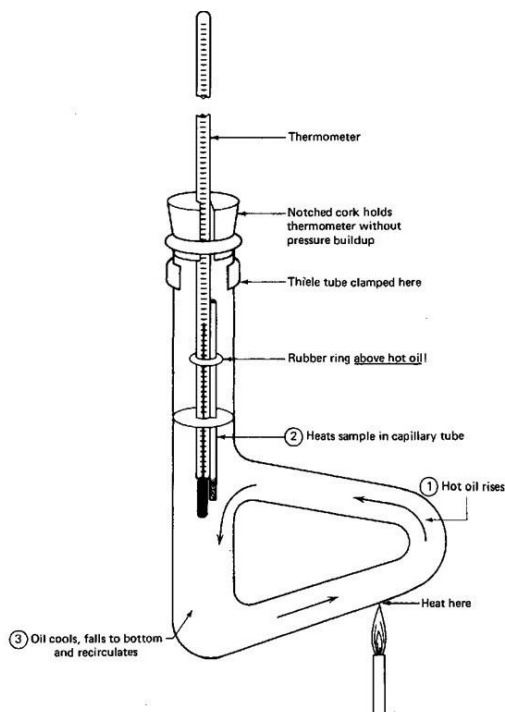
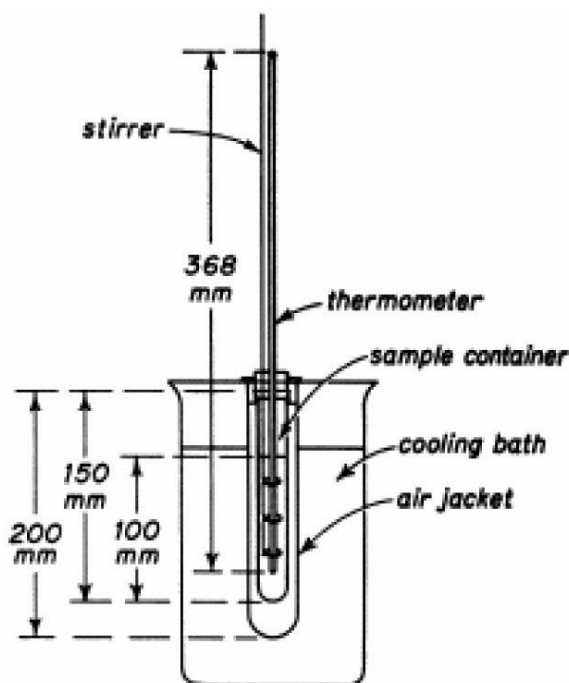
DETERMINATION OF MELTING POINT

AIM: To determine the melting point of given liquids.

APPARATUS: Thermometer, Capillary tube. Melting Point Apparatus.

PRINCIPLE: Melting point is the change from the highly order or arrangement of particles in the crystalline lattice to the more random arrangement that characterize a liquid. Melting occurs when the temperature reaches at which the thermal energy of the particles is great enough to overcome the intra crystalline forces that holds them in position.

PROCEDURE: A capillary tube is taken and its one end is closed by introducing into the tip of flame. Capillary tube is kept rotating during heating for the closing to be uniform. Care is taken to prevent the formation of bulb during heating.

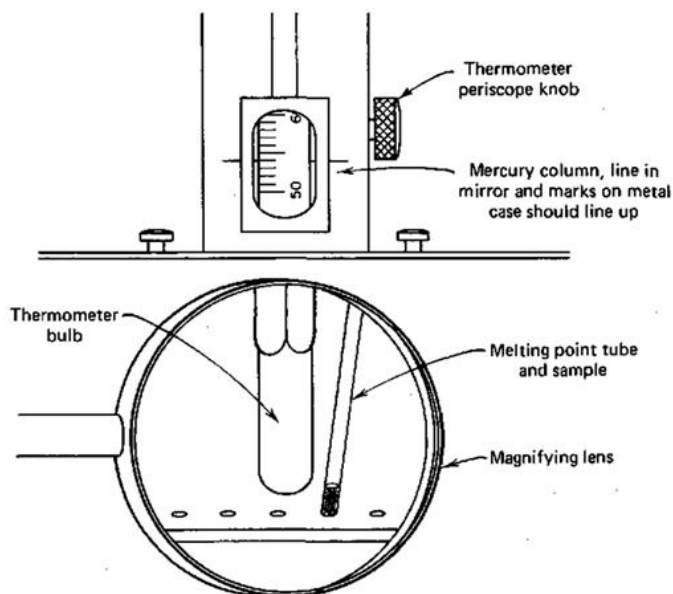


METHOD –I: The substance whose melting point is to be determined is introduced into capillary tube so that it occupies at least 3-5 mm of the length. The substance and capillary tube must be dried before actual procedure begins.

The filled capillary tube is tied to a thermometer with a thread such that the substance is against the mercury bulb of thermometer. This is introduced into a melting point apparatus which is filled with liquid paraffin or concentrated sulphuric acid. The liquid is heated gently using a blue flame. Care is taken, so that the temperature rise uniform is not more than 1-2⁰C per minute.

It is kept on heating until the substance in capillary tube begins to melt. Flame is removed at this stage and temperature is observed at which melting had begun and where it had melted.

METHOD II: Introduce small quantity of sample in sealed capillary tube and keep it in a melting point determining apparatus along with thermometer through hole of apparatus and note down the temperature where the sample starts melting and then finally where it completely melts, this gives melting point range.



REPORT: Melting point range of various compounds is

Naphthalene	:	80-82 °C
α- naphthol	:	94-96 °C
β- naphthol	:	120-123 °C
Benzoic acid	:	120-121°C
Cinnamic acid	:	133 °C
Anthracene	:	210 °C
Benzophenone	:	47-48 °C
Benzamide	:	117-119 °C

DETERMINATION OF BOILING POINT

AIM: To determine the boiling point of given liquids.

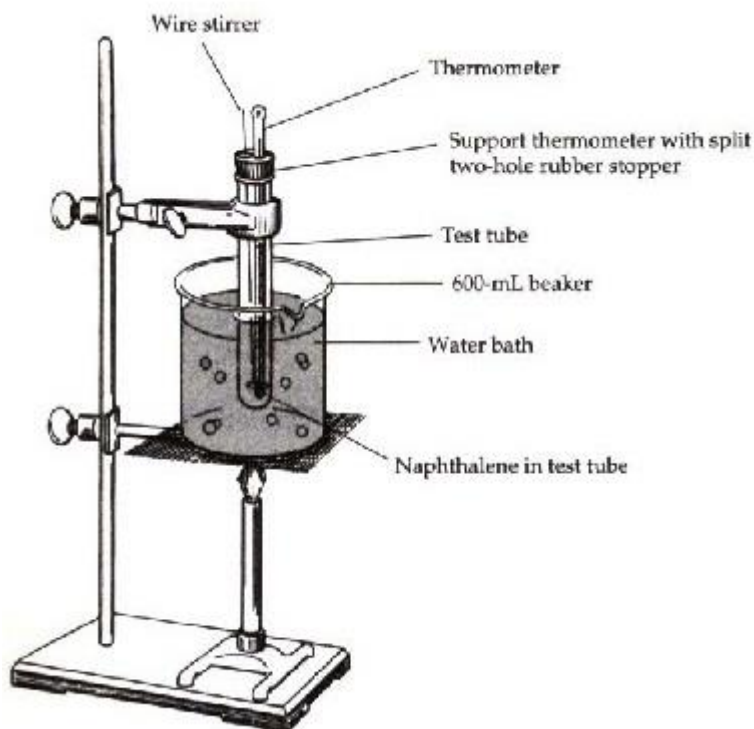
APPARATUS: Boiling tube, Thermometer, Capillary tube.

Principle: The boiling point of a substance is the temperature at which the vapor pressure of the liquid equals the pressure surrounding the liquid and the liquid changes into a vapor. The boiling point of a liquid varies depending upon the surrounding environmental pressure.

Procedure: This method is Siwoloboffs method. When reasonable amounts of liquid compound are available, the boiling point is readily determined by slowly distilling the material from a suitable flask and recoding the temperature at which the bulk of compound is distilled. For smaller quantities of the liquid, micro methods may be used.

Siwoloboffs Method:

In this procedure two tubes are used. One is the ordinary capillary tube i.e., 90 mm – 100 mm long and 1 mm in diameter and the other one is 100 mm long and 4-5 mm in diameter. Both the tubes are closed at one end. A small quantity of a liquid (0.5ml) is placed in a wide tube and the capillary tube with sealed end is introduced into the liquid. This tube is then attached to the thermometer with a thread and the thermometer is immersed in the bath of melting point apparatus. Heat the tube gradually. As the heating is continued, there will be a slow escape of bubbles from the end of capillary tube at a low speed in the beginning due to thermal expansion of the trapped air. Continue heating slowly. When the temperature of the liquid in the ignition tube equals to its boiling points, a rapid and continuous escape of the bubbles will occur. The reading of the thermometer, when rapid and continuous streams of bubbles first emerge from the capillary tube, is the boiling point of the liquid.



