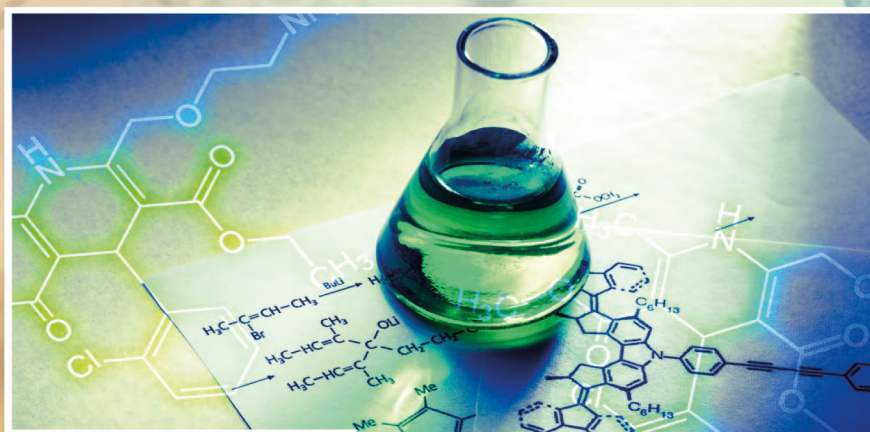


# Practical Lab Manual of Pharmaceutical Organic Chemistry - I

**As Per PCI Syllabus**  
**B. Pharm 2<sup>nd</sup> Semester**



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**B. Pharm 2<sup>nd</sup> Semester**

**Dr. Shivendra Kumar Dwivedi**  
**M. Pharm (Pharmaceutical Chemistry), Ph.D.**  
**Assoc. Professor**  
**University of Pharmacy,**  
**Oriental University, Indore (MP)**



**IP Innovative Publication Pvt. Ltd.**



**IP Innovative Publication Pvt. Ltd.**

A-2, Gulab Bagh, Nawada, Uttam Nagar, New Delhi - 110059, India.

Ph: +91-11-61364114, 61364115

E-mail: [info@ipinnovative.com](mailto:info@ipinnovative.com)

Web: [www.ipinnovative.com](http://www.ipinnovative.com)

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## **Dedicated**

Affectionately to my Father Mr. Ramkhelawan Dwivedi, he is also a great teacher in a sky and my Mother Mrs. Phool Kumari Dwivedi.

**Shivendra Kumar Dwivedi**

### About the Author



**Dr. Shivendra Kumar Dwivedi**, M. Pharm (Pharmaceutical Chemistry), Ph.D., presently working as an Assoc. Professor in University Institute of Pharmacy, Oriental University, Indore (M.P). He has 10 years of experience in academics and research. He is also the author of some of the other books for UG and PG in Pharmaceutical Chemistry and Practical manual. He has published more than 30 research papers in a different versatile International and National journals.

## **Acknowledgement**

I am indebted to all my family members particularly to my wife **Mrs. Namrata Dwivedi**, my daughter **Shravi Dwivedi** who have always remained my source of inspiration and encouragement.

I am grateful to **Dr. Neetesh Jain**, Principal, UIP, Oriental University, Indore (M.P.) for every step to encourage to publish the book.

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### **Note for the Students**

If you are a student, you will probably appreciate our effort to present you the book “Practical Lab manual of Pharmaceutical Organic Chemistry - I, which covers all practicals in the 2<sup>nd</sup> semester in organic chemistry - I.

The aim of this book is to give you an updated and comprehensive knowledge in a lucid manner. Considering your need, all the basic principles and concepts which underline the chemical reaction, have been explained.



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# Chapter 1

## Introduction of the Laboratory Safety

### Introduction

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 students 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's 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 dying 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 labor 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.

### **Condition and Care about Yourself to Chemicals and Hazard Substances**

Prime responsibility to the lab in charge or academician are required to see that students know and follow established safety rules and guidelines, have access to and know how to use appropriate emergency equipment and are aware of hazards of hazards associated with specific experiments. The lab instructor alone cannot prevent laboratory accidents, however. You also

have a responsibility to follow safe laboratory practices while performing experiments and to be ready to respond in case of accident.

People who work with chemicals should wear appropriate clothing and personal protective equipment (such as safety goggles) that reduce the likelihood of injury in case of an accident. Eye protection is always essential, and it should be the rule in every chemistry laboratory. Safety glasses provide only limited protection because they have no side shields, so it is best to wear safety goggles that protect your eyes from chemical splashes and flying particles from any direction.

If working a chemistry lab, you should properly wear clothing that is substantial enough and covers enough of your body to offer some protection against accidental chemical spills such as hazardous compounds and flying glass or other particles. Long-sleeved shirts or blouses and long pants or dresses are recommended, especially when they are made of denim or other heavy materials.

Some synthetic fabrics can be dissolved by chemicals such as acetone and could melt in contact with a flame or another heat source. Wear shoes that protect you from spilled chemicals and broken glass - not open sandals or cloth-topped athletic shoes. Always wear appropriate gloves when handling caustic chemicals, which can burn the skin, or toxic chemicals that can be absorbed through the skin. No single type of glove protects against all chemicals, but neoprene gloves offer good to excellent protection against many commonly used chemicals, and disposable nitrile gloves are adequate for use in most undergraduate labs. Latex gloves aren't recommended, because some people are allergic to latex because they are permeable to many hazardous chemicals.

### **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 vapors 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 materials can 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.

**Properly exercise great care when working with glass and when inserting or removing thermometers and glass tubing:** Most of the common injuries in a organic chemistry lab are cuts in hand from broken

glass and burns from touching hot glass. Protect your hands with gloves or a towel when inserting glass tubes or thermometers into stoppers or thermometer adapters, and when removing them. Grasp the glass close to the stopper or thermometer adapter and gently twist it in or out.

**Properly wear appropriate clothing in the laboratory:** Properly wear clothing that is substantial enough to offer some protection against accidental chemical spills, and shoes that can protect you from spilled chemicals and broken glass. Human hair is very flammable, to tie up your hair or wear a hair net while using a burner if you have long hair.

**Properly dispose of chemicals:** For reasons of safety and environmental protection, most organic chemicals shouldn't be washed down the drain. Except when your instructor or an experiment's directions indicate otherwise, place used organic chemicals and solutions in designated waste containers. Some aqueous solutions can be safely poured down the drain but consult your instructor if there is any question about the best method for disposing of a particular chemical or solution.

**No and never work alone in the laboratory or perform unauthorized experiments:** If you wish to work in the laboratory when no formal lab period is scheduled, you must obtain permission from the instructor and be certain that others will be present while you are working.

### **In a Laboratory Operations**

Students and laboratory instructor should read proper guidelines to the operation of practical in a chemistry lab, which types of care in carried out during laboratory operation. Should still read the descriptions carefully because an operation may require different equipment or be performed in a different way in the organic chemistry lab.

### **Cleaning Glassware**

Glassware must be clean in a water. Clean glassware is essential for good results in the organic chemistry laboratory. Even small amounts of impurities can sometimes inhibit chemical reactions, catalyze undesirable side reactions, or invalidate the results of chemical tests or rate studies. Always clean dirty glassware at the end of each laboratory period, or as soon as possible after the glassware is used.

If you wait too long to clean glassware, residues may harden and become more resistant to cleaning agents; they may also attack the glass itself, weakening it and making future cleaning more difficult. It is particularly



important to wash out strong bases such as sodium hydroxide promptly, because they can etch the glass permanently and cause glass joints to “freeze” tight. When glassware has been thoroughly cleaned, water applied to its inner surface should wet the whole surface and not form droplets or leave dry patches. However, used glassware that has been scratched or etched may not wet evenly.

You can clean most glassware adequately by vigorous scrubbing with water and a laboratory detergent, using a brush of appropriate size and shape to reach otherwise inaccessible spots. Organic residues that can't be removed by detergent and water will often dissolve in organic solvents such as technical-grade acetone (Never use reagent grade solvents for washing). For example, it is difficult if not impossible – to scrub the inside porcelain Büchner or Hirsch funnel, but squirting a little acetone around the inside of the funnel stem and letting it drain through the porous plate should remove chemical residues that may have lodged there. Use acetone sparingly and recycle it after use (don't pour it down the drain), as it is much more costly than water and may harm the environment. After washing, always rinse glassware thoroughly with water (a final distilled-water rinse is a good idea) and check it to see if the water wets its surface evenly rather than forming separate beads of water. If it doesn't pass this test scrub it some more or use a cleaning solution. Note that some well-used glassware may not pass the test because of surface damage, but it may still be clean enough to use after thorough scrubbing.

### **Lubricating Joints**

Most specialized glassware components used in organic chemistry have rigid ground-glass joints called standard-taper joints. The size of a tapered joint is designated by two numbers, such as 19/22, in which the first number is the diameter at the top of the joint and the second is the length of the taper, measured in millimeters. Glassware from a commercial organic lab kit, or its equivalent purchased as separate parts, can be used to construct apparatus for many different laboratory operations. For some operations, such as vacuum distillation, glass joints should be lubricated with a suitable joint grease. For most other operations, lubrication of glass joints is unnecessary and may be undesirable. Your instructor should inform you if lubrication will be necessary. To lubricate a ground-glass joint, apply a thin

layer of joint grease completely around the top half of the inner (male) joint.

Do not lubricate the outer (female) joint. Be careful to keep grease away from the open end of the joint, where it may meet and contaminate your reaction mixture or product. When you assemble the components, press the outer and inner joints together firmly, with a slight twist, to form a seal around the entire joint with no gaps. Grease should never extend beyond the joint inside the apparatus. After disassembling the apparatus, remove the grease completely by using a suitable organic solvent. You can remove petroleum-based greases with petroleum ether or hexanes, and silicone greases by thorough cleaning with dichloromethane.

An inner joint can be cleaned by wrapping a small amount of cotton loosely around the end of an applicator stick, dipping it in the solvent, and wiping the joint with the moist cotton. Assembling glassware Standard-taper joints are rigid, so a glassware apparatus must be assembled carefully to avoid strain that can result in breakage. First, place the necessary clamps and rings at appropriate locations on the ring stand (use two ring stands for distillations setups). Then, assemble the apparatus from the bottom up, starting at the heat source. Position the heat source on a ring or a Boy elevator so that it can be removed easily when the heating period is over; otherwise it may continue to heat a reaction mixture or an empty distilling flask even after it is switched off, causing a danger of breakage, tar formation, or even an explosion. Clamp the reaction flask or boiling flask securely at the proper distance from the heat source. As you add other components clamp them to the ring stand(s) but don't tighten the clamp jaws completely until all of the components are in place and aligned properly.

### **Basic Safety Rules**

These basic rules provide behaviour, hygiene, and safety information to avoid accidents in the laboratory. Laboratory specific safety rules may be required for specific processes, equipment, and materials, which should be addressed by laboratory specific SOPs.

Basic safety rules for laboratory conduct should be observed whenever working in a laboratory. Many of the most common safety rules are listed below:

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 fume hood 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. The PEL and the Threshold Limit Values (TLV) will be observed in all areas. If exposure above a PEL/TLV is suspected of an ongoing process, please contact EHS immediately.
26. Laboratory employees should have access to a chemical inventory list, applicable SDSs, department laboratory safety manual, and relevant SOPs.
27. Determine the potential hazards and appropriate safety precautions before beginning any work.
28. Procedures should be developed that minimize the formation and dispersion of aerosols.
29. If an unknown chemical is produced in the laboratory, the material should be considered hazardous.
30. Do not pour chemicals down drains. Do not utilize the sewer for chemical waste disposal.
31. Keep all sink traps (including cup sink traps and floor drains) filled with water by running water down the drain at least monthly.
32. Access to laboratories and support areas such as stockrooms, specialized laboratories, etc. should be limited to approved personnel only.
33. All equipment should be regularly inspected for wear or deterioration.
34. 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.
35. Designated and well-marked waste storage locations are necessary.



## Chapter 2

### To the Knowledge About the Basic Glassware are Used in the Laboratory

#### Background

Glassware are basically used as laboratory apparatus offers a wide range of containment and transport functions for solutions and other liquids used in laboratories. Most laboratory glassware is manufactured with borosilicate glass, a particularly durable glass that can safely be used to hold chemicals being heated over a flame and to contain acidic or corrosive chemicals. All laboratory glassware should be cleaned immediately following use to prevent chemical residue from congealing or hardening.

#### Beakers

Beakers are glass containers that come in a variety of sizes and can be used for mixing and transporting fluids, heating fluids over an open flame and containing chemicals during a reaction. While most beakers have graduated volume, measurements etched into their glass, the measurements are an approximation that may deviate from the actual volume by five percent, making them unsuitable for use as a precision measurement tool.

#### Test Tube

**Test tubes** are the archetypal image of chemistry, whilst beakers also make frequent appearances in school practical.

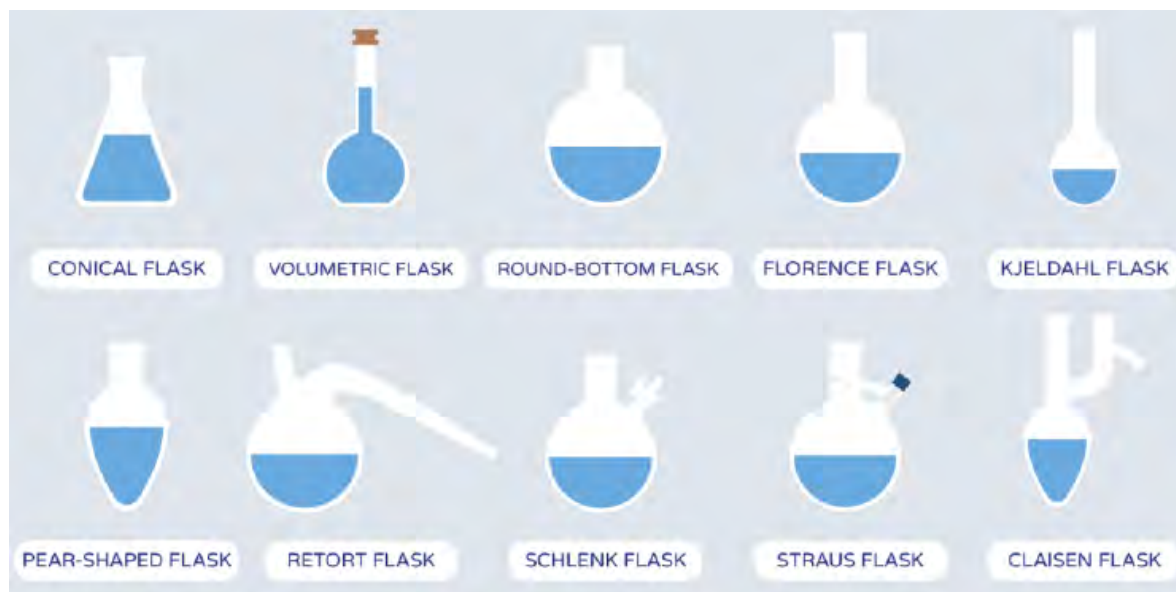
**Boiling tubes** are not that different from test tubes in appearance but are used when heating is required – they tend to be made of thicker glass and are slightly larger and wider.

#### Flask

**Flasks are used in the chemistry lab** is the most iconic is the conical (Erlenmeyer) flask. This also makes them useful for boiling liquids, and additionally their necks can support filter funnels.

**Volumetric flasks** are used to create precise quantities of solutions. A graduation line is etched into the volumetric flask's neck to indicate

volume, and the lab worker begins to fill the flask by first adding the solute. She continues by pouring in her solvent and then gradually adds drops of water as needed to bring the level of her solution up to the flask's graduated line. Which is also are used primarily in the preparation of standard solutions. To create a solution of a specific concentration, we need to know the volume of the solution; the narrow neck of the volumetric flask will have a thin graduation to show where a specific volume is reached.



**Round-bottomed flasks** and Florence flasks look very similar, but there is a slight difference between the two. Both have round bottoms, designed to spread out heat evenly when they are heated. They are frequently used by chemists for reactions and in rotary evaporators. Whereas round-bottomed flasks will usually have a ground glass joint on their neck, to allow connection to other apparatus, Florence flasks. They can also come with either a flat bottom, so they are free-standing, or a rounded bottom, and have longer necks.

**Kjeldahl flask** has an even longer neck, and was developed for use in the Kjeldahl method, which is used to determine the nitrogen content in a substance.