A more accurate result is obtained by removing source of heat, when the rapid stream of bubbles rises from the end of the capillary tube. Note the temperature at which the bubbles just fail to come out of the capillary and the liquid starts to enter the capillary. This temperature is taken as boiling point of the liquid.

Carbon tetrachloride	-	Literature Value - 77 °C
Chlorobenzene	-	Literature Value - 132 °C
Acetone	-	Literature Value - 56 °C

REPORT: Boiling point range of various compounds was found to be as

Acetone	:	56
Glycerine	:	290
Ethanol	:	78
Acetic anhydride	:	140
Benzene	:	80
Benzyl alcohol	:	206

PREPARATION OF *p*-BROMOACETANILIDE FROM ACETANILIDE

Aim: To synthesize *p*-Bromoacetanilide from Acetanilide

CHEMICALS	APPARATUS
Acetanilide 4 gm	Conical Flask,
Bromine – 1.5 ml,	Glass rod
Glacial acetic acid – 15 ml	Beaker,
Sodium bisulphate	Buckner funnel
Sodium metabisulphite	Burette.

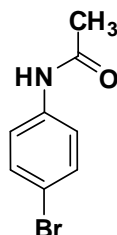
Principle:

IUPAC Name: N-(4-bromophenyl) acetamide

Synonyms: 4-bromoacetanilide, mono bromoacetanilide, bromoanilide

Chemical Formula: C₈H₈BrNO

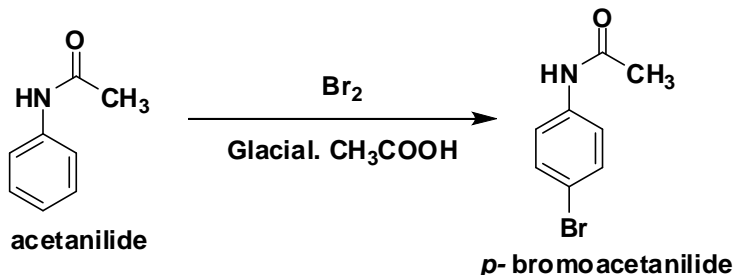
Molecular weight: 214.06 g/mol

Structure

p-bromo acetanilide

Explanation:

Acetanilide on reaction with bromine in presence of Glacial acetic acid (Nuclear bromination) to give *p*-bromoacetanilide. This mechanism is a classic example of **Electrophilic aromatic substitution**. An amine may lead to di- and tri- substituted products. If an amide is used in place of the amine, mono substitution usually predominates (the electron-withdrawing carbonyl group makes the benzene ring less nucleophilic). This *Ortho*-, *Para*- directing group will tend to only add groups *para*- to itself because of the steric bulk of the amide group.

Reaction

Procedure:

Dissolve 4 gm of acetanilide in glacial acetic acid (15ml) in a clean conical flask and add slowly drop wise a solution of bromine (1.5ml) in acetic acid (5ml). Shake the flask vigorously and cool during the addition. The addition of bromine solution allows the reaction mixture to stand for 30 minutes with frequent shaking. Pour the contents of the conical flask in to beaker containing cold water (200ml) with constant stirring and rinse the conical flask and transfer the contents into same beaker. If the product separates, it is colored, and then add sodium sulphite or sodium metabisulphite until it becomes colorless. Then filter through the Buchner funnel. Wash with cold water and dry.

Recrystallization:

A pure sample of *p*-bromo-acetanilide can be obtained as colorless crystalline solid on recrystallization from hot alcohol.

Percentage yield:

S. No	Chemical name	Quantity required	Quantity taken

$$\text{Percentage yield} = \frac{\text{Practical Yield}}{\text{Theoretical Yield}}$$

$$\text{Theoretical yield} = \frac{\text{Molecular Weight Of the Product}}{\text{Molecular Weight Of the Reactant}} \times \text{Gram Weight Of the R}$$

Description:

Melting point : 168 °C,
 Solubility : insoluble in cold water,
 : Sparingly soluble in hot water,
 : Soluble in benzene, chloroform, ethyl acetate,
 : Moderately soluble in alcohol.

Uses: 1. Used as an Analgesic agent
 2. Used as Anti-pyretic agent.

Precaution: Handle bromine carefully as it digests the bones and produces irritation to the eyes

Result: *P*–Bromoacetanilide was synthesized from Acetanilide, the % yield was found to be _____ % w/v and the melting point was found to be _____.

Viva questions:

- 1) What is the mechanism involved in the preparation of parabromo acetanilide?
- 2) What is the use of sodium sulphite or sodium metabisulphite?
- 3) Which of the reagents should be taken excess and why?
- 4) What are the uses of *P*-bromoacetanilide?
- 5) What is the principle involved in it?

PREPARATION OF PARA-BROMOANILINE FROM PARA- BROMOACETANILIDE

AIM: To prepare *p*-Bromoaniline from *p*-Bromoacetanilide by Hydrolysis process.

CHEMICALS	APPARATUS
<i>p</i> -Bromoacetanilide- 18 gm	Round bottom flask
Alcohol – 35 ml	Condenser
5% NaOH	Adaptor
Con. H ₂ SO ₄ – 1 to 2 drops	Conical Flask, Funnel

Principle:

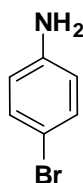
IUPAC name: 4-bromoaniline

Synonyms: *p*-bromoaniline, 4-bromobenzenamine, *p*-bromophenylamine

Chemical Formula: C₆H₆BrN

Molecular weight: 172.02 g/mol

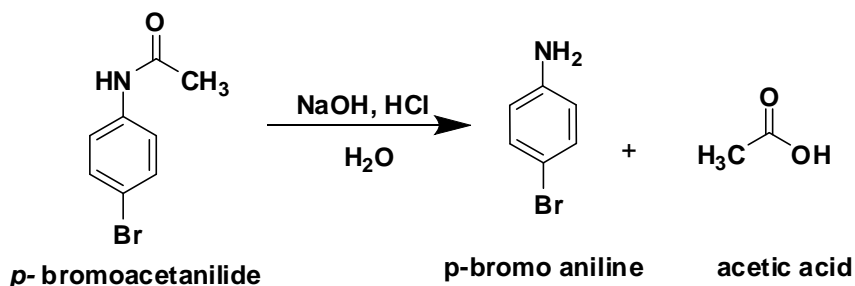
Structure:



p-bromo aniline

Explanation:

p-Bromoaniline cannot be prepared by bromination of aniline, since the major product of bromination is 2,4,6-tribromoaniline. Therefore, the amino group is protected by mineral acids and then bromination gives *p*-Bromoacetanilide as the major product which on **hydrolysis** gives *p*-Bromoaniline in the presence of mineral acids.

Reaction:**Procedure:**

Dissolve 18 g (0.084 mol) of *p*-Bromoacetanilide in 35 ml of boiling ethanol contained in a 250 ml round bottomed flask equipped with a reflux condenser. With the aid of a pressure equalizing dropping funnel add 22 ml of concentrated hydrochloric acid down the condenser in small portions to the boiling solution, Reflux for 30-40 minutes or until a test portion remains clear when diluted with 150 ml of water, and fit the flask with a condenser set for downward

distillation. Distil the mixture from an air bath and collect about 100 ml of distillate the latter consists of ethyl acetate, ethanol and water. Pour the residual solution of *p*-bromoaniline hydrochloride in to 100 ml of ice-water, and add with vigorous stirring, 5% sodium hydroxide solution until just alkaline. The *p*-bromoaniline separates as an oil, which soon crystallizes. Filter the crystals and wash with cold water and dry in the air upon pads of filter paper.

Recrystallisation: A pure sample of *p*-bromo-aniline can be obtained as colourless crystalline solid on Recrystallisation from hot alcohol.

Percentage yield:

S. No	Chemical name	Quantity required	Quantity taken

$$\text{Percentage yield} = \frac{\text{Practical Yield}}{\text{Theoretical Yield}}$$

$$\text{Theoretical yield} = \frac{\text{Molecular Weight Of the Product}}{\text{Molecular Weight Of the Reactant}} \times \text{Gram Weight Of the R}$$

Description:

Melting point : 60 to 64 °C
Solubility : soluble in water

Uses:

1. Used as an Analgesic agent
2. Used as Anti-pyretic agent.

Result: *P* -Bromoaniline was synthesized from hydrolysis of *p*-Bromoacetanilide, the percentage yield was found to be _____ % w/v and the melting point was found to be _____.