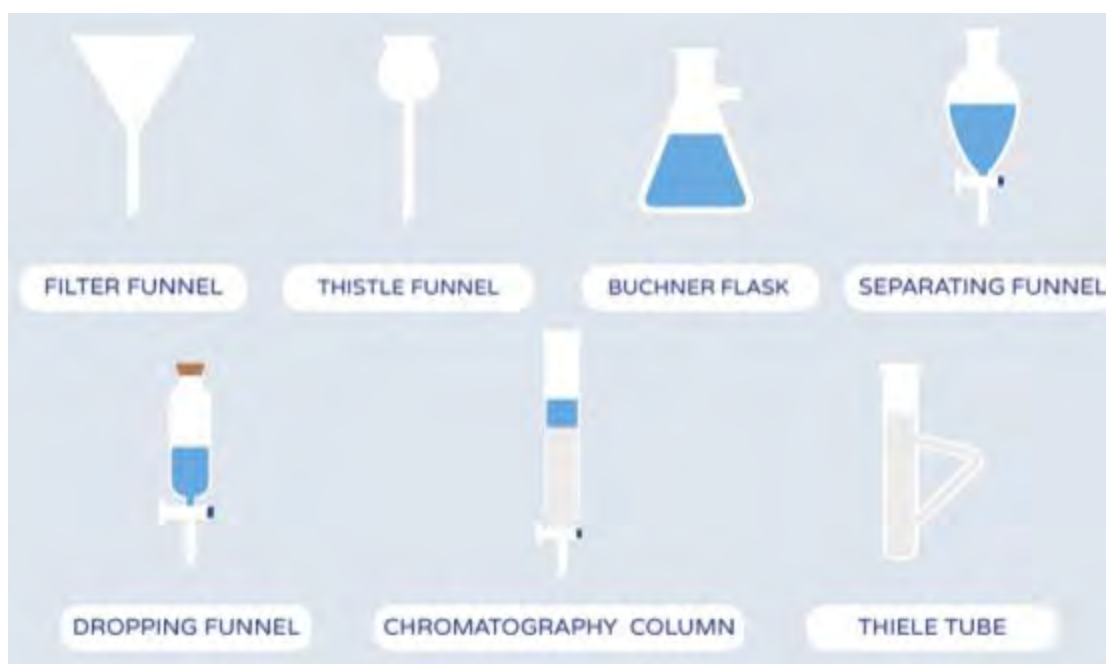
**Pear-shaped flasks** are usually rather small flasks, used for small-scale distillations. Their shape allows recovery of more material than the round-bottomed flasks.

**Schlenk flask and the Straus flask** are another two that look similar. Schlenk flasks are commonly used in air-sensitive chemistry, as the side arm allows an inert gas such as nitrogen to be pumped into the vessel. The Straus flask, on the other hand, is used to store dried solvents. The main neck is actually filled in halfway up and connected to a plugged smaller neck; this main neck can be connected to other apparatus and allows the solvent to be extracted when the plug is slightly withdrawn or removed entirely.

**Claisen flask** designed for vacuum distillation; distillation under vacuum produces problematic amounts of bubbles when solutions are boiled. Claisen's flask includes a capillary tube that inserts small bubbles into the liquid, easing the ferocity of boiling, whilst the branched portion of the flask hosts a thermometer.

### **Funnels, Analysis and Separation Funnels**

Glass funnels can be used to guard against spillage when pouring chemicals from one vessel to another, and they can also be fitted with a filter to separate solids from liquids. Separatory funnels are also used for filtration and extraction, having a bulb-shaped enclosed body fitted with a stopper on top to prevent spillage when the funnel is inverted, along with a stopcock at the spout's base, which can be used to gradually lower the bulb's internal pressure.



**Filter funnel**, into which filter paper can be placed, and a mixture can then be poured through. The smaller thistle funnel is not used for filtering at all, but to add liquids into apparatus.

**Buchner funnel** can be used in conjunction with a vacuum tube in vacuum filtration and is a much quicker process than the gravity filtration used with normal filter funnels. A vacuum tube can be affixed to the flask's sidearm, which rapidly sucks through the solvent, leaving any solid in the Buchner funnel.

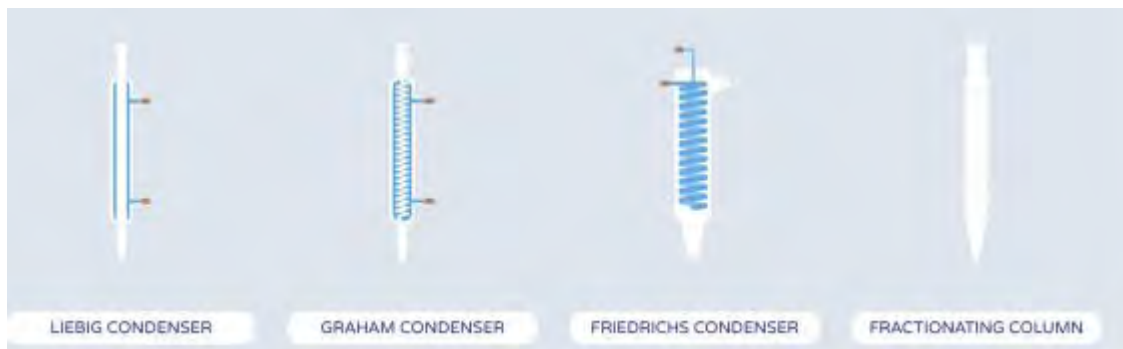
For separating solutions or liquids of different densities, separating funnels can be used. Liquids of a lower density will float to the top, then the mixtures can be tapped off separately. The similar-looking dropping funnel is used to add liquids or solutions to a reaction.

Another method of separating mixtures of compounds is column chromatography, which can be carried out using a chromatography column.

**Thiele tube** is a piece of apparatus used to determine the melting point of a solid compound. It contains and heats an oil bath, into which a sample can be placed along with a thermometer. Heating then allows melting point to be determined. The design of the flask allows the oil to circulate, ensuring even heating.

## Condensers

A range of different condensers can be utilized in laboratories as important components of distillation apparatus. The most commonly seen condenser in schools is the Liebig condenser, which has an inner tube through which vapour flows, surrounded by a 'jacket' through which cool water passes and condenses the vapour.



**Graham condenser** is like this but has a coiled path for the vapour to flow through and condense. Meanwhile, the Friedrichs condenser inverts

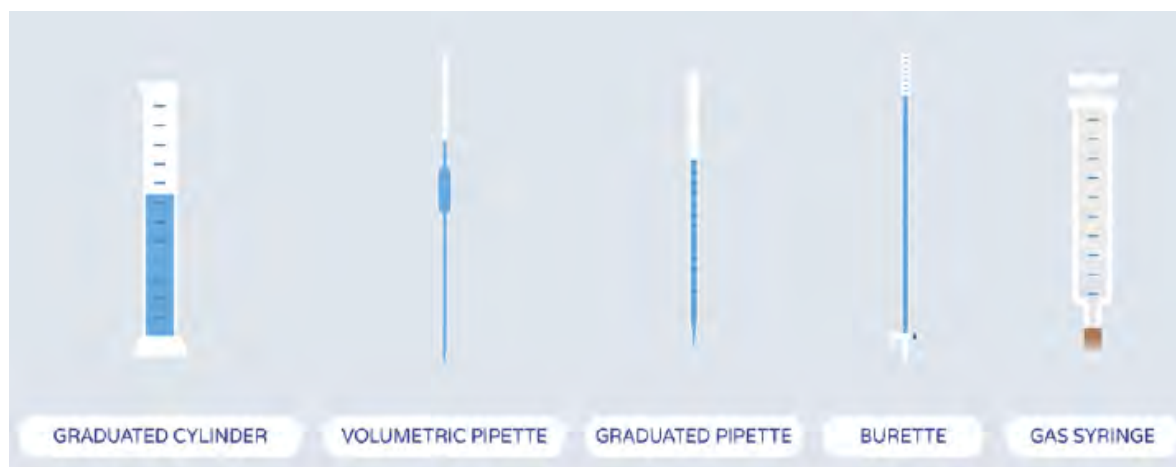


the arrangement, having a spiral coil through which the coolant flows, with the vapour surrounding it. Other varieties of condenser are also available, though are not shown here.

**Fractionating column** which helps separate a mixture during distillation, as vapours collect and distill on the small glass ‘trays’ ascending the column. Only the most volatile gases will ascend all the way to the top of the column to be distilled off.

### Measuring Apparatus

Graduated cylinders are tall, narrow containers used for measuring volume. While they are more accurate than beakers, measuring their contents to within one percent of actual volume, they’re not used for quantitative analysis of fluids that require a high degree of precision. Graduated cylinders are fitted with a “bumper ring,” a ring that shields the glass from impacting the work surface if the cylinder’s knocked over. Bumper rings should be placed near the graduated cylinder’s top for maximum protection.



**Volumetric pipette** which can be used to measuring volumes of solutions more precisely. These come in a variety of sizes, each measuring a fixed volume of solution.

**Graduated pipettes** can also be used, which allow various small volumes to be measured out. For measurement of volumes during titrations (the addition of one solution to another to determine an unknown concentration) burettes are used. These are long, narrow tubes, with incremental volume markings, which allow precise volumes of solutions to be dropped into another solution.

**Gas syringe** can be used to measuring the volume of gas produced in a reaction. This piece of apparatus can be attached to the top of a flask via a piece of tubing, and the gas produced pushes the plunger out of the syringe, allowing the gas volume to be measured.



# Chapter 3

## To Knowledge About the Laboratory Techniques of Recrystallization

Recrystallization process is, also known as fractional crystallization, is a procedure for purifying an impure compound in a given solvent. The method of purification is based on the principle that the solubility of most solids increases with increased temperature. This means that as temperature increases, the amount of solute that can be dissolved in a solvent increase.

### Principle

Recrystallization process is a laboratory technique for purifying different types of solids. The basic features of this technique are causing a solid to go into solution, and then gradually allowing the dissolved solid to crystallize. It is actually a very challenging process to get completely right. The goal is to obtain a compound in high purity as uniform crystals. Recrystallization is therefore a purification technique.

It is a crystallization technique used to purify a chemicals. By dissolving both impurities and a compound in an appropriate solvent, either the desired compound or impurities can be removed from the solution, leaving the other behind.

When an organic compound has been made it needs to be purified, particularly if it is a pharmaceutical chemical. This is because most organic reactions produce byproducts but, even if the reaction is a 'clean' one, the purity standards for many products are so stringent that small amounts of other compounds have to be removed. In the laboratory, this is often done by recrystallization. The general method is to find a solvent that dissolves the product more readily at high temperature than at low temperature, make a hot solution, and allow crystallizing on cooling. The crude product might contain:

1. Impurities which are insoluble in the solvent.
2. Impurities which are slightly soluble in the solvent and,
3. Impurities which dissolve readily in the solvent.

4. The solvent itself has also to be removed or it behaves as an impurity in its own right. It must not leave behind any residue.

## **Theory**

The basic concept of purification technique. It works because of:

1. Different substances have different solubilities in the same solvent, and
2. Only molecules of the same compound will fit easily into the crystal lattice of that compound. Impurities remain in solution or stick on the outside of the crystal lattice.
3. In practice you purify by slowly cooling a hot, saturated solution of your compound. While cooling, molecules of the same type align in a crystal lattice, forming crystals.
4. After cooling, crystals are collected by vacuum filtration and washed by rinsing with ice-cold solvent.
5. There are also different recrystallization techniques that can be used such as:

## **Single-Solvent Recrystallization**

1. Single solvent recrystallization is the most basic and commonly used recrystallization method.
2. An ideal solvent does not dissolve the solid at room temperature but dissolves the solid well in hot solvent.

## **Two Solvent Recrystallization**

1. Two solvent recrystallizations is an alternative and very useful recrystallization method to single solvent recrystallization.
2. The first solvent should dissolve your crude product very well at room temperature (or in hot solvent). The second solvent should not dissolve your crude product at room temperature or in hot solvent.
3. The two solvents should be completely miscible and preferably have similar boiling points.

## **Key Points of Recrystallization Process to Carried Out**

### **Choosing a Good Recrystallization Solvent**

1. A good solvent for recrystallization is one that your compound not very soluble in at low temperatures, but very soluble in at higher temperatures.
2. For example, if the solubility of your compound in ethanol is 1 g/100 mL at 0 °C and 2 g/ml at 50 °C, then you will not be able to purify much more than 1 gram.
3. Conversely, if the solubility of your compound in ethanol is 10,000 g/100 ml at 0 °C and 20,000 g/100 ml at 50 °C, this is also equally useless.
4. When are you going to be purifying that much of your compound like 10 kg; In this case, ethanol is just too soluble?
5. Ethanol is a good solvent to recrystallize acetanilide because of the wide solubility range 18 g/100 ml at 0 °C and 80 g/ml at 50 °C.

### **Like Dissolves Like**

1. Polar compounds are soluble in polar solvents such as water, methanol, and ethanol. If your compound contains a polar group (see image below), it is best to use these solvents.
2. Nonpolar compounds are soluble in non-polar solvents such as hexanes and diethyl ether.

### **Crystal Size and Cooling Speed**

1. The speed at which you allow a saturated solution to cool affects the size of the crystals that form.
2. If you take a hot solution and slam it into an ice bath, you will get smaller crystals.
3. If you allow a hot solution to cool without any added heat for 10–20 minutes, and then place it in an ice bath, you will get larger crystals.
4. This makes an important difference- if you have smaller crystals, they are more likely to clog a filter! Everyone likes to get out of organic lab as soon as possible, but a clogged filter could add a good 30–40 minutes to your procedure.
5. It's worth it not to place your solution in an ice bath right away; let it cool close to room temperature first.

### **Removing Insoluble Impurities**

1. Let us say you accidentally spilled group black pepper in a solution of acetanilide and ethanol. The black pepper will not dissolve.
2. So how do you remove it? By filtering the liquid– the black pepper will get stuck in the filter paper, and you can continue to recrystallize the liquid solution as normal.
3. The best way to get product to crash out is to add a seed crystal. This is just a small amount of pure material.
4. You can also try scratching the sides of the container with a stirring rod. This works because you pick up a small amount of solvent which evaporates and leaves behind a small amount of pure product.
5. So, if you wanted to, you can allow some of the mother liquor to evaporate and collect more products.

The key features necessary for a successful recrystallization process, are a very controlled temperature decrease and enough time. Because most solids have a better solubility at higher temperatures, we can saturate or almost saturate a solution at high temperature (usually the boiling temperature of the solvent), and then slowly allow the solution to reach room temperature.

### **Recrystallization Works Best When**

1. The quantity of impurities is small.
2. The solubility curve of the desired solute rises rapidly with temperature.

The slower the rate of cooling, the larger the crystals are that form. The disadvantage of recrystallization is that it takes a long time. Also, it is very important that the proper solvent is used. This can only be determined by trial and error, based on predictions and observations. The solution must be soluble at high temperatures and insoluble at low temperatures. The advantage of recrystallization is that, when carried out correctly, it is a very effective way of obtaining a pure sample of some product, or precipitate.

### **Requirements**

Chemicals: as per specific requirement

1. Acetanilide = 5 gm

2. Aspirin = 20 ml
3. Ethanol = 100 ml
4. Distilled water = 100 ml

### **Glassware/Apparatus**

1. Volumetric flask (200 mL)
2. Glass rod
3. Beaker
4. Measuring cylinder
5. Pipette
6. Burette
7. Tripod stand
8. Thermometer
9. Oil bath
10. Water bath

### **Standard Procedure**

These are the important steps to the recrystallization process

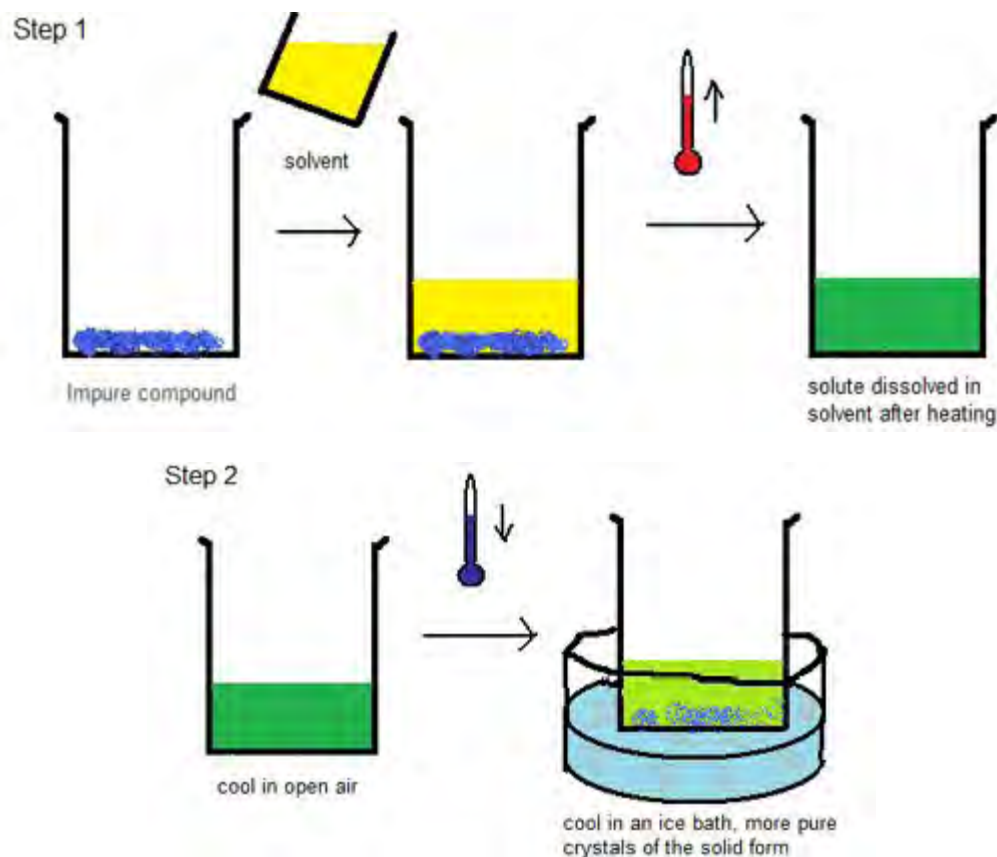
**Dissolve the solute in the solvent:** Add boiling solvent to a beaker containing the impure compound. Heat the beaker and keep adding solvent until the solute is completely dissolved.

**Cool the solution:** The solution is cooled in open air first, and then cooled in an ice bath. Slow cooling often leads to purer crystals. Crystals should form on the bottom of the beaker. The process of “seeding” can be used to aid the formation of crystals-this means adding a pure crystal of the compound. The pure crystal forms a surface for the solute to crystallize upon.

**Obtain the crystals of the solute:** The purer crystals of the solute are the desirable part of the mixture, and so they must be removed from the solvent. The process used for isolating the crystals that remain in the beaker still is called vacuum filtration. Suction is created using an aspirator, and whatever remains in the beaker is poured through a Buchner funnel. If for some reason there are no crystals visible, a gravity filtration can be performed. Activated carbon is added to the solution, the mixture is boiled, and a funnel system is used to transfer the new mixture to a new beaker of

boiling solvent. Filter paper is used in the funnel to remove excess carbon. After this mixture cools slowly, there should be large crystals present.

**Dry the resulting crystals:** The crystals are dried by leaving them in the aspirator and then by removing them to a glass dish to wait a while longer. The purity of the crystals can be tested by performing a “melting point determination”.



## Examples

### Procedure for Recrystallization of Acetanilide

1. The solubilities of most solids in solution increase as the temperature of the solution increases.
2. For example, the solubility of acetanilide in ethanol at 0 °C is about 18 g/100 ml.
3. This means that if you drop 50 grams of acetanilide in 100 ml of ethanol at 0 °C, about 18 grams will dissolve in the ethanol and the rest (~32 g) will remain suspended in the solution.



4. But the solubility of acetanilide in ethanol increases to about 80 g/100 ml at 500 °C.
5. This means that if we heated up the same acetanilide-ethanol suspension to 500 °C, all the acetanilide would dissolve.
6. In fact, we can add about another 30 grams of acetanilide to this solution and it would still dissolve, but once we added more than 80 g, additional solid acetanilide would no longer dissolve, and we would have a suspension once more.
7. At this point the solution is said to be saturated. So, let us say we had 50 g of acetanilide dissolved in 100 ml of ethanol at 500 °C.
8. What would happen if we allowed the temperature to cool back down again to 0 °C? At this point, we have more acetanilide dissolved in the ethanol than it should be able to hold, 50 g vs. 18 g, so the solution is said to be supersaturated.
9. That means that sooner or later, the extra ~32 grams of acetanilide should precipitate out of solution (also known as crashing out).
10. We then collect the acetanilide via vacuum (or gravity) filtration.
11. The liquid that remains after the filtration is known as the supernatant mother liquor (or just mother liquor for short).
12. It's usually a good idea to save it. Because there were relatively little impurities compared with the relatively large amounts of acetanilide and ethanol used, those impurities should remain dissolved in the solution; the ~32 grams of acetanilide we collect should be pure.

### **Recrystallization of Salicylic Acid Contaminated Aspirin [Acetylsalicylic Acid (ASA)]**

1. Aspirin does not like to dissolve in water. It sometimes dissolves slightly in hot alcohols.
2. To purify by recrystallization, place your crude aspirin (5 gm) in a 50 ml Erlenmeyer flask and dissolve it in a minimum amount of boiling propanol/Ethanol (Do this in a water bath).
3. Remove the flask and add warm water drop wise until the solution becomes slightly cloudy.
4. Allow the solution to cool until crystallization starts then finish crystallizing in the ice-bath.

5. Collect your product by filtration, wash with small quantities of cold water. Place in the oven and complete its drying overnight.
6. Then weigh and determine the melting point of the pure aspirin.

### **Result**

The recrystallization of aspirin and acetanilide was done in a solvent ethanol.

### **Uses**

Recrystallization is most important methods of purifying nonvolatile solids.



## Chapter 4

### To Obtain Pure Components from a Mixture of Organic Compounds Using Steam Distillation

#### Principle

Steam distillation technique is used for separating substances which are immiscible with water, volatile in steam and having high vapour pressure at the boiling temperature of water. Consider two immiscible liquids. In the mixture one liquid cannot mix the properties of the other. So, each liquid behaves as if the other is not present. Therefore, each liquid will show its own vapour pressure but the sum of the vapour pressures will be much higher than the vapour pressures of liquids. Hence the mixture of two immiscible liquid will boil at a lower temperature than the normal boiling point. So, this method can be used for purifying liquids with very high boiling points. e.g. Aniline.

If water is used as one of the immiscible liquids the method is called steam distillation. It is also used for purifying liquids which decompose at their normal boiling points. e.g. Glycerol.

This technique is used for separating organic compounds from plant parts. e.g. Lemon grass oil, Eucalyptus oil etc.

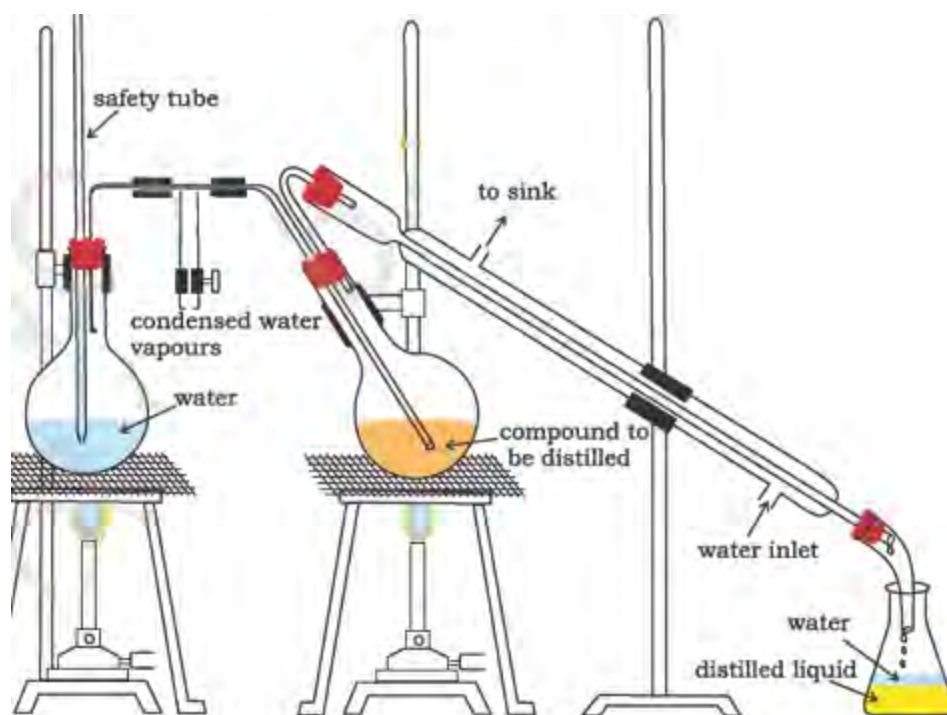
The steam distillation process works on the principle that when a mixture of two or more undissolved liquids are heated, while ensuring that the surfaces of both liquids are in contact with the atmosphere, the vapor pressure exerted by the system increases.

For example, the boiling point of **bromobenzene** is 156 °C and the boiling point of **water** is 100 °C, but a mixture of the two boils at 95 °C. Thus, **bromobenzene** can be easily distilled at a temperature 61 °C below its normal boiling point.

#### Basic Importance of Steam Distillation

1. Steam distillation is used to separate **heat-sensitive components**.

2. Steam is added to the mixture, causing some of it to vaporize. This vapor is cooled and condensed into two liquid fractions. Sometimes the fractions are collected separately, or they may have different density values, so they separate on their own.
3. An example is steam distillation of flowers to yield essential oil and a water-based distillate.
4. Steam distillation is employed in the isolation of essential oils, for use in perfumes.
5. Steam distillation also is an important means of separating fatty acids from mixtures and for treating crude products such as tall oils to extract and separate fatty acids, soaps and other commercially valuable organic compounds. Steam distillation was invented by the Persian chemist, **Ibn Sina** (known as **Avicenna** in the West), in the early 11th century. He invented it for the purpose of extracting essential oils, which are used in aromatherapy and the drinking and perfumery industries.



*Steam distillation. Steam volatile component volatilizes, the vapours condense in the condenser and the liquid collects in conical flask.*

## Requirements

**Chemicals:** as per specific requirement.

1. Bromine water, no more than 0.2% v/v.
2. Potassium manganate (VII), 0.00 M.
3. Cyclohexene (Highly flammable, Harmful if swallowed).
4. Cyclohexane (Highly flammable, skin and respiratory irritant).
5. Distilled water (100 ml).

### **Glassware/Apparatus**

1. Eye protection
2. Grater
3. Bunsen burner
4. Heat resistant mat
5. Tripod and gauze
6. Oranges (2)
7. 1100 °C thermometer
8. Measuring cylinder (100 ml)
9. Measuring cylinder (50 ml)
10. Distillation apparatus
11. 250 ml round bottomed flask
12. Still head
13. Thermometer pocket
14. Condenser
15. Receiver adapter
16. Test tubes and bungs
17. Dropping pipette
18. Anti-bumping granules
19. Oil bath

### **Importance of Extracting Limonene from Oranges by Steam**

#### **Distillation**

The peel of oranges is boiled in water and the oil produced (limonene) distilled in steam at a temperature just below 100 °C, well below its normal boiling point. The immiscible oil can then be separated. Direct extraction by heating would result in decomposition whereas steam distillation does not destroy the chemicals involved.

Limonene (1-methyl-4-prop-1-en-2-yl-cyclohexene) is an unsaturated hydrocarbon, classed as a terpene. At room temperature it is a colourless

oily liquid with the smell of oranges. Its molecular formula is  $C_{10}H_{16}$  and its boiling point is  $176\text{ }^{\circ}\text{C}$ .

Limonene is a chiral molecule with two optical isomers (enantiomers). The major biological form (d)limonene, the (R)-enantiomer, is used in food manufacture and medicines. It is also used as a fragrance in cleaning products, a botanical insecticide, and due to its flammability, a potential biofuel. The (S)-enantiomer, l-limonene, is also used as a fragrance but has a piney, turpentine odour.

## Procedure

### Step 1

1. Grate the outer orange coloured rind of two oranges and add to 100 ml of distilled water in the 250 ml round bottomed flask. Add anti-bumping granules to the round bottomed flask.
2. Set up the distillation apparatus as shown in the apparatus section.
3. Heat the flask so that distillation proceeds at a steady rate, approximately one drop per second of distillate. (Note: Take care not to let the liquid in the round bottomed flask boil too strongly).
4. Collect approximately  $50\text{ cm}^3$  of distillate in the measuring cylinder. The oil layer will be on the surface.
5. Using a dropping pipette carefully remove the oil layer into a test tube for the next stage.

### Step 2

**Odour:** Cautiously smell the extracted oil by wafting the fumes towards the nose. Do not breathe in directly from the test tube.

### Step 3

#### Action of Bromine Water

1. Measure out approximately 1 ml of bromine water into each of three test tubes.
2. Add a few drops of the limonene oil to one test tube, a few drops of cyclohexane to another, and a few drops of cyclohexene to the third.

Place in the bungs and agitate. If the bromine water is decolorized the molecule contains double bonds.

3. 0.001 M potassium manganate (VII) can be substituted for the bromine water for class use.

### **Result**

Extraction (steam distillation) of pure components from a mixture of organic compounds (limonene) was performed, so the bromine water is decolourized hence confirm that the molecule contains double bond (limonene) as compared to cyclohexane.



# Chapter 5

## To Determine and Report the Melting Point of the Given Sample Naphthalene and Benzoic Acid

### Principle

Melting point serves as a criterion of purity of an organic compound. Ions or molecules in a crystalline solid are arranged in a regular geometric fashion. Melting point occurs when a temperature is reached at which the thermal energy of the particles is great enough to overcome the inter crystalline forces that hold them together. A pure organic compound usually has a sharp and definite melting point. Chemical compounds which contain carbon is known as an organic compound. Study of manufacturing and synthesis of chemical compounds is known as organic chemistry. The temperature at which the chemical compound changes its state from solid to liquid is said to be the melting point of the compound. Let us learn to determine the melting point of organic compounds like naphthalene and benzoic acid.

### Requirements

**Chemicals:** as per specific requirement.

1. Naphthalene
2. Benzoic acid
3. Distilled water (100 ml)
4. Paraffin Liquid

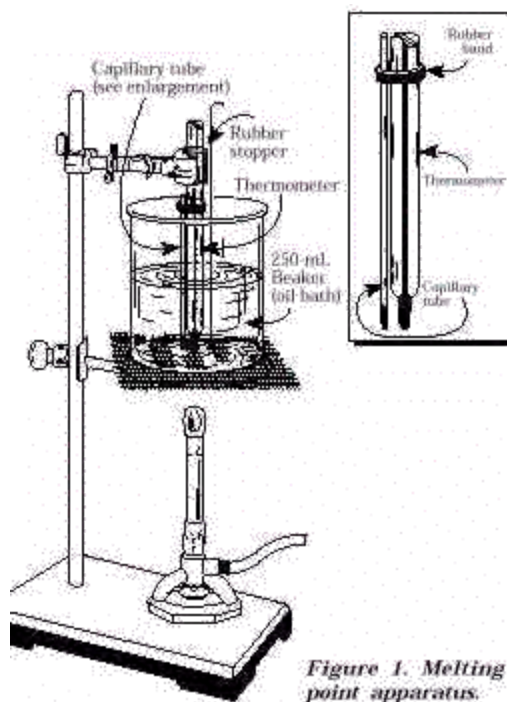
### Glassware/Apparatus

1. Eye protection
2. Aluminum block
3. Test tube
4. Bunsen burner
5. Tripod stands with clamp
6. Gauze



7. Capillary tube
8. 300 °C thermometer
9. Test tubes and bungs
10. Oil bath
11. Water bath
12. Watch

## Apparatus Setup



**Melting Point Apparatus**

## Procedure

### Determination of Melting Point of Naphthalene

1. Take a capillary tube and seal the capillary tube at one end by keeping 2–3 mm of capillary tube in Bunsen flame contact and continuously rotating it till the end is completely sealed by forming a bulb at the end.
2. Take naphthalene in a tile and crush it into a fine powder.
3. Now fill the capillary tube with compound the melting point of which is to be determined, about 0.05 g of compound (naphthalene) is placed on the plate and open end is then gently tapped.

4. Tie the capillary tube with thermometer and place the whole in Thiel's tube filled with liquid paraffin.
5. Now put Bunsen flame under the tube constantly and properly.
6. Keep continuous watch of the temperature and note the temperature as soon as the substance starts to melt.
7. Note the temperature ( $T-1$ ) as the substance begins to melt.
8. Make a note of the temperature ( $T-2$ ) when the substance has completely melted.
9. Take the average of the two temperatures.

### Determination of Melting Point of Benzoic Acid

1. Take Benzoic acid on a tile and crush it into a fine powder.
2. Take a capillary tube and by heating one end close the end of the tube.
3. hold the closed end of the capillary tube between your finger.
4. Dip the open end of the capillary tube in the finely powdered Benzoic acid.
5. Fill the compound in the capillary tube to about a length of 1–2 cm by gently tapping the capillary.
6. Attach the thermometer to a capillary tube using a thread.
7. Place the capillary tube in the groove of the aluminum block.
8. Check if the capillary tube holding the benzoic acid is exactly in the middle of the groove.
9. Place the aluminum block on a tripod stand above the kerosene burner and start heating the block with the burner.
10. Keep continuous watch of the temperature and note the temperature as soon as the substance starts to melt.
11. Note the temperature ( $T-1$ ) as the substance begins to melt.
12. Make a note of the temperature ( $T-2$ ) when the substance has completely melted.
13. The average temperature of the substance is determined.