Viva Questions:

1. Which compound is produced on hydrolysis of *p*-bromo acetanilide?
2. Define bromination.

3. Which compound is produced on bromination of aniline?
4. Give the IUPAC name of aniline.
5. What is the pharmaceutical significance of *p*-Bromoaniline?

PREPARATION OF *m*- DINITROBENZENE

AIM: To synthesize *m*- Dinitrobenzene from Nitrobenzene.

CHEMICALS	APPARATUS
Conc. Nitric acid – 7 ml,	Measuring jar,
Conc. H ₂ SO ₄ – 8 ml	Round bottom flask
Nitro Benzene – 6 ml	Condenser
Distilled Water – 100 ml	Beaker, Funnel etc.

Principle:

IUPAC Name: 1, 3- dinitrobenzene

Synonyms: Dwunitrobenzene; Binitrobenzene; o-Dinitrobenzene; m-dinitro-benzene

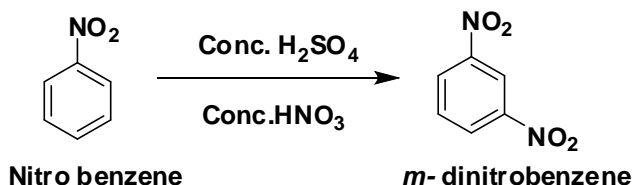
Chemical Formula: C₆H₄N₂O₄

Molecular weight: 168.02 g/mol

Structure:

**Explanation:**

Nitrobenzene on nitration with Conc. Nitric acid in the presence of sulphuric acid 60 °C gives *m*-dinitrobenzene. The nitro group present on the benzene ring is electron deficient or electron withdrawing or ring deactivating group, due to the presence of this group the electron density on *Ortho* and *Para* positions becomes very less when compare to that of the *meta* position. So, its attacking position may be *Meta*. Hence, Nitrobenzene on nitration will result in the formation of *m*-dinitrobenzene.

Reaction:**Procedure:**

Place 7 ml of Nitric acid in 100 ml of round bottom flask and add 8 ml of Conc. H_2SO_4 in small portion with shaking, Keep the reaction mixture cool while adding by immersing the flask in crushed ice or ice water. Introduce 6 ml of nitrobenzene in small portions, cooling the flask in ice or ice water, shake the flask to ensure thorough mixing after each addition of nitrobenzene, do not allow the temperature of the mixture to rise above 55°C .

When all the nitrobenzene has been added, fit a reflux condenser to the flask. Heat on water-bath and maintain at 60°C for 30-40 minutes. Remove the flask from water bath for time to time and shake it vigorously to ensure good mixing to the immiscible liquids layers. Later remove the flask from water bath and pour the contents of the flask into 100 ml of cold water taken in a beaker. Stir the mixture well to remove acids as much as possible from the *m*-dinitrobenzene and allow standing. When the *m*-dinitrobenzene settles to the bottom, pour off the acid layer as completely as possible and transfer the remaining liquid to separating funnel. Again, wash with 50 ml of water and allow it to settle. Collect the lower layer of *m*-dinitrobenzene into a dry test tube or boiling tube adds anhydrous calcium chloride which acts as dehydrating agent. After 24 hours a clear liquid with pale yellow color crystals will be obtained. Collect the crystals by filtration.

Recrystallization:

A pure sample of *m*-dinitrobenzene can be obtained as pale-yellow color crystalline solid on Recrystallization from alcohol.

Precaution: *m*-dinitrobenzene is toxic and do not inhale its vapor. Let it not fall on your skin.

Percentage yield:

S.no	Chemical name	Quantity required	Quantity taken

$$\text{Percentage yield} = \frac{\text{Practical Yield}}{\text{Theoretical Yield}}$$

$$\text{Theoretical yield} = \frac{\text{Molecular Weight Of the Product}}{\text{Molecular Weight Of the Reactant}} \times \text{Gram Weight Of the R}$$

Description:

Melting Point : 89.6 °C

Solubility : freely soluble in chloroform, ethyl acetate

Uses: 1. Used in pesticides.
2. Used in dyes.

Result: *m*-dinitrobenzene was synthesized from nitrobenzene, the percentage yield was found to be _____ % w/v and the melting point was found to be _____.

Viva questions:

- 1) What is nitration mixture?
- 2) What is the name of the reaction?
- 3) What is the function of sulphuric acid?
- 4) What is the mechanism of reaction?
- 5) What is the function of calcium chloride?
- 6) What is electrophile in this experiment?
- 7) What precautions are taken in handling nitrobenzene?

PREPARATION OF N-BUTYL ACETATE FROM N-BUTYL ALCOHOL

AIM: To prepare and submit the pure sample of n-butyl acetate.

CHEMICALS	APPARATUS
Glacial acetic acid – 15ml	Round bottom flask
N-butyl alcohol – 12ml	Reflux condenser
H ₂ SO ₄ – 1 ml	Beaker
5% Na ₂ CO ₃ solution,	Water bath, Glass rod,
Anhydrous sodium sulphate	Boiling tube, Measuring jar, Separating funnel.

Principle:

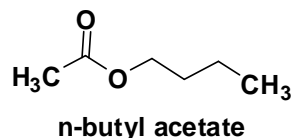
IUPAC Name: *n* - Butyl acetate

Synonym: *n*-butyl ethanoate, Acetic acid-butyl ester,

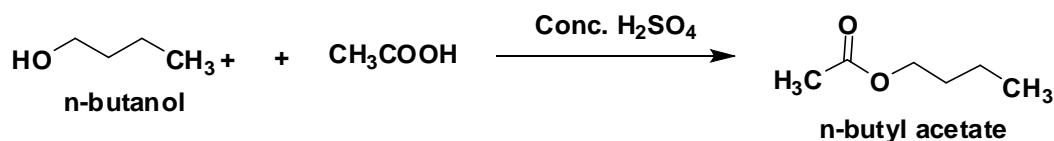
Chemical Formula: C₆H₁₂O₂

Molecular Weight: 116.16 g/mol

Structure

**Explanation:**

n-butyl alcohol undergoes esterification with Glacial acetic acid in the presence of sulphuric acid to give *n*-butyl acetate.

Reaction**Procedure:**

Take 12 ml of *n*-butyl alcohol into a clean, dry round bottom flask and 15 ml of glacial acetic acid and followed by 1 ml of concentrated sulphuric acid. Fit the round bottom flask with reflux condenser and heat it on non luminous flame for about 1 hour. Cool the reaction mixture and pour it into cold water with stirring. A pleasant smelling, light colorless oil separates on the surface of water as upper layer. Transfer into separating funnel and add dilute sodium carbonate

solution. Finally wash it with cold water and draw into a dry test tube. Dry over anhydrous sodium sulphate. Further purification can be done by distillation method.

Percentage yield:

S.no	Chemical name	Quantity required	Quantity taken

$$\text{Percentage yield} = \frac{\text{Practical Yield}}{\text{Theoretical Yield}}$$

$$\text{Theoretical yield} = \frac{\text{Molecular Weight Of the Product}}{\text{Molecular Weight Of the Reactant}} \times \text{Gram Weight Of the R}$$

Description:

1. Boiling Point: 126 °C
2. Solubility : miscible in ethanol, soluble in [acetone](#), [CHCl₃](#)

Uses: 1. Used in foods as anti flavouring agent.
2. used in the manufacture of lacquers, artificial leather, photographic films, plastics, and safety glass.

Result: *n*- butyl acetate was synthesized from *n*- butanol, the percentage yield was found to be _____ % w/v and the boiling point was found to be _____.

Viva questions:

- 1) How is the concentrated n-butyl alcohol separated?
- 2) Why should we use sodium carbonate?

- 3) Why do we use anhydrous sodium sulphate?
- 4) Write the principle involved in preparation of N-butyl acetate.
- 5) Write the structure of N-butyl acetate.

PREPARATION OF β - NAPHTHYL METHYL ETHER (NEROLIN) FROM β - NAPHTHOL

AIM: To synthesize and submit the Nerolin from β - naphthol

CHEMICALS	APPARATUS
β -Naphthol – 3.6 gm	Conical flask
Dimethyl sulphate – 2.5 ml	Glass rod
10% Sodium hydroxide – 20 to 30 ml	Water bath,
Con. H_2SO_4 – 1 to 2 drops	Measuring cylinder, Beaker.