### Observation

	Temperature ( $T - 1$ )	Temperature ( $T - 2$ )	Difference between = ( $T - 1$ ) + ( $T - 2$ )/2

M.P. of naphthalene			
M.P. of benzoic acid			

### Result

The melting point of given organic compound naphthalene 80.26 °C is about and benzoic acid to be 122.3 °C.

### Uses

It is useful in supporting the identification of a compound, as well as serving as a rough guide to the relative purity of the sample.

### Precautions to be Taken During the Experiment

1. Make sure the sample of naphthalene and benzoic acid are dry and powdered.
2. Keep the capillary tube and the thermometer at a similar level.
3. Tightly pack the powder into the capillary tube without any air gaps.

### Questions

1. What is the temperature at which a substance changes from solid state to liquid state called?
2. Why benzoic acid or Naphthalene needs to be crushed?
3. How is the melting point of the compound determined?
4. What is a sharp melting point?
5. Due to the high melting point which element is used as filaments in the light bulb?



## Chapter 6

### To Determine and Report the Boiling Point of the Given Sample (Benzaldehyde and Benzene)

#### Principle

In a liquid, the particles are not arranged in a regular manner and are thus free to move. A liquid boils when a temperature is reached at which the thermal energy of the particles is great enough to overcome the cohesive forces that hold them together. This is the point at which the vapour pressure of the liquid becomes equal to the atmospheric pressure. Boiling point of the liquid is significant for a compound. The boiling point of organic compounds can give important information about their physical properties and structural characteristics. Boiling point helps identify and characterize a compound. A liquid boils when its vapour pressure is equal to the atmospheric pressure. Vapour pressure is determined by the kinetic energy of a molecule.

Kinetic energy depends on the temperature, mass, and velocity of a molecule. When the temperature increases, the average kinetic energy of particles also increases. When the temperature reaches the boiling point, the average kinetic energy becomes enough to overcome the force of attraction between the liquid particles. As the force of attraction decreases, the molecules in the liquid state escape from the surface and turn into gas.

The boiling point of a liquid varies with the surrounding atmospheric pressure. A liquid at a higher pressure has a higher boiling point than when that liquid is at lower atmospheric pressure.

The normal boiling point of a compound is an indicator of the volatility of that compound. The higher the boiling point, the less volatile is the compound. Conversely, the lower the boiling point, the more highly volatile is the compound. At a given temperature, if a compound's normal boiling point is lower, then that compound will generally exist as a gas at atmospheric pressure. If the boiling point of the compound is higher, it then exists as a liquid or a solid.

## **Requirements**

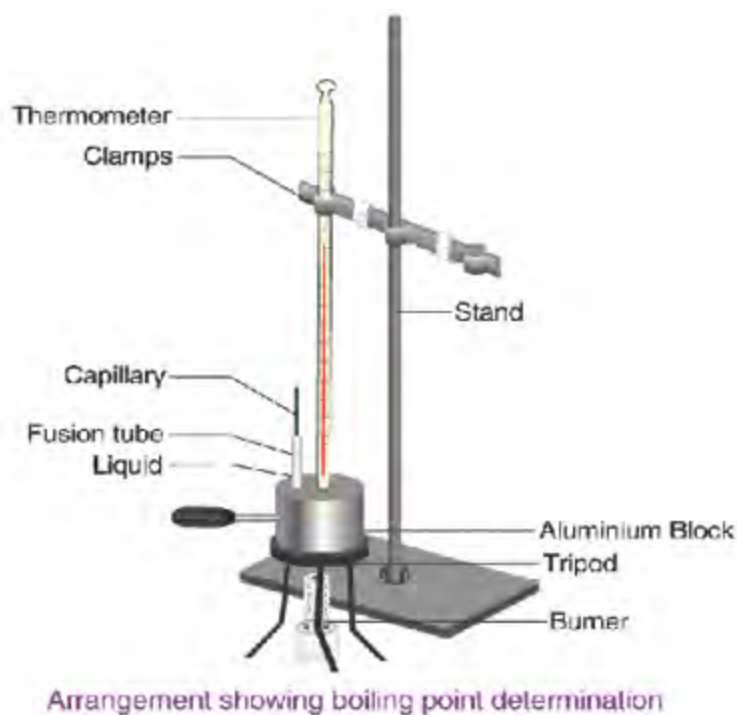
**Chemicals:** as per specific requirement.

1. Benzaldehyde
2. Benzene
3. Distilled water (100 ml)
4. Paraffin Liquid

## **Glassware/ Apparatus**

1. Theils tube
2. Test tube
3. Bunsen burner
4. Tripod stands with clamp
5. Gauze
6. Capillary tube
7. 300 °C thermometer
8. Test tubes and bungs
9. Oil bath
10. Water bath
11. Watch

## **Apparatus Setup**



### **Procedure to Determine the Boiling Point of Benzene**

1. Take a capillary tube and close its one end by holding the end in the flame and rotate it for 2–3 minutes.
2. Transfer a few mL of benzene in the fusion tube.
3. Dip the capillary tube into the liquid in the fusion tube keeping the sealed end up.
4. Insert the tube in one of the holes of aluminium block and insert the thermometer in the other hole.
5. Make sure that the liquid is visible in the fusion tube after it is put in the hole.
6. Place the aluminum block on the tripod.
7. Start heating the block with the help of kerosene burner.
8. Note down the temperature soon as the regular streams of bubbles are seen out of liquid in the fusion tube.

### **Procedure to Determine the Boiling Point of Benzaldehyde**

1. Close one end of the capillary tube by holding one end in the flame and rotate it for 2–3 minutes.
2. Transfer a few drops of benzaldehyde in the fusion tube.

3. Dip the capillary tube into the benzaldehyde liquid filled in the fusion tube keeping the sealed end up.
4. insert the thermometer in one hole of aluminium block and Insert the tube in one of the holes.
5. Make sure that the benzaldehyde liquid is visible in the fusion tube after it is put in the hole.
6. Place the aluminum block on the tripod and begin heating of the block with the help of kerosene burner.
7. Note down the temperature soon as the regular streams of bubbles are seen out of liquid in the fusion tube.

### Observation

	Temperature (T – 1)	Temperature (T – 2)	Difference between = (T – 1) + (T – 2)/2
M.P. of Naphthalene			
M.P. of Benzoic acid			

### Result

The boiling point of given organic compound benzaldehyde 178 °C is about and benzene to be 78 °C.

### Uses

1. It is useful in supporting the identification of a compound, as well as serving as a rough guide to the relative purity of the sample.
2. Precautions to be taken during the experiment.
3. Ensure that the capillary tube is properly sealed.
4. The seal point of the capillary tube should be well within the liquid.

### Questions

1. What is the boiling point of water?
2. What is the boiling point of benzene?
3. What is the boiling point of benzaldehyde?
4. Why carboxylic acids have higher boiling point than hydrocarbons?

5. What is the boiling point of water in hilly regions?





# Chapter 7

## To Determine and Report the Solubility Behaviour of the Given Sample

### Principle

The objective of this experiment is to investigate the solubility characteristics of some simple organic molecules and to look at relationships between the solubility properties of an organic molecule and its structure.

This leads to the important idea that structure dictates function which means that by knowing the structure of a molecule, we should be able to predict its “function” such as its solubility, acidity or basicity, stability, reactivity etc. Predicting the solubility of an organic molecule is a useful skill. For example, it can be useful when trying to purify (e.g. recrystallization: picking a suitable solvent) or isolate it from a multi-component reaction mixture (e.g. via extraction), or when extracting a molecule from a natural source such as a plant, or designing a new pharmaceutical that needs to be soluble in the blood stream (aqueous and alkaline), the stomach (aqueous and acidic) or enter the central nervous system (“fatty”). You will learn more about some of these topics and techniques later this semester. For example, you will extract and then isolate caffeine from tea leaves - an “experiment” that you may perform several times per day while making tea (or similarly for coffee)! Solubility is also an important consideration when performing reactions to synthesize molecules; usually, reagents and starting materials are mixed together in a solution. The partitioning of drug molecules within the body between the blood and various tissues is also related to solubility properties.

The solubility of the molecule depends on several intermolecular forces such as ion dipole, solvent-solute, solvent-solvent, etc. A compound dissolves in water if it can form hydrogen bonds with water. During dissolution of a solid or liquid, the ions or molecules separate out and the spaces in between them are taken up by the formation of new bonds

between solvent and the solute particles. In general, the solubility of organic compound gives valuable information regarding its chemical nature.

Most organic molecules are typically relatively non-polar and are usually soluble in organic solvents (e.g. diethyl ether, dichloromethane, chloroform, petroleum ether, hexanes etc.) but not in polar solvents like water. However, some organic molecules are more polar and therefore more soluble in water.

### **Requirements**

**Chemicals:** as per specific requirement.

1. Diethyl ether
2. Methanol
3. Distilled water (100 ml)
4. Ethanol
5. 5 % NaOH
6. 5 % HCl
7. Conc.  $\text{H}_2\text{SO}_4$
8. 5 %  $\text{NaHCO}_3$

### **Glassware/Apparatus**

1. Micro spatula glass
2. Glass rod
3. Graduated cylinder (10 ml)
4. Latex bulb
5. Transfer pipette
6. pH paper
7. Test tubes (6–10)

### **Procedure**

1. Take few test tubes and label them as A, B, C, D..... and so on.
2. To the first test tube add sample and water, shake well and observe the solubility.
3. To the second test tube add ether, shake well and observe the solubility.
4. Take Ethanol as a solvent and observe solubility in another test tube.
5. Take 5 % NaOH solution and sample and check the solubility.

6. Take 5 %  $\text{NaHCO}_3$  and check the solubility.
7. Take 5 %  $\text{HCl}$  and observe the solubility.
8. Take concentrated  $\text{H}_2\text{SO}_4$  and sample and observe the solubility of the given sample.

### **Water Solubility**

Place 0.05 mL or 25 mg of compound in a small test tube and add 0.75 mL of water in small portions. Shake test tube vigorously after the addition of each portion of solvent. If water soluble; otherwise proceed to step.

### **Ether Solubility**

Place 0.05 mL or 25 mg of compound in a small test tube and add 0.75 mL of diethyl ether in small portions. Shake test tube vigorously after the addition of each portion of solvent. If the compound is both water and ether soluble, the acid-base properties of the compound should be determined with litmus.

litmus turns red - water soluble acidic compound

litmus turns blue - water soluble basic compound

litmus neutral - water soluble general compound

### **Result**

The solubility of given organic compound methanol and ethanol is soluble in water.

### **Uses**

It is useful in supporting the identification of a compound, as well as serving as a rough guide to the relative purity of the sample.

### **Precautions to be taken during the Experiment**

1. Properly Wear gloves and goggles throughout this experiment.
2. Phenol is very toxic substance; if contact with skin may cause severe burns or systemic poisoning as phenol is readily absorbed through the skin, which cause irritation on skin.
3. Benzoyl chloride is very toxic; it's lachrymatory.
4. Phenol and Benzoyl chloride should be handled with care under a fume cup board.

5. If any problems occur you spill a lot of either of these, wash it off with lots of water and notify your laboratory instructor or teacher.



## Chapter 8

### To Determine and Report the Elements Present in the Given Sample (Sulphur, Nitrogen, Carbon and Halogens)

#### Principle

**Qualitative chemical analysis** deals with the identification of elements or grouping of elements present in each sample. These techniques can be employed in qualitative analysis vary in complexity, depending on the nature of the sample. It is necessary only to verify the presence of certain elements or groups for which specific tests applicable directly to the sample (e.g., flame tests, spot tests) may be available.

In more cases the sample is a complex mixture, and a systematic analysis must be made in order that all the constituents may be identified. It is to classify:

1. Qualitative inorganic analysis
2. Qualitative organic analysis

The basic organic nature of a compound is generally indicated by its behaviour on being heated in air; solids usually melt, then burn with either a smoky or non-smoky flame, in some instances leaving a black residue of carbon. The most of the elements (carbon, hydrogen, oxygen, nitrogen, sulfur, and, occasionally, phosphorus, halogens and other metal) are usually present in these organic compounds. These elements are covalently bonded to the different organic compounds. These elements are detected by using a different test in order to, must be converted into their ionic forms. This is proceeding by fusing the organic compound with a sodium metal. These ionic compounds formed during the fusion are extracted in aqueous solution and can be detected by simple chemical tests. This test or extract is mostly called as sodium fusion extract or Lassaigne's extract.

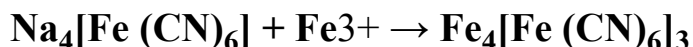
#### Test for Nitrogen and Carbon elements

Carbon and nitrogen molecule are present in the organic compound on fusion with sodium metal gives sodium cyanide ( $\text{NaCN}$ ) soluble in water. Which is converted into sodium ferrocyanide by the addition of small

quantity of ferrous sulphate. The ferric ions are formed during the process react with ferrocyanide to form prussian blue precipitate of ferric ferrocyanide.



Sodium ferrocyanide

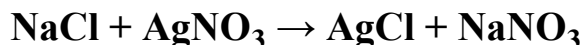


Ferric ferrocyanide

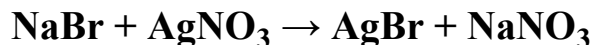
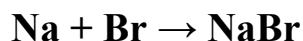
### **Test for Halogen Elements**

Halogens are functional group are present in a different organic compound forms sodium halide on fusion with sodium metal. Sodium halide extracted with water can be easily identified by addition of silver nitrate solution then acidifying with dilute  $\text{HNO}_3$ .

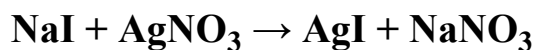
White curdy precipitate soluble in ammonium hydroxide solution is formed, when confirm that chlorine is present in this organic compound.



If bromine group is present in organic compound, a yellowish white precipitate may be occurring, which is sparingly soluble in ammonium hydroxidesolution is formed.

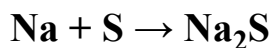


If iodine molecule is present in organic compound, a yellow precipitate may be occurring which is insoluble in ammonium hydroxide solution is formed.



### **Test for Sulphur**

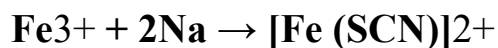
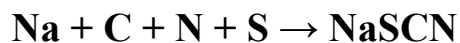
Sodium fusion will convert it into sodium sulphide. These sulphide ions are readily identified using sodium nitroprusside, which confirm that sulphur are present in this organic compound.



Sodium nitroprusside (violet colour)

### **Test for both Nitrogen and Sulphur**

If both elements such as nitrogen and sulphur are present in an organic compound, sodium fusion will convert it into sodium thiocyanate which then react with  $\text{Fe}^{3+}$  to form blood colour complex  $[\text{Fe}(\text{SCN})]^{2+}$



### **Requirements**

**Chemicals:** as per specific requirement.

1. Sodium metal
2. Distilled water (100 ml)
3. Ferric chloride solution
4. Ferrous sulphate solution (freshly prepared)
5. Conc. HCl
6. Acetic acid
7. Sodium nitroprusside
8. Lead acetate solution
9. Chlorine water
10. Carbon disulphide
11. Conc.  $\text{HNO}_3$
12. Aluminum hydroxide solution
13. Silver nitrate solution

### **Glassware/Apparatus**

1. Fusion tube (Ignition tube)
2. Test tube

3. Test Tube holder
4. Bunsen burner
5. Tripod stands with clamp
6. Gauze
7. Spatula
8. Wire gauze
9. Test tubes stand
10. Tongs
11. Funnel
12. Forceps
13. China dish
14. Filter paper
15. Glass rod
16. Dropper

## **Procedure**

### **(i) Preparation of sodium fusion extract (Lassaigne's extract)**

1. Take a small piece of dry sodium in a fusion tube.
2. Then heat the tube slightly on a Bunsen burner so that the sodium melts to a shining globule.
3. Add a pinch of the organic compound.
4. Heat it slowly to start with so that the compound reacts with sodium metal.
5. Now heat the tube strongly till it becomes red hot.
6. Plunge the red hot tube into a china dish containing distilled water.
7. Crush the contents with a glass rod and heat to boiling point.
8. Stop heating and remove the insoluble matter by filtration.
9. The filtrate is called Lassaigne's Extract.

### **(ii) Detection of Nitrogen**

1. Firstly, select organic compound, which element is identified.
2. Place ferrous sulphate ( $\text{FeSO}_4$ ) solution into the Lassaigne's extract in a test tube.
3. Start on the knob of the burner to turn it on.
4. Heat the test tube over the Bunsen burner.



5. Then added conc. HCl into the test tube containing Lassaigne's extract, by the using of the dropper towards the test tube.
6. Carefully added ferric chloride ( $\text{FeCl}_3$ ) solution into the test tube containing Lassaigne's extract, by the using of dropper towards the test tube.
7. The ferric ions are formed during the process react with ferrocyanide to form prussian blue precipitate of ferric ferrocyanide.
8. Confirm that the nitrogen is present in this organic compound.