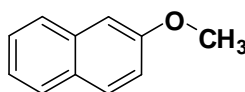
Principle:

IUPAC name : 2-Methoxynaphthalene

Synonyms: 2-Naphthol methyl ether, 2-Naphthyl methyl ether, β -Methoxynaphthalene, β -Methyl naphthyl ether, β -Naphthol methyl ether;

Molecular weight: 158.09 g/mol

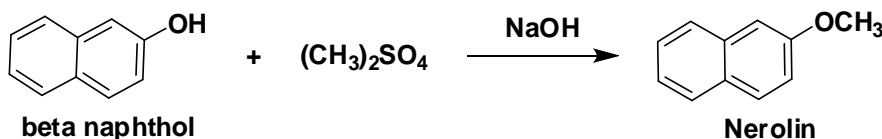
Structure:



2-methoxynaphthalene

Explanation:

β - Naphthol undergoes methylation in the presence of dimethyl sulphate and NaOH to give β - naphthyl methyl ether which is also called as nerolin. The mechanism involved in this reaction is nucleophilic substitution reaction.

Reaction:**Procedure:**

Take 3.6 gm of β -Naphthol in a conical flask and dissolve it in 20-30ml of 10% Sodium hydroxide solution. Add 2.5 ml dimethyl sulphate while the mixture is cooled on ice. Warm the mixture for 10-15 minutes at 60-70 $^{\circ}\text{C}$ and allow it for cooling. Filter off the separated nerolin at the vacuum pump, wash it with 10% sodium hydroxide solution and thoroughly with cold water. Dry the product and recrystallize it from either rectified spirit or methylated spirit.

Note:

Dimethyl sulphate decomposes to give sulphuric acid. Therefore, the reagent must be free from sulphuric acid. Thus, purification of dimethyl sulphate is required, which is done by shaking with 10% sodium carbonate or sodium bicarbonate.

(These are preferred to sodium hydroxide because on neutralization of acid, they release CO₂, which is visible to our eyes).

Caution: Dimethyl sulphate is highly corrosive and irritating substance. Therefore, contact to skin is to be avoided.

Percentage yield:

S.no	Chemical name	Quantity required	Quantity taken

$$\text{Percentage yield} = \frac{\text{Practical Yield}}{\text{Theoretical Yield}}$$

$$\text{Theoretical yield} = \frac{\text{Molecular Weight Of the Product}}{\text{Molecular Weight Of the Reactant}} \times \text{Gram Weight Of the R}$$

Description:

1. Melting Point : 70- 72 °C
2. Solubility : 1. it is soluble in [alcohol](#)
2. Insoluble in [water](#) and [dipropylene glycol](#).

Uses: 1. Used in paints and varnishes.
2. used as a [stabilizer](#) found in [gunpowder](#).

Result: β-Naphthol methyl ether was synthesized from β-Naphthol, the percentage yield was found to be _____ % w/v and the boiling point was found to be _____.

Viva questions:

- 1) What is the role of sodium hydroxide in this reaction?
- 2) Why should we purify dimethyl sulphate before use and how it is done?

- 3) What is the advantage of washing dimethyl sulphate with sodium carbonate or sodium bicarbonate rather than with NaOH?
- 4) The precipitate of nerolin is washed with NaOH solution, why?
- 5) Why should we wash the precipitate of nerolin thoroughly with water?
- 6) What is the methylated spirit?
- 7) What is the test for purity of β -naphthol methyl ether or nerolin?
- 8) What is the function of dimethyl sulphate?

PREPARATION OF OF IODOFORM FROM ETHYL ALCOHOL

AIM: To synthesize and submit iodoform from Ethyl alcohol.

CHEMICALS	APPARATUS
Alcohol – 30 ml	Conical flask,
10% NaOH solution – 30 ml	Measuring cylinder
Iodine solution – q.s	Glass rod, Thermometer, Funnel

Principle:

IUPAC Name: Triiodomethane

Synonyms: Iodoform

Chemical Formula: CHI_3

Molecular Weight: 393.73 g/mol

Structure:



ODOFORM

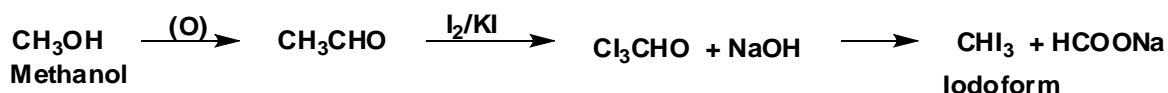
Explanation:

Many substances containing either acetyl or ketone group on treating with KI and NaOH yield Iodoform and its formation is used as test for identifying these groups. Examples for acetyl groups are ethanol, isopropyl alcohol etc. The compounds which contain ketone group are acetone, Pyruvic acid etc. It is synthesized by Haloform Reaction, where a Haloform (CHX_3 , where X is a halogen) is produced by the exhaustive Halogenation of any of the following 4 groups.

- Methyl ketone: CH_3COR ,
- Acetaldehyde (CH_3CHO),
- Ethanol ($\text{CH}_3\text{CH}_2\text{OH}$)
- secondary alcohols (CH_3CHROH , where R is an alkyl or aryl group).

Any one of these four kinds of organic compounds gives Haloform reaction with iodine and sodium hydroxide.

Reaction:



Procedure:

Place 30 ml of alcohol in a conical flask and add 30 ml of 10% NaOH solution to it. Add iodine solution in small quantities while shaking until a yellow colour persists, which is due to an excess of iodine. Warm the contents of the flask on water bath and maintain the temperature between 50 -60 °C. After 10-15 minutes of heating, remove the conical flask from the water bath, cool and collect iodoform by filtration.

Recrystallization:

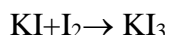
Recrystallize it either from ethyl alcohol or methyl alcohol. Take prepared iodoform into a boiling tube and add sufficient amount of methanol. Allow to cool to room temperature for 15 to 20 minutes until crystals of Iodoform settle at the bottom. The product should not be dissolved in methanol. Filter and collect crystals of iodoform.

Note:

While heating the iodoform and methanol in a water-bath, small porcelain pieces are added to the boiling tube, so as to prevent the bumping of the liquid.

Iodine Solution: Dissolve 2 gm of Iodine and 3 gm of KI in water to produce 100ml.

NOTE: Iodine solution is a solution of Iodine in potassium Iodide solution (KI). Iodine is insoluble in water. It dissolves in a solution of potassium Iodide (KI) due to formation of KI_3 .

**Percentage yield:**

S.no	Chemical name	Quantity required	Quantity taken

$$\text{Percentage yield} = \frac{\text{Practical Yield}}{\text{Theoretical Yield}}$$

$$\text{Theoretical yield} = \frac{\text{Molecular Weight Of the Product}}{\text{Molecular Weight Of the Reactant}} \times \text{Gram Weight Of the R}$$

Description:

Melting Point : 119-121 °C

Solubility : soluble in ether, [CHCl₃](#), [alcohol](#), slightly soluble in H₂O

Uses: 1. Used as an antiseptic.
2. Used as a disinfectant.

Result: Iodoform was synthesized from Iodine and ethyl alcohol, the percentage yield was found to be _____ % w/v and the melting point was found to be _____.

VIVA QUESTIONS:

1. Why should you use an excess of iodine in this experiment?
2. How do you know that you have used an excess of iodine?
3. Give examples for compounds that show positive result for iodoform test.
4. Give examples for compounds that do not show positive result for iodoform test.
5. What is Haloform reaction? Give one example.

PREPARATION OF 2, 4, 6- TRIBROMOANILINE FROM ANILINE

AIM: To prepare 2,4,6- tribromoaniline from aniline by Bromination process.

CHEMICALS	APPARATUS
Aniline	Round bottom flask
Glacial Acetic acid – 35 ml	Reflux Condenser
Bromine in Glacial Acetic acid	Adaptor, Measuring Cylinder
Ethanol,	Conical Flask, Funnel

Principle:

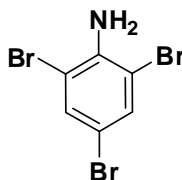
IUPAC name: 2, 4, 6- tribromoaniline

Synonyms: 2, 4, 6-Tribromobenzenamine, Tribromoaniline

Chemical Formula: $C_6H_4Br_3N$

Molecular weight: 329.81 g/mol

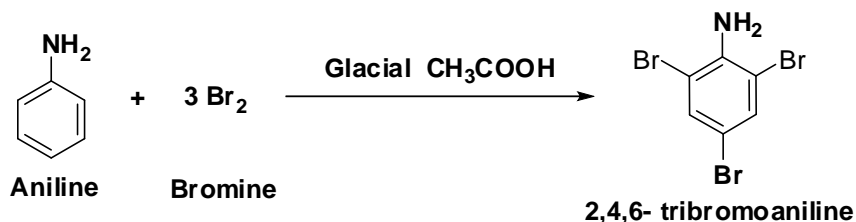
Structure:



2,4,6- tribromoaniline

Explanation:

Aniline undergoes Bromination with bromine in the presence of glacial acetic acid and results in the formation of 2, 4, 6- tribromoaniline, at first Bromine attack the ring between the ortho carbon and the carbon bearing the amine group. The electron pair of the amine moves to the C-N bond (making a double bond and a quadravalent ammonium group), the electrons of the ring move to one of the bromine atoms of Br_2 , causing Br^- to form. The ortho carbon now bears a hydrogen atom and a bromine atom; and the positive charge is delocalized via resonance around the ring and the ammonium to make a stable cation. Once the Br^- plucks the hydrogen from the ring, bromoanilide and HBr are formed. Since the amino group is an activating o-p director, this will happen twice more to give 2, 4, 6-tribromoaniline as final product provided sufficient Br_2 is present.