### **(iii) Detection of Sulphur**

#### **(a) Sodium nitroprusside test**

1. Place a Lassaigne's extract in a test tube.
2. Then added a 1 ml of sodium nitroprusside solution into it test tube by using a dropper.

#### **(b) Lead acetate test**

1. Take 1 ml of acetic acid into the test tube then added a small quantity of Lassaigne's extract.
2. Then added lead acetate solution into the test tube containing Lassaigne's extract by using a dropper.

### **(iv) Detection of Chlorine**

#### **Silver nitrate test:**

1. Place a conc.  $\text{HNO}_3$  into the test tube containing Lassaigne's extract, drag the test tube towards it.
2. Heat the test tube over the Bunsen burner.
3. Then added a 1 ml of silver nitrate ( $\text{AgNO}_3$ ) solution into the test tube containing Lassaigne's extract, drag the dropper towards the test tube.
4. Pour ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) solution into the test tube containing Lassaigne's extract, drag the dropper towards the test tube.

### **(v) Detection of Bromine and Iodine**

#### **a) Silver nitrate test**

1. Place conc.  $\text{HNO}_3$  into the test tube containing Lassaigne's extract, drag the test tube towards it.
2. Drag the test tube over the Bunsen burner to heat it.
3. Then carefully added a 1 ml of silver nitrate ( $\text{AgNO}_3$ ) solution into the test tube containing Lassaigne's extract, drag the dropper toward the test tube.
4. To pour ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) solution into the test tube containing Lassaigne's extract, drag the dropper towards the test tube.

**b) Carbon disulphide test**

1. Take a dilute  $\text{HCl}$  in a test tube then added a small quantity of Lassaigne's extract
2. Then added a Carbon disulphide into the test tube containing Lassaigne's extract, drag the dropper towards the test tube.
3. Carefully added chlorine water into the test tube containing Lassaigne's extract by using a dropper.
4. Then shake well the test tube.

**Observation**

S. No.	Test	Experiment	Observation	Inference
1	Sodium nitroprusside Test	To a small portion of Lassaigne's extract, add a few drops of sodium nitroprusside solution.	Appearance of a purple color.	Sulphur present
2	Lead acetate Test	Acidify a small portion of Lassaigne's extract with a few drops of acetic acid and add a few drops of lead acetate solution.	Black precipitate occurs.	Sulphur present
3	Nitrogen Test	To a small portion of the Lassaigne's extract, add 2 ml of freshly prepared ferrous sulphate solution and heat. Now add 2-3 drops of ferric	Appearance of a prussian blue color occurs.	Nitrogen present.

		chloride solution and acidify with conc. hydrochloric acid.		
4	Silver nitrate Test	To a small portion of Lassaigne's extract add 1 ml of conc. nitric acid and boil for some time. Cool the contents and to it add silver nitrate solution.	White ppt. soluble in ammonium hydroxide forms.	chlorine present
			Pale yellow precipitate sparingly soluble in ammonium hydroxide forms.	Bromine present
5	Carbon disulphide Test	Acidify a small portion of Lassaigne's extracts with dil. HCl and add a few drops of carbon disulphide. Now add freshly prepared chlorine water and shake vigorously.	Appearance of orange colour in the carbon disulphide layer.	Bromine present
			Appearance of violet colour in the carbon disulphide layer.	Iodine present

### Result

The given sample of organic compound contain.....

### Precautions

1. Sodium metal is highly reactive. When exposed to air, it reacts even with the moisture present in the atmosphere. It also reacts with the sweat of hands. Hence do not hold it with hands. Always use forceps while handling sodium.

2. Before using sodium, press it within the folds of the filter paper to remove kerosene oil.
3. Use dry ignition tubes for sodium fusion. Sodium reacts with water violently.
4. Put the unused sodium piece back in the bottle containing kerosene oil. Do not throw it into the sink.
5. Repeat the process of sodium fusion with three ignition tubes. This is to ensure that the fusion has taken place.
6. After immersing the red-hot ignition tube in water, break it with a glass rod gently. Boil the contents for 2–3 minutes to extract the soluble sodium salts in water.



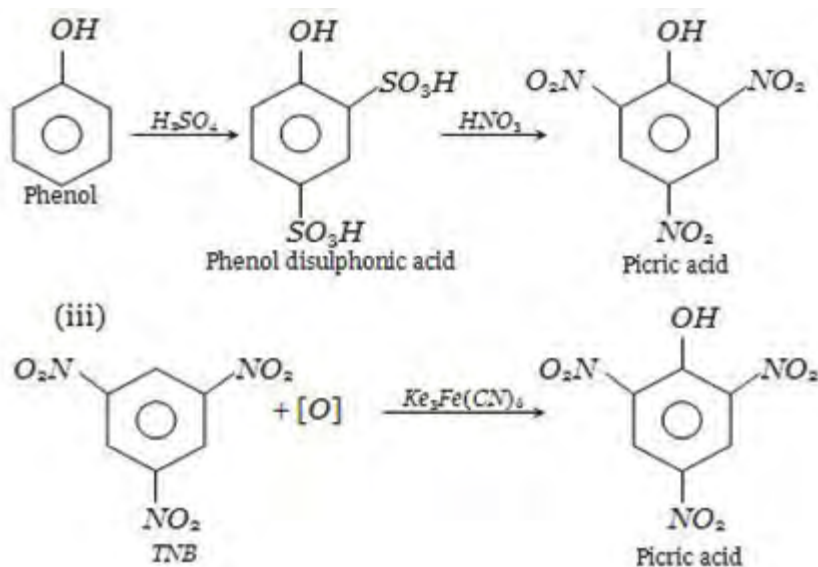
## Chapter 9

### To Prepare and Submit 2, 4, 6 – Tribromophenol(Picric Acid) from Phenol and Report its Melting Point

#### Principle

2,4,6-trinitrophenol are aromatic hydrocarbons, these or their substituted derivatives when react with con. nitric acids in the presence of concentrated sulphuric acid undergo nitration reaction (electrophilic substitution reaction), (i. e., hydrogen atoms are replaced by nitro group). In some cases, nitration can be carried out even in the absence of sulphuric acid.

Electrophilic aromatic substitution reaction is an important class of synthesis of organic compound. Substituents already present in the benzene nucleus determine the position and extent of substitution of the new incoming groups. Picric acid is an organic compound with the formula  $(\text{O}_2\text{N})_3\text{C}_6\text{H}_2\text{OH}$ . Its IUPAC name is 2,4,6-trinitrophenol (TNP). The name «picric» comes from the Greek (pikros), meaning “bitter”, reflecting its bitter taste. It is one of the most acidic phenols. Like other highly nitrated organic compounds, picric acid is an explosive, hence its primary use. It has also been used in medicine (antiseptic, burn treatments) and dyes.



## Requirements

### Chemicals

Phenol	= 7.5 ml
Nitric Acid	= 22.5 ml
Sulphuric acid	= 20 ml
Cold water	= 100 ml

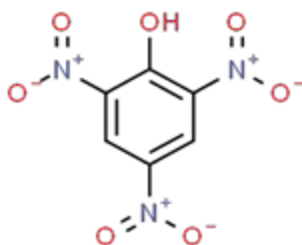
### Glassware/Apparatus

1. Erlenmeyer flask (250 mL)
2. Volumetric flask
3. Pipette
4. Glass stirring rod
5. Buchner funnel
6. Suction pump
7. Filter papers
8. Measuring cylinder
9. Rubber clock
10. Fume hood

### Chemical Formula



#### Chemical Formula



### IUPAC Name

2,4,6-Trinitrobenzenol

### Other Names

1. Picric acid
2. Carbazotic acid

3. Phenol trinitrate
4. Picronic acid
5. Trinitrophenol
6. 2,4,6-Trinitro-1-phenol
7. 2-Hydroxy-1,3,5-trinitrobenzene
8. TNP
9. Melinite

## Procedure

1. Take 7 g of phenol and 20 ml of conc. Sulfuric acid in a 500 ml dried round bottomed flask. Shake the mixture and heat on a water bath for 30 min.
2. During this period, a clear solution of o – and p-phenol sulfonic acids are obtained. Cool the flask in an ice-bath.
3. Now add 22 ml of conc.  $\text{HNO}_3$  drop wise with constant shaking. An exothermic reaction takes place and copious red fumes (oxide of nitrogen) are evolved and the liquid becomes deep red in colour.
4. Heat the flask in a water-bath for 2 hr. with occasional shaking. Cool the flask and to it add 100 ml of water and cool again.
5. Filter the solid and wash thoroughly with cold water, recrystallize the crude picric acid from aqueous hot alcohol, m.p.  $122^\circ\text{C}$ , yield 15.3 g.

## Calculation

Here limiting reagent is phenol; hence yield should be calculated from its amount taken.

Molecular formula of phenol	$= \text{C}_6\text{H}_6\text{O}$
Molecular formula of picric acid	$= \text{C}_6\text{H}_3\text{N}_3\text{O}_7$
Molecular weight of phenol	$= 94 \text{ g/mol}$
Molecular weight of picric acid	$= 229.10 \text{ g/mol}$

## Theoretical Yield

94 g of phenol reacts with benzoyl chloride to give a 229.10 gm of picric acid. Therefore, 7.5 of phenol will give? (X)..... gm of picric acid.

$$X = (229.10 \times 7.5)/94 = 18.27 \text{ g}$$

Theoretical yield = 18.27 g

Practical yield = assume 15.3  
gm  
Percentage = (Practical yield  
practical yield  $\times 100$ )  
=  $(15.3 \times 100) /$   
18.27  
= 83.74 %

## Properties

1. It is a yellow crystalline solid, melting point 122 °C.
2. It is insoluble in cold water but soluble in hot water and in ether.
3. It is bitter in taste.
4. Due to the presence of three electronegative nitro groups, it is a stronger acid than phenol and its properties are comparable to the carboxylic acid.
5. It neutralizes alkalies and decomposes carbonates with evolution of carbon dioxide.

## Result

Picric acid was synthesized by the reaction of phenol and nitric acid, the crude product was obtained, and the practical yield was found to be 15.3 gm and % yield about 83.74%.

The melting point of picric acid was found to be 122 °C.

## Uses

1. It is used as a yellow dye for silk and wool, as an explosive and as an antiseptic in treatment of burns.
2. By far, the largest use has been in munitions and explosives.
3. Explosive D, also known as Dunnite, is the ammonium salt of picric acid more powerful but less stable than the more common explosive TNT (which is produced in a similar process to picric acid but with toluene as the feedstock).
4. Picramide, formed by aminating picric acid (typically beginning with Dunnite), can be further aminated to produce the highly stable explosive TATB.



5. It has found some use in organic chemistry for the preparation of crystalline salts of organic bases (picrate's) for the purpose of identification and characterization.
6. In metallurgy, a 4% picric acid in ethanol etch called "picral" has been commonly used in optical metallographic to reveal prior austenite grain boundaries in ferritic steels.
7. The hazards associated with picric acid have meant it has largely been replaced with other chemical etchants. However, it is still used to etch magnesium alloys, such as AZ31
8. Clinical chemistry lab testing utilizes picric acid for the Jaffe reaction to test for creatinine. It forms a colored complex that can be measured using spectroscopy.

### **Safety and Precautions must be taken during Reaction Performed**

1. Properly Wear gloves and goggles throughout this experiment.
2. Phenol is very toxic substance; if contact with skin may cause severe burns or systemic poisoning as phenol is readily absorbed through the skin, which cause irritation on skin.
3. Phenol should be handled with care under a fume cup board.
4. Overall mixing of nitric acid and sulphuric acid in a reaction is done in a fume cup hood/Fuming chamber.



# Chapter 10

## Synthesis and Characterization of Phenyl Benzoate from Phenol by Acetylation Reaction

### Principle

Synthesis of phenyl benzoate from phenol is an example of Schotten-Baumann Reaction where phenols react with an aromatic acid chloride in the presence of an excess of a base at room temperature to form an ester. In this reaction, phenol is shaken with benzoyl chloride and excess amount of sodium hydroxide solution, it is benzoylated to give the ester, phenyl benzoate. The phenol is first converted into the ionic compound sodium hydroxide to give sodium phenoxide (sodium phenate) by dissolving in sodium hydroxide solution. The phenoxide ions reacts more rapidly with benzoyl chloride than the original phenol does, but even so you have to shake it with benzoyl chloride for about 15 minutes. Solid phenyl benzoate is formed to give phenyl benzoate.

The introduction of a benzoyl group in place of the active hydrogen of hydroxyl, amino group is known as benzoylation reaction. The reaction is somewhat like acetylation except that here the reagent used is benzoyl chloride in presence of NaOH and not the benzoic anhydride.

### Requirements

**Chemicals:** as per specific quantity require to reaction.

Phenol	= 1 gm
Benzoyl chloride	= 2 ml
Ethanol	= 50 ml
10% sodium hydroxide	= 15 ml
Cold water	= 100 ml
Methanol	= 100 ml

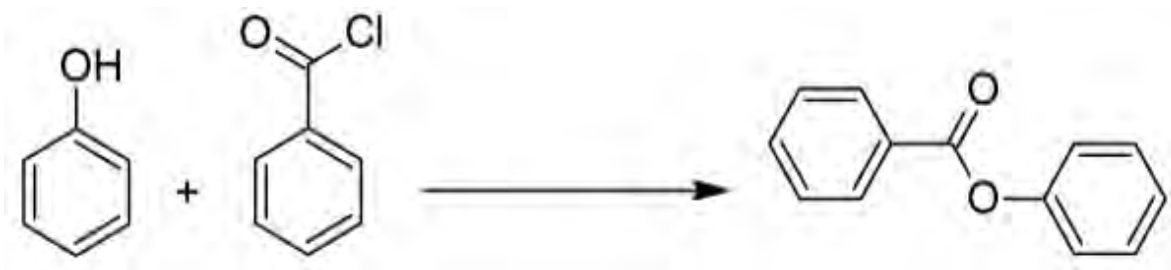
### Glassware/ Apparatus

1. Erlenmeyer flask (250 mL)

2. Volumetric flask
3. Pipette
4. Glass stirring rod
5. Buchner funnel
6. Suction pump
7. Filter papers
8. Measuring cylinder
9. Rubber cork
10. Fume hood
11. Spatula

### Chemical Reaction

Phenyl benzoate is a phenyl ester of benzoic acid. Phenol is treated with benzoyl chloride in presence of sodium hydroxide for preparation phenyl benzoate.



### Procedure

1. Take a 1.0 g of phenol and add a 15 mL of 10% sodium hydroxide solution in 250 ml Erlenmeyer flask with a good rubber cork and shake well at room temperature.
2. Carefully add 2.0 mL of benzoyl chloride (under fume cup hood), cork the flask and shake the mixture vigorously with intermittent pressure release for at least 15–20 minutes.
3. The reaction should be complete, and a solid product obtained is filter off with suction pump on Buchner funnel, breaking up any lumps occurs in a crude product on the filter with a spatula, and then wash thoroughly with ice water.
4. Erlenmeyer flask rinse with cold water in 2 times to remove any residual crystals which may be present in a crude product.

5. Phenyl benzoate as possible from the Buchner funnel to a clean, dry 250 mL beaker and recrystallize it from methanol.
6. Weigh the recrystallized phenyl benzoate and calculate the practical yield about 1.8 gm.
7. Determine the melting point of the synthesized phenyl benzoate.
8. It's obtained as colorless needles; melting point is 68–69°C.

### Calculation

Here limiting reagent is phenol; hence yield should be calculated from its amount taken.

Molecular formula of phenol	= C <sub>6</sub> H <sub>6</sub> O
Molecular formula of phenyl benzoate	= C <sub>13</sub> H <sub>10</sub> O <sub>2</sub>
Molecular weight of phenol	= 94 g/mol
Molecular weight of phenyl benzoate	= 198 g/mol

### Theoretical Yield

94 g of phenol reacts with benzoyl chloride to give a 198 g of phenyl benzoate.

Therefore, 1.0 g of phenol will give? (X)..... g of phenyl benzoate.

$$X = (198 \times 1.0) / 94 = 2.11 \text{ g}$$

Theoretical yield	= 2.11 g
Practical yield	= assume 1.8 gm.
Percentage practical yield	= (Practical yield / theoretical yield) × 100
	= (1.8 × 100) / 2.11
	= 85%

### Properties

1. White solid powder.
2. Insoluble in water and soluble in other organic solvent.
3. Boiling point is about 158°C.
4. Melting point is 68–69°C.

### Result

Phenyl benzoate was synthesized by the reaction of phenol and benzoyl chloride, crude product was obtained, and the practical yield was found to be 1.8 gm and % yield about 85%. The melting point of phenyl benzoate was found to be 68-69°C.