Reaction:**Procedure:**

Take 2.5 ml of aniline, 10 ml of glacial acetic acid in a 250 ml round bottomed flask. 4.2 ml of bromine is added drop wise (which is previously dissolved in 10 ml of glacial acetic acid) with constant stirring while keeping the round bottomed flask in ice bath. A solid mass is obtained which is filtered and washed with cold water. Recrystallize the product from dilute alcohol to obtain colorless crystals of 2, 4, 6-tribromoaniline.

Recrystallization: A pure sample of 2, 4, 6-tribromoaniline can be obtained as colorless crystalline solid on Recrystallization with small amount of activated charcoal from ethanol.

Percentage yield:

S. No	Chemical name	Quantity required	Quantity taken

$$\text{Percentage yield} = \frac{\text{Practical Yield}}{\text{Theoretical Yield}}$$

$$\text{Theoretical yield} = \frac{\text{Molecular Weight Of the Product}}{\text{Molecular Weight Of the Reactant}} \times \text{Gram Weight Of the R}$$

Description:

Melting point: 120-122 °C

Solubility : slightly soluble in water, freely soluble in ether and ethyl acetate

Uses:

1. Used in the preparation of dyes, polyamides,
2. Used in the preparation of antioxidants.

Result: 2, 4, 6-tribromoaniline was synthesized from aniline, the percentage yield was found to be _____ % w/v and the melting point was found to be _____

ANALYSIS OF ORGANIC COMPOUNDS**IDENTIFICATION OF ORGANIC COMPOUNDS**

EXPERIMENT	OBSERVATION	INFERENCE
<u>PRELIMINARY TEST</u>		
1. Physical state	Solid	The given compound may be carboxylic acid
	Liquid	The given compound may be alcohol, ether, ester, aliphatic amine aldehydes, ketone, hydrocarbon
2. Colour	Colorless	The compound may be carbohydrates, aldehydes, ketones, carboxylic acids, esters, alcohols or hydrocarbons
	Pale yellow	The compound may be nitro compounds, quinines(or) iodoform
	Pink	Alpha-Naphthol Beta- Naphthol (or) Resorcinol
	Dark brown(or) Reddish brown	Phenols or amines
	Yellowish orange	Nitro aniline
3. Odour	Pleasant odour	It may be alcohol, aromatic hydro carbons or alkyl halides
	Fruity odour	Esters
	Phenolic odour	Phenols
	Deep sweet smell	Chloroform
	Odour of bitter almond	Nitrobenzene Benzaldehyde or nitrotoulene
	Irritating odour	Aliphatic acids, acid chlorides or formaldehyde
	Fishy odour	Amines
4. Litmus test: A small quantity of the substance is shaken well with 5ml of water and solution and tested with litmus paper	Colour of the blue litmus is changed to red	Acidic compound
	Colour of the red litmus changes to blue	Basic compound
	No change in the colour of litmus paper	Neutral compound

Ignition test Heating on wire or spatula	Smoky flame	Aromatic compounds or compounds containing more than six carbon atoms
	No smoky flame	Aliphatic compounds
	Greenish flame	Compounds containing more number of halogen atoms
	Charring and smell of burnt sugar	Carbohydrates or citric acid.
Test for unsaturation 1. A little substance is dissolved in 1ml of water & treated with 3 to 4 drops of bromine water 2. A little substance is dissolved in 1ml of water & treated with 3-4 drops of 1% KMnO_4	The yellow color of the solution is decolorized	Unsaturated compound
	Yellow color of the solution remains	Saturated compound
	Yellow color of the sol is decolorized and precipitate formation is seen	Unsaturated compound like amine or phenol.
	Pink color of the sol is decolorized	Unsaturated compound
	Pink color of the solution remains	Saturated compound.
Test for aliphatic or aromatic compound 1. Flame test 2. Nitration test: A small amount of the substance is heated with a mixture of 1ml of conc. H_2SO_4 & 1ml of con HNO_3	Substance burns with smoky flame	Presence of aromatic compound
	Substance burns with non luminous flame	Presence of aliphatic compounds
	Substance burns with charring	Carbohydrates
	Yellow ppt or solution	Presence of aromatic compound
	No yellow colour is seen	Presence aliphatic compound.

SOLUBILITY CHART

REAGENT AND TEST	CLASS	GROUP OF COMPOUNDS
Soluble in cold or hot water. (If the unknown is soluble do NOT perform solubility tests below)	Neutral, acidic or basic. (Test with litmus or universal indicator paper)	Lower members of series. Neutral, e.g., alcohols; Acidic, e.g., acids, phenols; Basic, e.g., amines
Soluble in dil. HCl	Basic	Most amines (except III amines with only aromatic groups)
Soluble in dil. NaOH	Acidic	Most acids, most phenols.
Soluble in NaHCO ₃	Strongly acidic	Most carboxylic acids.
Insoluble in water, acid and alkali	Neutral	Hydrocarbons, nitrohydrocarbons, alkyl or aryl halides, esters and ethers. Higher molecular weight alcohols, aldehydes and ketones

DETECTION OF EXTRA ELEMENTS

Lassaignes Test:**Procedure for preparation of Sodium Fusion extract:**

Take in a sodium fusion tube, small pieces of shining freshly cut metallic sodium in excess which is previously dried between the folds of filter paper. Heat the tube on the flame so that sodium metal melts. Remove it from the flame and allow it to stand, so that sodium occupies the bottom portion of the tube. Add a pinch of the substance to be tested and heat the tube once again, gently with removing the tube from the flame time to time. Then heat the tube strongly until it becomes red hot and drops it immediately in about 10-15 ml of distilled water, taken in a clean mortar. Close the mortar with wire gauge. After the tube has broken and the reaction stopped. Remove the wire gauge and triturate with a pestle. Filter the contents and the filtrate is called sodium fusion extract.

TEST	OBSERVATION	INFERENCE
Test for Nitrogen: Take 2-3 ml of sodium fusion extract in a test tube. Add 0.1-0.2 gm of ferrous sulphate crystals. Heat the mixture gently with shaking until it boils without cooling, add sufficient amount of dilute sulphuric acid to dissolve the Iron hydroxides and give the solution of acidic medium.	If Prussian blue color appears	Indicates presence of nitrogen.
Test for sulphur: Acidify 2ml of sodium fusion extract with acetic acid and add a few drops of lead acetate solution.	If black precipitate of lead sulphide is formed	Indicate the presence of sulphur.
Test for halides: Acidify a portion (1 mL) of the 'fusion' filtrate with 2N nitric acid, and if nitrogen and/or sulphur are present, boil for 1 - 2 minutes. * Cool and add aqueous silver nitrate (1 mL), compare with a blank. Formation of a heavy, white or yellow precipitate of silver halide indicates halogen. If a positive result is obtained: acidify the remaining portion of the 'fusion' filtrate with dilute sulphuric	(a) If the carbon tetrachloride layer remains colourless. (b) If the carbon tetrachloride layer is brown.	Indicates presence of chlorine. Indicates presence of bromine

acid, boil and cool. Add carbon tetrachloride (1 mL) and a few drops of freshly prepared chlorine water. Shake the mixture.	(c) If the carbon tetrachloride layer is violet.	Indicates presence of iodine.
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NOTE: *If nitrogen and/or sulphur are also present, the addition of silver nitrate to the acidified 'fusion' solution will precipitate silver cyanide and/or silver sulphide in addition to the silver halides. The removal of hydrogen cyanide and/or hydrogen sulphide is affected by boiling the 'fusion' solution.