### **Uses**

1. Phenyl Benzoate is used as an excellent starting material to produce optical components, particularly high-quality lenses for still and motion picture cameras.
2. It is also used in a variety of polyesters, which have application in products from clothing to heavy industry to the preparation of new generation of cloths. Phenyl benzoate is a white powdery organic compound that falls into the broad category of chemicals known as esters.
3. The compound is formed in a reaction between phenol, sodium hydroxide and benzoyl chloride.
4. The compound is solid at room temperature but can form an oily liquid at a relatively low temperature.
5. One use that takes advantage of the electrical properties of phenyl benzoate is the development of liquid crystal displays.
6. Phenyl benzoate based liquid crystals have excellent compatibility characteristics with other materials used in liquid crystal displays, such as biphenyl, phenyl cyclohexane, bicyclohexane and fluorine types, especially at low temperatures.

### **Safety and Precautions must be taken during Reaction Performed**

1. Properly Wear gloves and goggles throughout this experiment.
2. Phenol is very toxic substance; if contact with skin may cause severe burns or systemic poisoning as phenol is readily absorbed through the skin, which cause irritation on skin.
3. Benzoyl chloride is very toxic; it's lachrymatory.
4. Phenol and Benzoyl chloride should be handled with care under a fume cup board.
5. If any problems occur you spill a lot of either of these, wash it off with lots of water and notify your laboratory instructor or teacher.

## Questions

1. What is the basic purpose of the addition of 10% sodium hydroxide to the benzylation of phenol with benzoyl chloride?
2. What is benzylation reaction?
3. Explain the basic mechanism and reaction of Schotten–Baumann Reaction.
4. Which solvent may be used to the recrystallization of crude product?
5. Define the melting point and recrystallization process of phenyl benzoate.

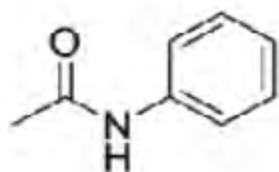


# Chapter 11

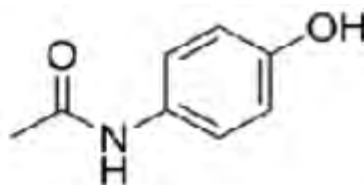
## Synthesis and Characterization of Acetanilide from Aniline by Acetylation Reaction

### Principle

**Primary amines react with acid chlorides or anhydrides to form mono acetyl derivatives.** Acetanilide is an organic chemical compound (meaning it's composed of carbon and hydrogen mostly) that is classified as an amide in terms of its functional group. This means that it has the carbonyl group (carbon-oxygen double bond) bonded directly to a nitrogen atom. It also contains an aromatic ring, which is a ring composed of six carbon atoms and an alternating double-single-double-single bonding pattern all around the ring. Acetanilide is an analgesic, which was formally known as Antifebrin, and is structurally like acetaminophen (or Tylenol). However, unlike acetaminophen, acetanilide is toxic. Acetanilide is prepared from aniline using an acetylation reaction. Acetylation is often used to place an acetyl protecting group on primary or secondary amines to reduce their reactivity toward oxidizing agents or electrophiles. Acetamides are usually crystalline solids which can be a help in purification by recrystallization. The melting points can be used for characterization and identification of the corresponding compounds.



acetanilide



acetaminophen (Tylenol)

### Requirements

**Chemicals:** as per specific quantity required.

Aniline	= 1 gm
Glacial acetic acid	= 2 ml

Acetic anhydride	= 50 ml
Zinc dust	= 15 ml
Distilled water	= 100 ml

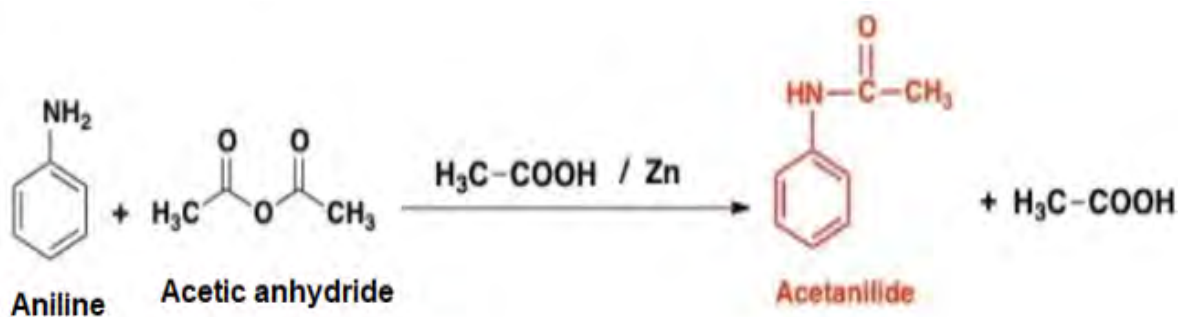
### **Glassware/Apparatus**

1. Erlenmeyer flask (250 mL)
2. Conical flask
3. Pipette
4. Reflux condenser
5. Glass stirring rod
6. Buchner funnel
7. Suction pump
8. Filter papers
9. Measuring cylinder
10. Rubber cork
11. Fume hood
12. Spatula

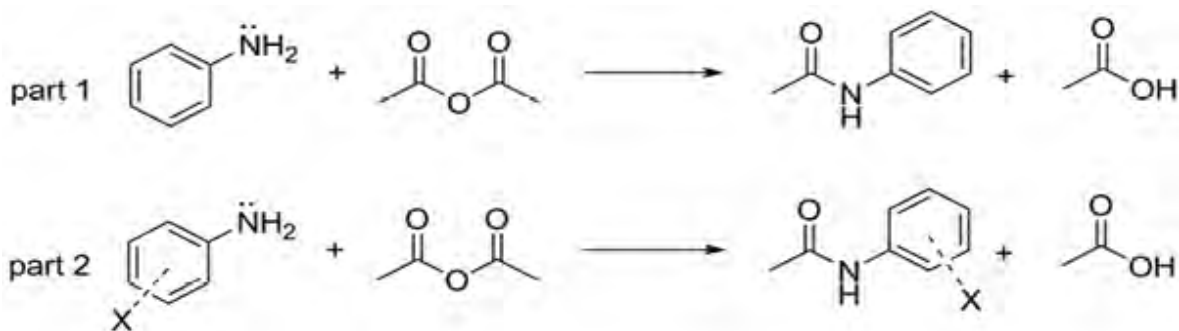
### **Chemical Reaction**

Acetanilide is prepared from aniline when it acylating with acetic anhydride in presence of glacial acetic acid and zinc dust. Aniline or phenylamine is a primary amine and basic in nature. Acetic anhydride as anhydride of acetic acid, act as a source of acyl group. Aniline reacts with acetic anhydride to form acetanilide by nucleophilic substitution reaction and the reaction is called acylation reaction. In this reaction aniline acts as a nucleophile and acyl ( $\text{CH}_3\text{CO}-$ ) group from acetic anhydride act as a electrophile. Hence the hydrogen atom of  $\text{NH}_2$  group is replaced by the acyl group. Zinc is used to prevent the oxidation of aniline during the chemical reaction. Acetanilide is medicinally important, and it is used as febrifuge.:





Other names – N-phenyl acetamide, N-phenylethanamide, Acetanil



*X = unknown substituent group*

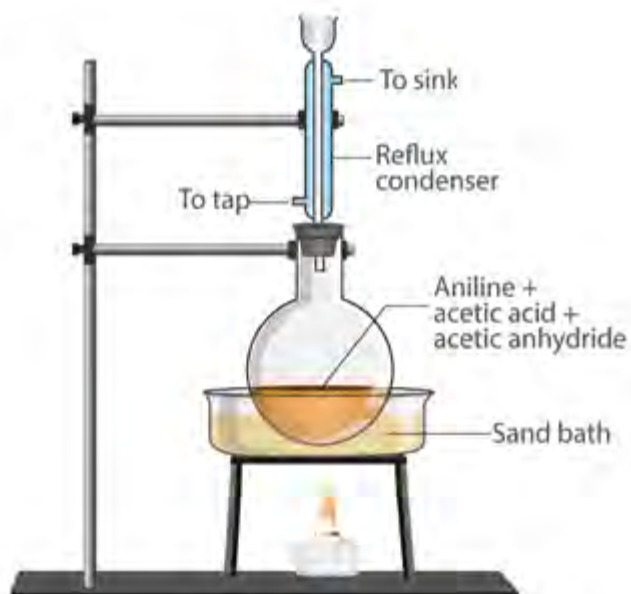


Fig 1 Preparation of acetanilide

## Procedure

1. Wash out all the apparatus with distilled water before starting the experiment.
2. Prepare a mixture of 10 ml of glacial acetic acid and 10 ml acetic anhydride in a beaker.
3. Place 10 ml (10.3 gm) of aniline in a round bottom flask and carefully add 20 ml of acetic anhydride and glacial acetic acid mixture (equal volumes) and add a zinc dust.
4. Set up the reflux condenser with the round bottom flask.
5. Heat the reaction mixture gently for about 15–20 minutes on oil bath.
6. Reaction mixture quickly crystalize.
7. Pour the hot reaction mixture in a beaker containing ice cold water with constant stirring.
8. Carefully stir the reaction mixture vigorously to hydrolyze excess of acetic anhydride.
9. Reaction mixture was recrystallized from about 60 ml mixture of one volume of acetic acid and two volumes of water.
10. Crude product of acetanilide is precipitated, collect and filter of the colourless crystals at the suction pump, again wash thoroughly with water.
11. Dried the crude product of acetanilide
12. Recrystallization the crude product by using a 30 ml ethanol.
13. Weigh the crude product and calculate the practical yield and obtained 12 gm and measure the melting point about 114°C.

### Calculation

Here limiting reagent is aniline; hence yield should be calculated from its amount taken.

Molecular formula of acetanilide	= $C_8H_9O_1N_1$
Molecular formula of aniline	= $C_6H_7N_1$
Molecular weight of aniline	= 93 g/mol
Molecular weight of acetanilide	= 135 g/mol

### Theoretical Yield

93 gm of aniline reacts with acetic anhydride to give a 135 gm of acetanilide.

Therefore, 10.3 g of aniline will give? (X)..... gm of acetanilide

$$X = (135 \times 10.3)/93 = 14.95 \text{ g}$$

Theoretical yield = 14.95 gm.

Practical yield = assume 12 gm.

Percentage practical yield =  $\frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100$

$$= \frac{12 \times 100}{14.95}$$

$$= 93.6\%$$

## Properties

1. It is a white solid with a flaky appearance. This is an odorless compound.
2. Insoluble in water and soluble in other organic solvent.
3. The boiling point for this substance is 304°C.
4. Melting point is 114°C.

## Result

Acetanilide was synthesized by the reaction of aniline, acetic anhydride and glacial acetic acid. The crude product is obtained, practical yield was found to be 12 gm and % yield to be 80.26 %.

The melting point of acetanilide was found to be 114°C.

## Uses

1. Acetanilide is used in the synthesis of penicillin and in other pharmaceuticals.
2. It is also used as an antipyretic agent means fever reducing agent.
3. It was used in the past to treat fever and headache and was known as Antifebrin.
4. Acetanilide is used as an inhibitor of hydrogen peroxide decomposition and is used to stabilize cellulose ester varnishes. It is also found uses in the intermediation in rubber accelerator synthesis, dyes and dye intermediate synthesis, and camphor synthesis.
5. Acetanilide is used for the production of 4-acetamidobenzenesulfonyl chloride, a key intermediate for the manufacture of the sulfa drugs.
6. It is also a precursor in the synthesis of penicillin and other pharmaceuticals.

7. In the 19<sup>th</sup> century acetanilide was one of a large number of compounds used as experimental photographic developers.
8. Acetanilide was the first aniline derivative found to possess analgesic as well as antipyretic properties.

### **Precautions**

1. Prolonged heating and uses of excess of anhydride should be avoided, otherwise variable amount of the diacetyl would result.
2. Recrystallization of the product from an aqueous solvent such as dilute alcohol avoids the diacetyl derivatives because the latter hydrolyses to the mono acetyl compound in the presence of water.
3. Do not inhale the fumes of acetic anhydride.
4. Always carry out experiments in fuming chamber or near the window.
5. Use the water condenser for refluxing the reaction mixture.
6. Dry the crystals of acetanilide before finding the weight and its melting point.

### **Questions**

1. Name any two-acetylating agent.
2. What is the need to add zinc during the preparation of acetanilide?
3. What is nitrating mixture.
4. What is the IUPAC name for acetanilide?
5. Mention any two uses of acetanilide.



## Chapter 12

### To Identify and Report the Functional Groups Present in the Given Sample (Aldehyde and Ketone)

#### Principle

Organic compounds have been classified into various types, depending upon the elements present in them, for identification purposes. Hence the correct determination of the element or elements present in the compound is of immense importance for locating the class of the organic compound. In general, all organic compounds are expected to contain carbon and Hydrogen. Therefore, it is not essential to test for these elements. In addition to these the organic compound may contain one or more of the other elements especially nitrogen, sulfur, halogen (chlorine, bromine, iodine) and oxygen. Aldehydes and ketones constitute an important class of organic compounds containing the carbonyl group. Aldehyde has the structure  $\text{RCH(=O)}$  while a ketone has the structure of  $\text{R}_2\text{C(=O)}$ . Where R may be an alkyl, alkenyl, alkynyl or aryl group.

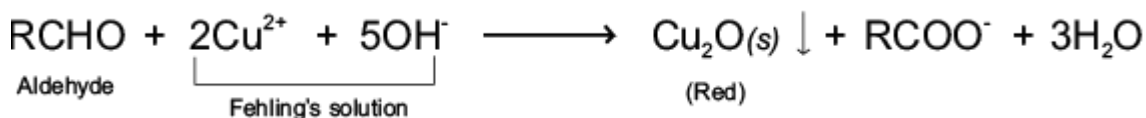
Aldehydes and ketones of low molecular weights are volatile compounds. Identification of aldehydes and ketones is based on two types of reactions, addition reaction to double bond and oxidation reaction.

The difference between ketone and aldehyde is the carbonyl group present in aldehydes can be easily oxidized to carboxylic acids whereas the carbonyl group in ketones are not oxidized easily. This difference in reactivity is the basis for distinction of aldehydes and ketones. They are generally distinguished by the following tests.

1. Schiff's test
2. Fehling's test
3. Tollen's test
4. Test with chromic acid
5. Sodium nitroprusside test

#### Fehling's Test

Fehling's solution is a complex compound of  $\text{Cu}^{2+}$ . When aldehyde compound is treated with Fehling's solution  $\text{Cu}^{2+}$  is reduced to  $\text{Cu}^+$  and the aldehyde is reduced to acids. During the reaction a red precipitate is formed.



Aromatic aldehydes do not respond to Fehling's test. An aqueous solution of the compound may be used instead of an alcoholic solution. Formic acid also give this test.

**Note: Appearance of red precipitate confirms the presence of aldehydic group.**

### Tollen's Test: (Silver Mirror Test)

This test is also called silver mirror test. Tollens reagent consists of silver ammonia complex in ammonia solution. Aldehydes reacts with Tollens reagent gives a grey black precipitate or a silver mirror. Always a freshly prepared Tollen's reagent should be used. Aldehydes are oxidized to the corresponding acid and silver in Tollens reagent is reduced from +1 oxidation state to its elemental form. Generally, ketones do not respond to this test.



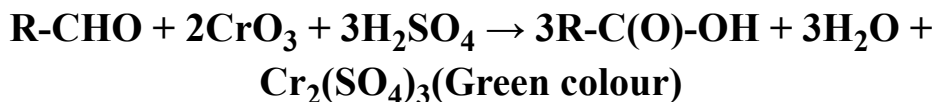
Apart from aldehydes some other compounds also respond to Tollen's test, but the presence of aldehydes is conformed when the given substance shows positive test for Tollens test but if the given compound passes 2,4-dinitrophenylhydrazine test.

Note: The appearance of shiny silver mirror conforms the presence of aldehydes.

### Test with Chromic Acid

Aldehydes react with chromic acid gives a green to blue precipitate. Ketones do not react with chromic acid. Some of the primary and secondary alcohols also gives this test but they do not give dinitrophenylhydrazine test.

The chemical reaction is given below.

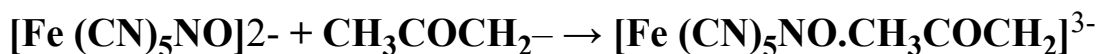
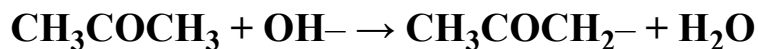


**Note:** The appearance of green or blue colour precipitate conforms the presence of aldehydes.

### Sodium Nitroprusside Test

Ketone responds to this test. Ketone reacts with alkali forms an anion further it reacts with sodium nitroprusside forms a colored complex ion. Aldehydes do not respond to this test.