PRECAUTIONS TO BE TAKEN DURING PREPARATION OF SODIUM FUSION EXTRACT
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- 1) Sodium is always stored under kerosene.
- 2) Sodium should not be exposed to skin because it reacts with water and produces a burning sensation.
- 3) The mortar should be covered with a wire gauge after plunging the red hot tube into it; to avoid fire caused by excess of unreacted sodium on reaction with water.
- 4) The metal absorbs small quantities of moisture, the outer layer is encrusted with sodium hydroxide therefore while taking the metal for this test, one must scrap off the outer surface with a blade or knife, until sodium hydroxide is removed and a bright luster of sodium metal persists.
- 5) Always handle the metal with a forceps.
- 6) While heating one must be careful the metal does not come out of the tube and fall on the skin or eyes.

FUNCTIONAL GROUP TEST

ANALYSIS OF PHENOLS

Experiments	Observation	Inference
Sodium carbonate solution Test: Add 0.1 gms of substance to about 5ml of Na_2CO_3 solution taken in a test tube. A drop of test sample is placed on blue litmus paper.	No effervescence and no gas evolved and it changes the blue litmus to red.	May be phenol
Ferric Chloride Test: Take a very dilute solution of phenol; add a drop of ferric chloride solution.	Blue or violet, green-violet color	May be phenol, resorcinol, catechol, p-bromophenol
Phthalein Test (or) reaction with phthalic Anhydride: Place in a dry test tube about 0.2gms of phenol, add equal quantity phthalic anhydride, add 3 drops of sulphuric acid and heat for about 10 minutes until red homogenous mass is formed. pour the mixture into water taken in a beaker and add NaOH solution in excess.	Colourless solution turns blue or green in NaOH medium.	Indicates presence of phenolic compound. (Except p-cresol)
Action of Bromine water / Bromine water test: In a concentrated substance of phenol add Bromine water gradually.	Reddish brown colour is discharged and a white or pale yellow ppt is formed	Presence of phenolic compound.
Liebermann Reaction: Minute crystals of sodium nitrite are taken in a clean test tube, add 0.1 gms of phenol and heat gently for 20 seconds Allow it to cool and add 1ml of Conc. H_2SO_4 . The test tube is rotated to mix the contents	Blue-green color is developed on dilute the contents with H_2O the color changes to red which turns to blue with NaOH.	Indicates presence of phenol with free para position
To a small amount of substance add 2ml of NaOH and add 2 drops of in a test tube and add 2ml of aqueous NaOH and 1 drop of chloroform and warm it.	Orange dark red color observed.	Presence of phenol

Azo-Dye formation: Dissolve 2-3 drops of aniline in 1ml of conc. HCl and 3 ml of water. Shake it to dissolve any HCl cool in ice, add a few drops of sodium nitrite solution. Add a thick cool diazonium solution to phenol in excess of NaOH solution.	Brown red colour is observed.	Presence of 1-naphthol
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ANALYSIS OF AMIDES

1) Action of NaOH solution: To a little substance, add a little quantity of sodium hydroxide solution, boil in water bath until the smell of ammonia is obtained.	Smell of NH ₃ observed up on heating.	May be amide
2. Test for amides: To the compound add excess of 2N NaOH solution. Boil well and expose a wet red litmus paper to the vapours; also expose the vapours to glass rod dipped in HCl. Cool the solution and add excess of HCl	a) Red Litmus paper turns blue b) Dense white fumes.	May be amide
3) To a little substance, add a little quantity of 20% sodium hydroxide solution and boiled strongly. Then cool the solution and acidified with dil HCl.	If white crystalline ppt is observed	It indicates presence of aromatic amides.
4) Hydroxamic acid test: Boil approximately 0.1 gm of substance with 5 ml of water. Add few drops 3% hydrogen peroxide and 2 drops of 5%FeCl ₃ .Heat the solution.	Magenta colour is observed.	Indicates presence of aromatic primary amide.

ANALYSIS OF NITRO COMPOUNDS

ANALYSIS OF ALDEHYDES AND KETONES

Identification of Aldehydes and Ketones:

Test with 2,4-Dinitro Phenyl Hydrazine: Take 2-3ml of 2, 4-dinitro phenyl hydrazine in a test tube. Add few drops or milligrams of the substance in the reagent. If any yellow oil colour or yellow precipitate appears. The inference is a carbonyl compound.

Note: There are two kinds of 2, 4 DNP reagents.

- a) One for water – soluble compound.
- b) One for water insoluble compound.

The yellow oil or organic yellow precipitate formed in the 2,4 DNP hydrazine corresponding aldehydes or ketones.

- c) Some hindered ketones will not give this reaction immediately. So, shake the solution and set aside for 5-10 minutes. If no precipitate is seen. Hereafter this period, conclusion is not a carbonyl compound. The hindered carbonyl compound may not give the positive reaction.

Distinguish test for aldehydes and ketones

Schiff's Test: Take 2-3 ml of Schiff's reagent in a test tube and add the substance under investigation to it. If the magenta color (Rose-Pink colour) appears then the substance is aldehydes. If does not appear, then the substance is ketones.

Note: Schiff's reagent is a solution of pararosaniline hydrochloride saturated with SO_2 . Pararosaniline hydrochloride is in solution form with roasting colour when SO_2 gas is passed into the solution. The dye pararosaniline hydrochloride is oxidized and the color disappears. When you add aldehyde to this solution. The aldehydes will oxidize the solution to carboxylic acid and thus the magenta (pink – rose colour) of dye is restored. Aldehydes are easily oxidizable substances, ketones are not.

Tollen's Test: Take 2ml of silver nitrate and 1ml of NaOH solution in a test tube. Add ammonia solution to this solution. Until, the precipitate formed is completely dissolved (this is a Tollen's reagent)

ANALYSIS OF ALDEHYDES

Tollen's Test: Take 2ml of silver nitrate and few drops of 5% NaOH solution in clean test tube. Add NH_3 solution to this until the precipitate formed is completely dissolved (this is Tollen's reagent). Add the substance under investigation (few drops or milligrams), heat on

water bath. If silver mirror is formed on the inner walls of test tube, the substance is aldehydes. If no silver mirror is formed, it is ketones.

Note: When silver nitrate is added to sodium hydroxide, a double decomposition reaction takes place to give AgOH or Ag₂O. This precipitate is dissolved by NH₃, which forms a complex with silver ions. The reagent produced is called Tollen's reagent. i.e, silver ammonium hydroxide or ammonical silver nitrate. This is reduced by metallic hydroxide, with goes on oxidation to form acids. The metallic silver gets deposited on the test tube giving the appearance of mirror. The test tube used for this test must be clean and must not be greasy (wet). If it is greasy, the metallic silver formed cannot stick to the inner wall of the test tube, but slides down and sit on the bottom of test tube. So, the test tube must be clean before this test done.

Fehling's Test: Take in a test tube 1ml of Fehling solution. A (Fehling's solution-1) and add 1ml of fehling's solution-B (fehling solution – 2) To this add few drops of substance, heat on the flame. The appearance of green, yellow, orange red colour or a brick red precipitate indicates the presence of aldehydes. If no reduction takes place the substance is ketones.

Note: 1) Fehling's solution – A is a solution of copper sulphate in water.

2) Fehling's solution – B is a solution of NaOH and sodium potassium tatarate (Rochelle's Salt). When fehling's solution – A is mixed with Fehlings solution-B, CuSO₄ and sodium hydroxide react to give Na₂SO₄ & Cu(OH)₂ (CuSO₄ and sodium hydroxide react to give sodium sulphate and copper hydroxide Cu(OH)₂, the Cu(OH)₂ is a red ppt, this is kept in a solution by sodium potassium tatarate, which act as complexing agent.

Iodoform Test: Take a small quantity of substance; add 1 of NaOH solution followed by Iodine solution. Heat gently on water bath and look for the colour of iodoform. If iodoform is formed, it is methyl ketone. It is soluble and gives an acetaldehyde.

Identification test for Aldehydes:

Experiment	Observation	Inference
Formation of Phenyl hydrazone: To 5ml of water, add about 5ml of glacial acetic acid and 0.5 ml of phenyl hydrazine. Then add 3 drops of given substance and shake the test tube for 1-2 minutes.	A derivative of aldehyde	May be an aldehyde
Formation of 2, 4 dinitro phenyl hydrazone (or) Reaction with 2, 4 DNP: Take 2-3 ml of 2, 4 – DNP reagent in a test tube and add few drops or few mg of substance to the reagent.	Bright orange solid separates out	Presence of carbonyl compound

<p>Formation of semicarbazone Test: Add 0.5 gm of semicarbazine hydrochloride to 5ml of water. Add 0.5 gm of anhydrous sodium acetate and warm gently until clear solution is obtained. Then add a solution of 0.5 ml of the substance in 2-3ml of alcohol and warm on water bath.</p>	Yellow orange color crystals are formed	Presence of carbonyl compound
<p>Reduction Ammonical silver nitrate solution or tollen's Reagent: Place 5ml of AgNO₃ solution in a thoroughly cleaned test tube, add 2-3 drops of dilute NaOH and dilute NH₃ solution, drop wise, until the precipitate of Ag₂O or AgOH is almost redissolved, then add 2-3 drops of substance and warm on waterbath.</p>	Black ppt	May be an aldehyde
<p>Fehling's Solution: 1ml of Fehling's solution A+1ml of Fehling's – B and add few ml or few gm of sample and heat on water bath.</p>	Yellow to red solid separate out	May be an aldehyde
<p>Sodium bisulphate addition compound: shake 1ml of compound with about 0.5ml of saturated sodium bisulphate solution. The mixture becomes warm and white addition product separates out on cooling, the substance is regenerated on addition of dil. H₂SO₄.</p>	White crystalline ppt	May be an aldehyde

KETONES

Characteristics reaction of Ketones:

Experiment	Observation	Inference
<p>Formation of Phenyl Hydrazone: To 5ml water, add about 0.5ml of glacial acetic acid and 0.5ml of phenyl hydrazine. Then add 3 drops of given substance and shake the mixture for 1 to 2 minutes.</p>	A florescent ppt of phenyl dihydrazone is produced	May be ketone
<p>Formation of oximes: Take 1 gm of hydroxyl amine hydrochloride, 2gms of sodium acetate and 0.5gm of sample and warm it on water</p>		

<p>insoluble (aldehydes) ketones, with pyridine catalyst, cool the contents and add 5ml of water to the cooled residue cool in ice bath and stir until crystals of oxime separate. Then filter off and recrystallize from alcohol and benzene.</p> <p>Formation of 2,4Dinitrophenyl hydrazone: Dissolve a few drops of substance in a few ml of water or methanol. Adds 2 drops of 2,4 dinitro phenyl hydrazine reagent.</p> <p>Formation of semi carbazone test: Add 0.5 gm of semi carbazine hydrochloride to 5ml of water. Add 0.5gm of anhydrous sodium acetate and warm gently until a clear solution is obtained. Then add 0.5 ml of substance and 2-3ml of alcohol and warm on water bath.</p> <p>Reduction of Ammonical AgNO₃ solution (or) Tollen's Reagent: Place about 5ml of AgNO₃ solution in a thoroughly cleaned test tube. Add 2-3 drops of dil. NaOH add dil. NH₃ solution drop wise until precipitate of silver oxide is almost redissolved, then add 2-3 drop of substance and warm on water bath.</p> <p>Fehling's Solution: 1ml of fehling's solution – A, 1ml of fehling's solution – B and few ml/gm of sample are taken in a test tube and to be heated on water bath.</p> <p>Sodium Bisulphite addition compound: Shake 1 ml of compound with about 0.5ml of saturated sodium bisulphate solution. The mixture become warm and a white addition product separation occurring on cooling. The substance is regenerated on addition of dil. H₂SO₄.</p>	<p>Bright orange solid separates out</p> <p>Yellow orange color crystals are formed</p>	<p>May be ketone</p>
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<p>Iodoform Test: To 0.5 ml methyl ketone, add 3ml of 10% KI solution and 10ml of freshly prepared. Sodium hypochlorite solution and mix well. (OR) To the sample add excess iodine solution and sodium hydroxide solution</p> <p>Nitro Prusside Test: Add 3ml of freshly prepared sodium nitro prusside to 0.5ml methyl ketone and add dil. NaOH solution in excess.</p> <p>Dinitro Benzene Test: To 1ml of methyl ketone, add about 0.1 gm of fine powder dinitro benzene and excess of NaOH solution and shake well.</p>		
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Result: The given compound is aliphatic compound and it is a

ANALYSIS OF AMINES

ANALYSIS OF PRIMARY AMINES

Experiment	Observation	Inference
1) Carbylamine test: Add few drops of CHCl_3 to about 0.2gm of the substance and the 2-3ml of alcoholic KOH. Mix well and warm gently, cool the tube and add carefully excess of Conc. HCl.	A foul or pungent odour of isonitrile is produced.	Maybe aliphatic/aromatic amine
2) Copper sulphate test: Add few drops of 10% Copper sulphate solution to 0.2gm of the substance.	blue or blue-green coloration or precipitate with the reagent	Indicates the presence of a 1^0 amine.
3) Diazo-Coupling: Dissolve 0.2 gm of substance in 1ml of Conc. HCl and dilute with about 2ml of cool in ice and add few drops of sodium nitrite solution. Now add this whole diazonium solution slowly to a cold solution of 2-Naphthol in an excess of 10% NaOH solution.	Formation of an orange to red dye is observed	Maybe an aromatic 1^0 amine
	No dye formation	Aliphatic 1^0 amine
4) Test with phenol: Add 5ml phenol solution which is dissolved in NaOH solution to the test sample.	Formation of red or brown or yellow ppt	Maybe an aromatic 1^0 amine
5) a. To 0.3 mL or 300 mg of unknown substance in a test tube add 5 mL of 10% NaOH solution and 0.4 mL of benzenesulfonyl chloride. Close the test tube with a cork and shake the mixture vigorously. Test the solution to make sure that it is still alkaline using litmus paper. b. Cool the solution & add 10% HCl solution dropwise.	soluble in base.	Presence of aliphatic primary amines.
	A precipitate is formed.	Presence of aliphatic primary amines.

ANALYSIS OF SECONDARY AMINES

General Reactions:

Experiment	Observation	Inference
1) A little amount of substance is heated in a test tube.	Brown ppt is formed	Indicates presence of 2° amine.
2) A little amount of substance is treated with nitrous acid.	formation of yellow oil or solid.	Indicates presence of 2° amine
3) Formation of Nitroso – amine: Dissolve 1ml of substance in about 3ml of dilute HCl and add NaNO ₂ solution gradually with shaking until the yellow layer separates out at the bottom of the solution. Transfer completely into a small separating funnel, add about 2ml of ether and shake it run off the lower layer and evaporate the ether to get the yellow oil of Nitroso derivative.	a green solution turns deep blue on making it alkaline	Presence of 2° amine

ANALYSIS OF TERTIARY AMINES

General Reactions:

Experiment	Observation	Inference
1. Dissolve the substance in dilute HCl and add potassium Ferro cyanide solution.	White crystalline ppt is observed	Presence of 3° amine
2. Formation of p-nitroso derivative: Dissolve 0.5 ml of substance in about 5ml of dilute HCl, cool in ice and then add carefully 2ml of 20% NaNO ₂ solution. Allow it to stand for 5 minutes and cooled, then add dilute NaOH solution.	a) a reddish solution is obtained b) A green ppt of p-nitro derivative is formed.	Presence of 3° amine

3. a. Test with nitrous acid: to the sample add 1 ml of nitrous acid and 1ml of HCl.	The orange-colored hydrochloride salt of the C-nitrosoamine.	Presence of 3° amine
b. Treating the C- nitosomine solution with NaOH base.	Liberates the blue or green C-nitrosoamine.	Indicates the presence of 3° aromatic amine.

Hinsberg's test (To different between 1°, 2° & 3° amines).			
A	Take about four drops (or 0.1 gm) of the unknown amine in a test tube; add about 0.2 gm of p-toluene sulphonyl chloride and 5ml of 10% NaOH. Shake the tube for 5 minutes	No reaction occurred	May be a tertiary or secondary amine
B	If no reaction appears to occur, heat the reaction mixture on a water bath for 1 minute and cool in ice.	i) No precipitation appeared	Indicates Tertiary amine
		ii) Precipitation produced	May be a secondary or primary amine
C	If precipitation appears in the alkaline medium and 5ml of water and shake	i) precipitation dissolved	Indicates the presence of primary amine
		ii) precipitation not dissolved	Indicates of presence of secondary amine
D	If the precipitation dissolves and the solution is clear, acidify with dil. HCl	Precipitation dissolves	Confirms the presence of primary amine

ANALYSIS OF ALCOHOLS

Experiment	Observation	Inference
Specific Test: To 1ml of Given Test solution add 1 pinch of Sodium Metal in dry test tube.	Evolution of bubbles/ gas is observed	May be an alcohol
Cerric ammonium nitrate reagent: to the 1ml of substance add 0.1 gm of ceric ammonium nitrate reagent.	Formation of red or yellow colour.	May be an alcohol
Ester Formation: Take 1 ml of substance in A dry Test Tube and 0.5 Ml of Acetic Anhydride or Glacial acetic acid are added. To It 2 Drops of conc. H_2SO_4 Is added Mix Thoroughly and Heat on water Bath and the Mixture Is poured into a Beaker containing sodium Bicarbonate Solution	Fruity odour is observed	May be an alcohol
Test with chromic acid: Add few drops of chromic acid reagent in 1ml of substance the solution will remain clear. Cool the solution then heats the solution.	Orange colour solution turn to bluish green due to formation of chromium ion	May be an alcohol
Iodoform test Take 1ml of the substance in the test tube and add 1ml of sodium hydroxide solution and few drops of iodine solution and few drops of iodine solution. Heat gently on water bath.	A characteristic color of iodoform is evolved (IF_3)	May be an alcohol