The chemical reaction is given below.



**Note:** The appearance of red colouration shows the presence of ketone.

### Requirements

**Chemicals:** as per specific requirement.

1. Silver nitrate solution
2. Fehling's solutions A
3. Fehling's solutions B
4. Dilute ammonium hydroxide solution
5. 2,4-Dinitrophenylhydrazine reagent
6. Chromic acid
7. Sulfuric acid
8. Sodium bisulfite

### Glassware/Apparatus

1. Test tube
2. Pipette
3. Beaker
4. Test tube holder

### Procedure

### **(a) 2,4-Dinitrophenyl Hydrazine Test**

1. Place a beaker and add a 1 gm of formaldehyde dissolve in ethanol.
2. To this solution add alcoholic reagent of 2,4-dinitrophenyl hydrazine.
3. Shake the mixture well.
4. If there is a formation of yellow to orange precipitate, then the given compound is an aldehyde or ketone.
5. An orange precipitate is obtained from carbonyl compounds in which the C=O groups is conjugated with C=C.

### **(b) Sodium Bisulfite Test**

1. Take saturated solution of sodium bisulfite in a clean test tube.
2. Add 1 ml of given organic compound to be tested.
3. Shake well and leave it for 15–20 minutes.
4. If there is formation of white precipitate, then the presence of carbonyl group is confirmed.

### **(c) Fehling's Test**

1. Fehling's solution is prepared by mixing equal amounts of Fehling's A and Fehling's B solution having a blue colour.
2. Take the 1 gm of given organic compound in a clean test tube.
3. Add 2 drops Fehling's solution, shake, and heat the solution gently.
4. If a blue colour of Fehling solution will changed to brick red precipitate appears then the presence of aldehyde is conformed.

### **(d) Tollen's Test: (Silver Mirror Test)**

1. Take 1 ml of silver nitrate solution in a clean test tube.
2. Add 2 ml of 5% dilute sodium hydroxide solution to it, a brown precipitate form.
3. Add dilute ammonia solution dropwise till the brown precipitate of silver oxide dissolves.
4. To this freshly prepared Tollen's reagent add the given organic compound to be tested.
5. Place the test tube in warm water bath for about 5–10 minutes.



6. If there is the appearance of silver mirror in the sides of test tube confirms the presence of aldehyde.

**(e) Test with Chromic Acid:**

1. Take the given organic compound in a clean test tube.
2. Add 1 ml of chromic acid reagent to the given organic compound.
3. Appearance of green or blue colour precipitate indicates the presence of aldehydes.

**(f) Sodium Nitroprusside Test**

1. Dissolve sodium nitroprusside in distilled water in a clean test tube.
2. Add 1 ml of given organic compound to be tested.
3. Shake well and add sodium hydroxide solution dropwise.
4. If there is the appearance of red colour then the presence of ketone is conformed.

**Observations**

Name of Identification Test	Observation
2,4-Dinitrophenyl Hydrazine Test	Formation of orange yellow crystals indicates the presence of carbonyl group
Fehling's Test	Appearance of red precipitate confirms the presence of aldehydic group.
Tollen's Test (Silver Mirror Test)	The appearance of shiny silver mirror confirms the presence of aldehydes.
Test with Chromic Acid	The appearance of green or blue colour precipitate confirms the presence of aldehydes.
Sodium Nitroprusside Test	The appearance of red coloration shows the presence of ketone.

**Results**

The given organic compound has confirmed (aldehyde/ketone) functional group.

**Precautions**

1. The reagents should be freshly prepared to perform the test.
2. Not to heat the reaction mixture directly on flame.
3. After performing the tollens test wash the test tube with nitric acid to destroy the silver mirror, because it's an explosive substance.

### **Questions on Tests for Aldehydes and Ketones**

1. Name any one test to distinguish aldehyde from ketone.
2. What is Tollen's reagent?
3. What is the use of Schiff's reagent?
4. What is Fehling's solution?
5. What is Rochelle's salt?



# Chapter 13

## Synthesis and Characterization of Iodoform

### Principle

Iodoform is also called as triiodomethane and carbon triiodide is the organoiodine compound with the formula  $\text{C H I}_3$ . A pale yellow, crystalline, volatile substance, it has a penetrating and distinctive odor (in older chemistry texts, the smell is sometimes referred to as the smell of hospitals, where the compound is still commonly used) and, analogous to chloroform, sweetish taste. It is occasionally used as a disinfectant.

### Requirements

**Chemicals:** as per specific quantity required.

Acetone	= 3 ml
Potassium Iodide	= 2 ml
Sodium Hypochlorite	= 15 ml
Methanol	= 15 ml
Distilled water	= 100 ml

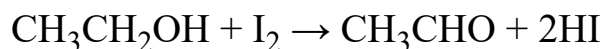
### Glassware/Apparatus

1. Erlenmeyer flask (250 mL)
2. Beaker
3. Pipette
4. Glass stirring rod
5. Buchner funnel
6. Suction pump
7. Filter papers
8. Measuring cylinder
9. Spatula
10. Water bath
11. Funnel

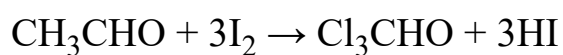
### Chemical Reaction

Iodoform were synthesized by the reactions of iodine vapour with steam over red-hot coals, and carefully added by the small quantity of potassium with ethanolic iodine in the presence of cold water. Another method which is also used, it is synthesized by the reaction of iodine and sodium hydroxide with the presence of a methyl ketone, acetaldehyde, ethanol & most of the secondary alcohols.

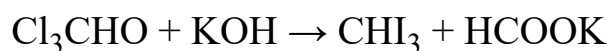
**From ethanol: Example.**



Acetaldehyde



Iodol

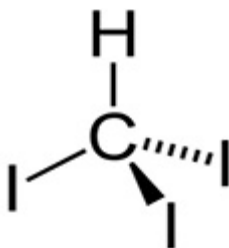


Iodol      Iodoform Potassium formate

### Haloform Reaction

The Reaction of iodine and base with methyl ketones is so reliable that the iodoform test (the appearance of a yellow precipitate) is used to probe the presence of a methyl ketone. This is also the case when testing for specific secondary alcohols containing at least one methyl group in alpha-position.

Chemical Structure



**IUPAC Name**

Triiodomethane

**Other Names**

Carbon triiodide

**Procedure**

1. Place 3 ml of acetone in a 30 ml water and add a 15 ml of sodium hydroxide (10 %) solution in an Erlenmeyer flask.
2. Then added an iodine solution (12.5 g of iodine dissolved in a solution of 25 g of KI in 100 ml of water drop wise) with constant shaking till the color of iodine persists.
3. Heat the contents on a water-bath at 60°C.
4. Add more iodine if the color disappears.
5. Heat till yellow precipitates settle down.
6. Cool the reaction mixture, filter the crude product and recrystallize from aqueous methanol.
7. Calculate the practical yield is 5 g, m.p. 119°C.

### Calculation

Here limiting reagent is acetone; hence yield should be calculated from its amount taken.

Molecular formula of acetone =  $C_3H_6O$

Molecular formula of Iodoform =  $CHI_3$

Molecular weight of acetone = 58.08 g/mol

Molecular weight of Iodoform = 397.732 g/mol

### Theoretical Yield

58.08 gm (3 ml) of acetone reacts with acetic anhydride to give a 397.732 g of Iodoform.

Therefore, 3.5 g of acetone will give? (X)..... g of Iodoform.

$$X = (397.732 \times 3.5) / 58.08 = 23.96 \text{ g}$$

Theoretical yield = 23.96 gm.

Practical yield = assume 20 gm.

Percentage practical yield =  $\left( \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100 \right)$

$$= \left( \frac{20}{23.96} \times 100 \right) / 23.96$$

$$= 83.6\%$$

### Properties

1. It is a pale light yellow, opaque crystals. This is a saffron like odour compound.
2. Soluble in water other organic solvent such as acetone, ethanol, diethyl ether.
3. The boiling point for this substance is  $218^{\circ}\text{C}$ .
4. Melting point is  $119^{\circ}\text{C}$ .

### **Result**

Iodoform was synthesized by the reaction of acetone. The crude product is obtained, practical yield was found to be 20 gm and % yield to be 83.6%. The melting point of phenyl benzoate was found to be  $119^{\circ}\text{C}$ .

### **Uses**

1. The compound finds small scale use as a disinfectant.
2. Around the beginning of the 20<sup>th</sup> century it was used in medicine as a healing and antiseptic dressing for wounds and sores, although this use is now superseded by superior antiseptics.
3. It is the active ingredient in many ear powders for dogs and cats, to prevent infection and facilitate removal of ear hair, along with zinc oxide and propanoic acid.
4. It is occasionally used as a disinfectant.

### **Safety and Precautions must be taken during Perform Practical**

1. Properly wear gloves and goggles throughout this experiment perform in a laboratory.
2. Acetone is more toxic and can be easily absorbed through the skin.
3. If any problems occur you spill a lot of either of these, wash it off with lots of water and notify your laboratory instructor or teacher.

### **Question**

1. Describe the observed changes in the reaction mixture. Are these chemical or physical changes?
2. What do you observe during this decomposition?
3. What elements do you think are present in iodoform? Can you guess a formula?

4. Give the formula and properties of iodoform.



# Chapter 14

## Synthesis and Characterization of Fluorescein Dye

### Principle

Fluorescein dye are synthesized by the reaction of phthalic anhydride and resorcinol in the presence of zinc chloride via the Friedel-Crafts reaction. Another method which can be used by the reaction of fluorescein uses methane sulfonic acid as a Brønsted acid catalyst. This method gives a high yield under milder conditions.

It is an organic compound and dye, which is available as a dark orange/red powder and slightly soluble in water and alcohol. Which is mostly used as a fluorescent tracer for many applications. Fluorescein is a fluorophore commonly used in microscopy, in a type of dye laser as the gain medium, in forensics and serology to detect latent blood stains, and in dye tracing. The color of its aqueous solution varies from green to orange as a function of the way it is observed: by reflection or by transmission, as can be noticed in bubble levels, for example, in which fluorescein is added as a colorant to the alcohol filling the tube in order to increase the visibility of the air bubble contained within (thus enhancing the precision of the instrument). More concentrated solutions of fluorescein can even appear red.

### Requirements

**Chemicals:** as per specific quantity required.

Phthalic anhydride	= 5 gm
Resorcinol	= 7.5 gm
Anhydrous zinc chloride	= 2.3 gm
Concentrated hydrochloric acid	= 3.5 ml
Sodium hydroxide	= 10 mg

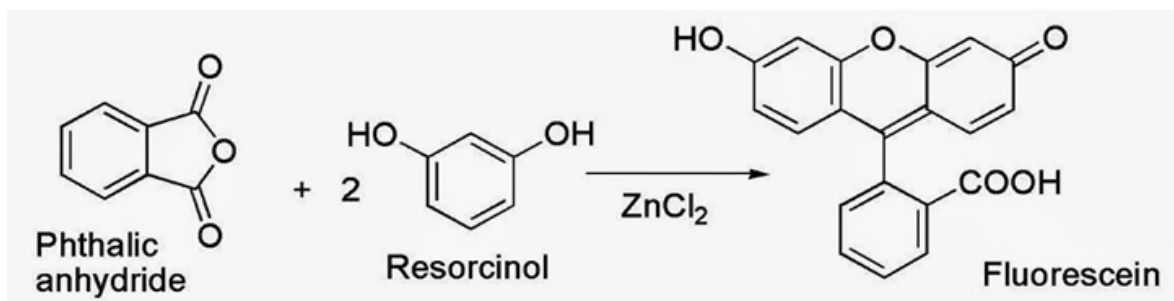


Distilled water = 100 ml

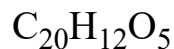
### Glassware/Apparatus

1. Erlenmeyer flask (250 mL)
2. Beaker
3. Stopper bottle
4. Thermometer
5. Pestle mortar
6. Pipette
7. Glass stirring rod
8. Buchner funnel
9. Suction pump
10. Filter papers
11. Measuring cylinder
12. Spatula
13. Water bath
14. Funnel

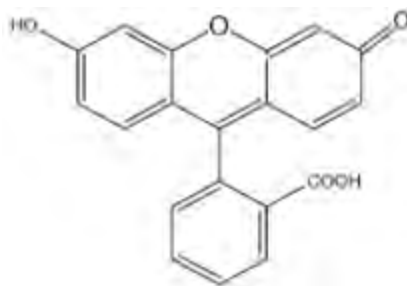
### Chemical Reaction



### Chemical Formula



### Chemical Structure



### IUPAC Name

3',6'-dihydroxyspiro[isobenzofuran-1(3H),9'-[9H] xanthen]-3-one

### Other Names

1. Fluorescein,
2. Resorcinol phthalein,
3. C.I. 45350,
4. Solvent yellow 94,
5. D & C yellow no. 7,
6. Angio Fluor,
7. Japan yellow 201,
8. Soap yellow

### Procedure

1. Grind 5 gm of phthalic anhydride and added 7.4 gm resorcinolin a mortar.
2. Then transfer the mixture to a 250 ml Erlenmeyer flask, immerse a thermometer and heat the flask slowly to 180°C on a sand bath.
3. Added slowly 2.3 g of anhydrous  $\text{ZnCl}_2$  in a small stopper bottle and add it to the reaction mixture in small lots stirring with the thermometer after each addition.
4. Continue heating the mixture till it becomes dark red and highly viscous.
5. Cool the flask to about 90°C and to this add 70 ml water and 3.5 ml conc. HCL.
6. Heat the reaction mixture till zinc salts have dissolved.
7. Filter the colored salt on a Buchner funnel and wash with water drain well.

8. Dried the crude product in an oven at 100°C.
9. Calculate the yield is 10.2 gm.

### Calculation

Here limiting reagent is phthalic anhydride; hence yield should be calculated.

Molecular formula of phthalic anhydride	= C <sub>8</sub> H <sub>4</sub> O <sub>3</sub>
Molecular formula of fluorescein Dye	= C <sub>20</sub> H <sub>12</sub> O <sub>5</sub>
Molecular weight of phthalic anhydride	= 148.1 g/mol
Molecular weight of fluorescein Dye	= 332.311 g/mol

### Theoretical Yield

148.1 g of phthalic anhydride reacts with acetic anhydride to give a 332.311 g of fluorescein Dye.

Therefore, 5 g of phthalic anhydride will give? (X)..... g of fluorescein Dye.

$$X = (332.311 \times 5) / 148.1 = 11.21 \text{ g}$$

Theoretical yield	= 11.21 gm.
Practical yield	= assume 10.2 gm.
Percentage practical yield	= (Practical yield / Theoretical yield) × 100
	= (10.2 / 11.21) × 100
	= 90.9 %

### Properties

1. It is a light solid which may use to fluorescence.
2. Slightly soluble in water and completely soluble in other organic solvent such as acetone, ethanol, diethyl ether.
3. The boiling point for this substance is 295.6°C.
4. Melting point is 314–316°C.

### Result

Fluorescence Dye was synthesized by the reaction of phthalic anhydride and resorcinol. The crude product is obtained, practical yield was found to be 10.2 gm and % yield to be 90.9%. The melting point of phenyl benzoate was found to be 314–316°C.

## Uses

1. Fluorescein drops being instilled for an eye examination.
2. Fluorescein sodium, the sodium salt of fluorescein, is used extensively as a diagnostic tool in the field of ophthalmology and optometry, where topical fluorescein is used in the diagnosis of corneal abrasions, corneal ulcers and herpetic corneal infections.
3. It is also used in rigid gas permeable contact lens fitting to evaluate the tear layer under the lens.
4. It is available as sterile single-use sachets containing lint-free paper applicators soaked in fluorescein sodium.
5. Intravenous or oral fluorescein is used in fluorescein angiography in research and to diagnose and categorize vascular disorders including retinal disease macular degeneration, diabetic retinopathy, inflammatory intraocular conditions, and intraocular tumors.
6. It is also being used increasingly during surgery for brain tumours.
7. Diluted fluorescein dye has been used to localize multiple muscular ventricular septal defects during open heart surgery and confirm the presence of any residual defects.

## Precautions

1. Wear gloves and goggles throughout this experiment.
2. When working with keep the safety rules with special care.
3. Use them only in the fume hood and be sure the hood fan is on! If you spill a lot of either of these, wash it off with lots of water and notify your instructor.

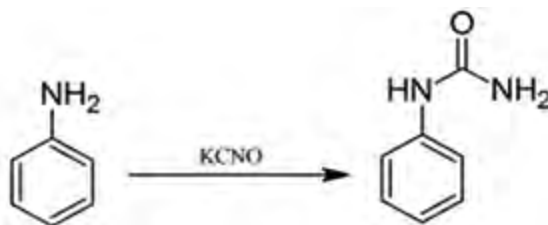


# Chapter 15

## Synthesis and Characterization of Phenyl Urea

### Principle

Phenyl urea is benzoyl urea derivative substituted with phenyl group on the opposite nitrogen atom.