Distinguish between 1°, 2° & 3° alcohols: Lucas test: To 2ml of Lucas reagent (anhydrous ZnCl ₂ and HCl) in a dry test tube and about 0.5ml of alcohol shake vigorously and set aside.	No visible reaction at room temperature and cloudy only on heating. Solution turns cloudy in 3–5 minutes solution turns cloudy immediately, and/or phases separate	It may be 1° alcohol. It may be 2° alcohol It may be 3° alcohol
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ANALYSIS OF ESTERS

EXPERIMENT	OBSERVATION	INFERENCE
<p>HYDROXAMIC ACID FROMATION: To a few drops of ester add 0.2gms of Hydroxylamine hydrochloride and about 5ml of 10% NaOH and gently boil the mixture for 1-2 minutes. Cool acidifies with dilute HCl and them adds a few drops of FeCl₃ solution.</p> <p>ii) Compound + dil NaOH + Phenolphthalein and warm with shaking.</p> <p>iii) Compound + 20% sodium hydroxide solution. Boil for about 10 minutes. Cool and add excess of dil H₂SO₄</p> <p>HYDROLYSIS: Place 2ml of ester in a 250ml round bottomed flask fitted with reflex condenser. Add about 20ml of 10% aqueous NaOH solution, 2-3 porcelain – pieces and reflex gently for 20-30 minutes. At the end of this time all oil drops of unchanged esters should have disappears. Distillation method is useful in separating the alcohol.</p>	<p>Violet or deep reddish-brown color develops immediately</p> <p>Pink colour gradually disappears</p> <p>A white precipitate is formed</p> <p>White ppt is formed. (a salt of an acid derivative of ester).</p>	<p>It is an ester</p> <p>It is an ester</p> <p>It is an ester</p> <p>Sodium salt of carboxylic acid</p>

ANALYSIS OF CARBOHYDRATES

Molisch's test: 0.1 g of the substance is dissolved in 1ml of water and treated with α -naphthol in alcohol (Molisch's reagent). shake well & add conc. H_2SO_4 through the sides of the test tube.	A red-cum-violet ring appears at the junction of the two liquids.	Presence of carbohydrates.
Fehling's test: To little substance 2-3 drops of Fehling's solution A&B are added and heated.	Formation of yellow or brownish-red precipitate.	Presence of reducing sugars
Benedict's Test To 2 mL of Benedict's reagent add five drops of the test solution. Boil for five minutes in a water bath. Cool the solution.	Formation of red, yellow or green colour/precipitate	Presence of reducing sugars
Barfoed's Test To 1 mL of the test solution add about 2 mL of Barfoed's reagent. Boil it for one minute and allow to stand for a few minutes.	Formation of brick-red precipitate	Presence of monosaccharides.
Tollen's test: A little substance is heated with tollens reagent & heated	Silver mirror on the walls of test tube. Black ppt is formed	Indicates presence of aldehyde. Indicates presence of ketone.
Osazone Test To 0.5 g of phenylhydrazine hydrochloride add 0.1 g of sodium acetate and 10 drops of glacial acetic acid. To this	Needle-shaped yellow crystals mushroom shaped crystals flower-shaped crystals.	Presence of glucose, fructose and mannose. Presence of lactosazone. Presence of maltose.

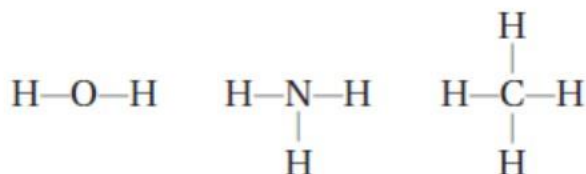
<p>mixture add 5 mL of test solution and heat on a boiling water bath for about half an hour. Allow the tube to cool slowly and examine the crystals under a microscope.</p>		
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Molecular Models – Full Lab

Purpose: To investigate the three-dimensional shapes of molecules by building molecular models.

Materials: Ball-and-stick model set.

Background: You can represent a molecule on paper with either formula or a structural formula. However, molecular formulas, such as NH_3 , provide no information concerning the actual arrangement of atoms in the molecule. Structural formulas, such as the following give some information about the arrangement of atoms in the molecule.



However, these structural formulas provide only limited information because they are two-dimensional. Actual molecular shapes are three-dimensional. A molecular model is far superior to a structural formula when it comes to visualizing atomic arrangement. Compared to molecular formulas and structural formulas, molecular models provide much more information about the true shapes of the molecules.

In this experiment, you will use ball-and-stick models to help you visualize the shapes of molecules. The balls are color-coded and sized to represent different atoms. The balls are also drilled with holes to accept stick and springs; the number of holes in a ball reflects the maximum number of bonds a given atom can form. Single bonds are represented by short wooded sticks; double and triple bonds are represented by springs.

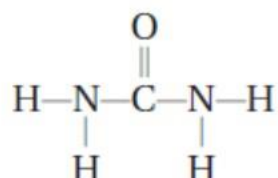
Procedure:

Data Table 1 shows color codes for balls representing different atoms. As you build the models, draw the structural formulas of the molecules in Data Table 2.

DATA TABLE 1				
Atom	Symbol	Color of Ball	Number of Holes	Maximum Number of Bonds
hydrogen	H	yellow	1	1
carbon	C	black	4	4
oxygen	O	red	2	2
nitrogen	N	blue	3 or 5*	3
chlorine	Cl	green	1	1
bromine	Br	orange	1	1
iodine	I	purple	1	1

* If the nitrogen has five holes, connect two adjacent holes with a spring bond.

1. Using the ball-and-stick model set, construct models of water, H_2O ; ammonia, NH_3 ; and methane, CH_4 . Draw a sketch of each molecule in Data Table 2. The shape shown by the water molecule is described as bent, the shape of the ammonia molecule is called trigonal pyramidal, and the shape of the methane molecule is called tetrahedral. Write these names below the matching structures you have drawn in the table.
2. Construct models of hydrogen sulfide, H_2S ; carbon tetrachloride, CCl_4 ; dichlorofluoromethane, CCl_2F_2 ; and ethane, C_2H_6 . Give the molecular formula for each of these compounds, and draw a sketch of each molecule in Data Table 2. Name the shape of each molecule.
3. The air above a burning candle contains nitrogen gas, carbon dioxide gas, and oxygen gas. Construct models of these substances. In Data Table 2, draw a sketch of each molecule.
4. The compound urea has the molecular formula $\text{CO}(\text{NH}_2)_2$. The structural formula of urea is:



Construct a model of urea and sketch its shape in Data Table 2.

5. Construct a model of butane, C_4H_{10} . (HINT: the carbons are bonded to one another in a continuous, unbranched chain.) Draw a sketch of this molecule in Data Table 2. Can you construct a model of a different molecule that has the same molecular formula as butane? Make a model of such a molecule and sketch its structure in the data table.

The two different compounds having the molecular formula, C_4H_{10} are called *structural isomers*. They have identical molecular formulas but different structural formulas. They also have different physical and chemical properties. Structural isomers play a very important role in organic chemistry.

6. Construct a model of bromochlorofluoromethane, CHBrClF . Sketch the compound in the table. Can you construct an isomer of this compound? (HINT: Is your left hand identical to your right?) Draw the new compound if you can.

The compound and the isomer have the same molecular formula, CHBrClF , but they are different from each other in the way that a left hand is different from a right hand. The compounds are mirror images of each other and are called *stereoisomers*. The phenomenon of "handedness" exhibited by pairs of stereoisomers is important in organic chemistry and biochemistry.

Conclusion:

1. Although the VSEPR model allows one to predict the geometry of a molecule, the prediction must still be verified through experiment. One experimental ?

DATA TABLE 2: SKETCHES OF MOLECULES

H_2O	NH_3	CH_4
bent triatomic	trigonal pyramidal	tetrahedral

H_2S	CCl_4	CCl_2F_2	C_2H_6
bent triatomic	tetrahedral	tetrahedral	

N_2	CO_2	O_2
linear triatomic		

$\text{CO}(\text{NH}_2)_2$	CHBrClF , stereoisomers
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C_4H_{10} , structural isomers
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