### Requirements

**Chemicals:** as per specific quantity required.

Aniline hydrochloride	= 6.5 gm
Glacial acetic acid	= 4 ml
Ethyl acetate	= 5 gm
Concentrated hydrochloric acid	= 4 ml
Urea	= 12 gm
Distilled water	= 100 ml

### Glassware/Apparatus

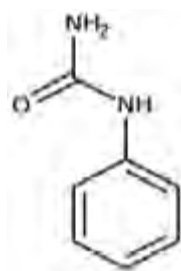
1. Erlenmeyer flask (250 mL)
2. Round bottom flask
3. Reflux condenser
4. Thermometer
5. Pestle mortar

6. Pipette
7. Water bath
8. Tripode stand
9. Funnel

### Chemical Formula



### Chemical Structure



### IUPAC Name

1. N-(Phenylcarbamoyl) benzamide
2. 1-Benzoyl-3-phenylurea

### Procedure

1. Dissolve 6.5 g of aniline hydrochloride and 12 gm of urea in 200 ml of water contained in a 1 litre round bottomed flask; filter the solution, if necessary.
2. Add 4 ml of conc. Hydrochloric acid and 4 ml of glacial acetic acid.
3. Fit a reflux condenser to the flask, introduce a few fragments of broken porcelain and boil the mixture for 30 minutes.
4. A fine white crystal appears after about 15 min. and gradually increase in amount as the refluxing is continued.
5. Cool the flask in ice and filter with suction.
6. Separate the mixture of phenyl urea and diphenyl urea by boiling with 500 ml of water and filter at the pump through a preheated Buchner funnel in to a warm flask; cool the filtrate, collect the phenyl urea, drain well and dry in steam oven.

7. The phenyl urea melts at 146–147°C and weighs 30 g (44%); recrystallization from hot water raise the m.p. to 148°C.
8. Recrystallization from glacial acetic acid or ethyl acetate with addition of a little decolorizing carbon gives a colorless product.

### Calculation

Here limiting reagent is aniline hydrochloride; hence yield should be calculated.

Molecular formal of aniline hydrochloride =  $C_6H_5N.HCl$

Molecular formal of phenyl Urea =  $C_7H_8N_2O$

Molecular weight of aniline hydrochloride = 129.59 g/mol

Molecular weight of phenyl urea = 136.151 g/mol

### Theoretical Yield

129.59 gm of aniline hydrochloride reacts with acetic anhydride to gives a 136.151 gm of phenyl Urea.

Therefore, 6.5 gm of aniline hydrochloride will give? (X).....gm of phenyl urea.

$$X = (136.151 \times 6.5) / 129.59 = 6.82 \text{ g}$$

Theoretical yield = 6.82 gm.

Practical yield = assume 5 gm.

Percentage = (Practical yield

practical yield  $\times 100$ )

$$= (5 \times 100) / 6.82$$

$$= 73.31\%$$

### Properties

1. It is a White to yellow crystal.
2. Slightly soluble in water and completely soluble in other organic solvent such as acetone, ethanol, diethyl ether.
3. The boiling point for this substance is 238°C.
4. Melting point is 145–147°C.

### Result

Phenyl urea was synthesized by the reaction of aniline hydrochloride with glacial acetic acid. The crude product is obtained, practical yield was found to be 5 gm and % yield to be 73.3 %. The melting point of phenyl urea was found to be 145–147°C.

### **Uses**

Phenyl Urea may be used as an herbicide for agriculture and agrochemicals preparation.

### **Precautions**

1. Wear gloves and goggles throughout this experiment.
2. When working with keep the safety rules with special care.
3. Use them only in the fume hood and be sure the hood fan is on! If you spill a lot of either of these, wash it off with lots of water and notify your instructor.

### **Questions**

1. What is Nature of phenyl urea?
2. What is the use of Phenyl urea?
3. Why aniline hydrochloride is used to the preparation of urea?
4. Define Solubility behavior of phenyl urea.





# Chapter 16

## Synthesis and Characterization of Benzanilide from Aniline by Acetylation Reaction

### Principle

Amine compounds on treatment with benzoyl chloride gives benzoyl derivatives, the reaction is known as **Schotten-Baumann Reaction**. The benzanilide is prepared by insertion of benzoyl moiety instead of an active hydrogen atom present in primary amino ( $\text{-NH}_2$ ) or secondary amino group ( $\text{-NH}$ ) is usually termed as benzoylation reaction.

This reaction essentially bears a close resemblance to the phenomenon of acetylation except that in specific instance the reagent (benzoyl chloride) which reacts in the present pyridine or 10% NaOH and not benzoic anhydride. The amines are more soluble in acid chloride than in NaOH, the reaction occurs preferably between benzoyl chloride and amine. In the preparation of benzanilide, NaOH neutralizes the liberated HCl and catalyzes the reaction. It is a base-catalyzed (aqueous sodium hydroxide or pyridine) reaction that is necessary to encourage an equilibrium shift towards the formation of amides. The base also neutralizes the hydrochloric acid, which is formed in the process, thereby preventing the further protonation of the amide product formed.

### Requirements

#### Chemicals

Aniline	= 2 ml
Benzoyl chloride	= 2.8 ml
Hot Ethanol	= 50 ml
10% sodium hydroxide	= 30 ml
Cold water	= 100 ml

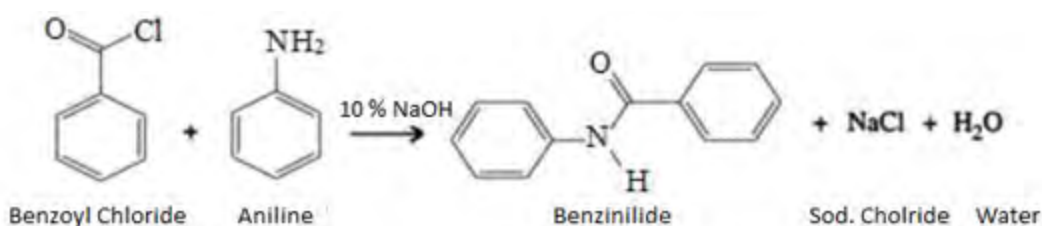
#### Glassware/Apparatus

1. Erlenmeyer flask (250 mL)

2. Volumetric flask
3. Pipette
4. Glass stirring rod
5. Buchner funnel
6. Suction pump
7. Filter papers
8. Measuring cylinder
9. Rubber clock
10. Fume hood

### Chemical Reaction

In this reaction benzoyl chloride is react with aniline in the presence of 10% sodium hydroxide solution. Benzoylation of compounds those are containing active hydrogen such as phenol, aniline, alcohol etc. form benzoyl chloride in the presence of aqueous NaOH (Schotten Baumann reaction).



### Procedure

1. Take a 2.0 mL (2.04 g) of aniline and 30 mL of 10 % sodium hydroxide solution in 250 mL Erlenmeyer flask with a good rubber cork and shake well at room temperature.
2. Sincerely add 2.8 mL (3.39 g) of benzoyl chloride (**using fume hood**), cork the flask and shake the mixture vigorously with intermittent pressure release (**perform under a fume hood**) for at least 15–20 minutes.
3. Reaction mixture is diluted with cold water, filter out the crude benzanilide with suction pump on a Buchner funnel, and wash with a coldwater/ice water.
4. The reaction mixture is a recrystallizing from hot alcohol in a at least 2 times.
5. The crude product of benzanilide is obtained.

6. Dried the product and calculate the practical yield about 3.2 gm.

### Calculation

The basic reagent is aniline; hence give a yield should be calculated from its amount taken.

Molecular formula of aniline	= $C_6H_7N$
Molecular formula of benzanilide	= $C_{13}H_{11}NO$
Molecular weight of aniline	= 93 g/mol
Molecular weight of benzanilide	= 197 g/mol

### Theoretical Yield as per specific quantity used in this reaction.

Here 93 g of aniline reacts with a benzoyl chloride to give a 197 g of benzanilide.

Therefore, 2.04 g of aniline will give?..... (X) g of crude product of benzanilide.

$$X = (197 \times 2.04)/93 = 4.32 \text{ g}$$

Theoretical yield	= 4.32 g
Practical yield	= assume 3.2 g
Percentage practical yield	$= (\text{Practical yield} \times 100)/\text{Theoretical yield}$ $= (3.2 \times 100)/4.32$ $= 74.07\%$

### Properties

1. White crystalline Solid.
2. Insoluble in water and soluble in 85% acetic acid and ethanol.
3. Boiling point is about 117°C.
4. Melting point is 163°C.

### Result

Benzanilide was synthesized by the reaction of aniline and benzoyl chloride and give the crude product. The practical yield was found to be 3.2 gm and

% yield about 74.07% and melting point of benzanilide was measured and found to be 163°C.

### **Uses of Benzimidazole**

Benzimidazole was used as amide model compound to study the reaction between the amide and epoxy. It is also used to study the influence of  $\beta$ -cyclodextrin on photo rearrangement of acetanilide, benzanilide and ethyl phenyl carbonate.

### **Safety and Precautions must be taken during Perform Practical**

1. Properly wear gloves and goggles throughout this experiment perform in a laboratory.
2. Aniline is more toxic and can be easily absorbed through the skin. Hence the mixing of aniline is done in a use in a fume cup hood/ Fuming chamber.
3. Benzoyl chloride is very toxic. It is lachrymatory and should be handled with care under the reaction the mixture in fume cup hood.
4. If any problems occur you spill a lot of either of these, wash it off with lots of water and notify your laboratory instructor or teacher.
5. In a reaction mixture freshly distilled aniline should be used to get a better result, or a small amount of zinc can be added in the reaction mixture.

### **Questions**

1. What is the purpose of the addition of NaOH to the benzylation of aniline with benzoyl chloride?
2. What is benzylation?
3. What are the advantages of benzylation over acetylation?

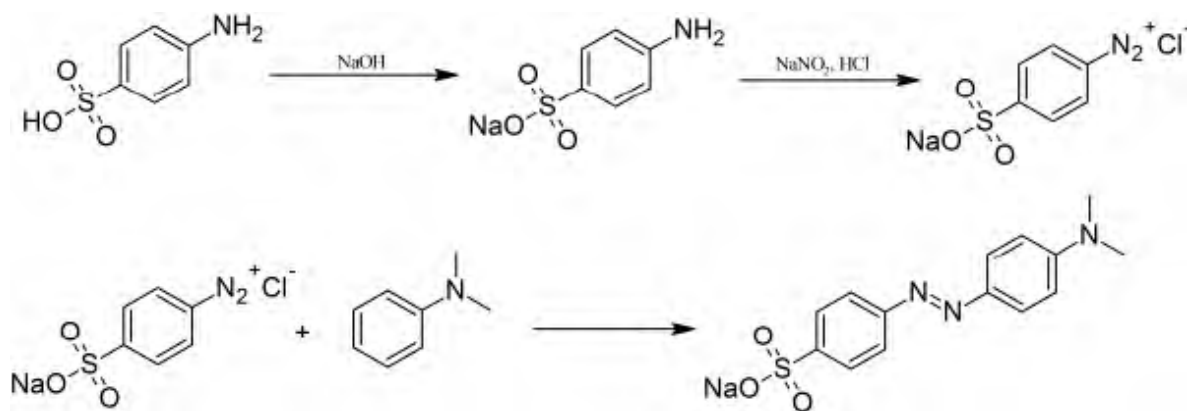


# Chapter 17

## Synthesize and Characterize Methyl Orange

### Principle

Methyl orange is a pH indicator mostly used in titration of different inorganic substance and limit test, because of its clear and distinct colour variance at different pH values. It shows red color in acidic medium and yellow colour in a basic medium. Because it changes color at the pH of a mid-strength acid, it is usually used in titration for acids. It is a universal indicator, methyl orange does not have a full spectrum of colour change, but it has a sharp end point. In a solution becoming less acidic, methyl orange changes from red to orange and, finally, to yellow with the reverse process occurring in a solution of increasing acidity.



### Requirements

**Chemicals:** as per specific quantity required.

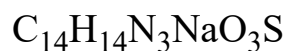
Sulfanilic acid	= 4.8 gm
Sodium carbonate	= 2 gm
Sodium nitrite	= 1.9 gm
Concentrated hydrochloric acid	= 5 ml
Dimethylaniline	= 3.2 ml

Glacial acetic acid	= 2 ml
Sodium chloride	= 25 gm
Ethanol	= 20 ml
Crushed ice	= q.s
Distilled water	= 100 ml

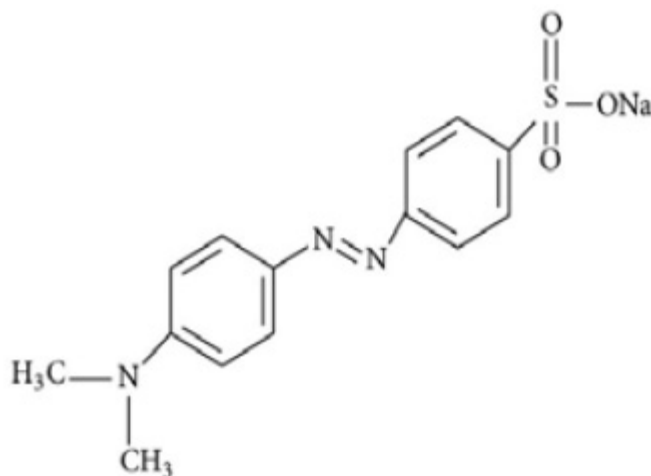
### Glassware/Apparatus

1. Erlenmeyer flask (250 mL)
2. Round bottom flask
3. Reflux condenser
4. Thermometer
5. Pestle mortar
6. Pipette
7. Water bath
8. Tripode stand
9. Funnel

### Chemical Formula



### Chemical Structure



### IUPAC Name

1. Sodium 4- {[4-(dimethyl amino) phenyl] diazenyl} benzene-1-sulfonate,
2. Sodium 4- [(4-dimethylamino) phenyl azo] benzenesulfonate.

## Procedure

1. Place 4.8 g of sulfanilic acid in 50 ml of Erlenmeyer flask.
2. Then added 2.5%  $\text{Na}_2\text{CO}_3$  in a reaction mixture.
3. Boil the reaction mixture in a using a water bath.
4. Cool the solution to  $15^\circ\text{C}$  and then added add 1.9 g  $\text{NaNO}_2$  and stir well.
5. Carefully pour the reaction mixture in a beaker containing 25 g of crushed ice and 5.0 ml conc.  $\text{HCl}$ .
6. White precipitate of the diazonium salt separates out.
7. In a second volumetric flask mix 3.2 ml of dimethylaniline and 2 ml glacial acetic acid to dissolve amines as its salt.
8. Continuous stirring to add this solution to the cold suspension of diazotized sulfanilic acid.
9. Coupling reaction occurs in the acid medium and the dye gives red color to the solution.
10. Then added 25 ml of 10%  $\text{NaOH}$  solution to produce an orange colored sodium salt.
11. The reaction mixture is stirring well with a using of glass rod and then added a 25 gm of  $\text{NaCl}$ .
12. Heat the reaction mixture to boiling, after cool in an ice-bath for 10 minutes.
13. The crude product of methyl orange is separates out as orange crystals.
14. Filter out the crude product with a use of 20 ml of ethanol
15. Dried the product by using a Buchner funnel.
16. Measure the practical yield about 8 gm.

## Calculation

Here limiting reagent is sulfanilic acid; hence yield should be calculated.

Molecular formal of sulfanilic acid =  $\text{C}_6\text{H}_7\text{NO}_3\text{S}$

Molecular formal of methyl orange =  $\text{C}_{14}\text{H}_{14}\text{N}_3\text{NaO}_3\text{S}$

Molecular weight of sulfanilic acid = 173.19 g/mol

Molecular weight of methyl orange = 327.33 g/mol

### Theoretical Yield

173.19 gm of sulfanilic acid reacts with acetic anhydride to give a 327.33 gm of methyl orange. Therefore, 4.8 gm of sulfanilic acid will give? (X) ..... gm of methyl orange.

$$X = (327.33 \times 4.8) / 173.19 = 9.07 \text{ gm}$$

Theoretical yield = 9.07 gm.

Practical yield = assume 8 gm.

Percentage = (Practical yield

practical yield  $\times 100$ )

$$= (8 \times 100) / 9.07$$

$$= 88.20\%$$

### Properties

1. It is an Orange solid
2. Soluble in water and insoluble in diethyl ether.
3. The boiling point for this substance is decomposes.
4. Melting point is 300°C.

### Result

Methyl Orange was synthesized by the reaction of sulfanilic acid with diethylamine. The crude product is obtained, practical yield was found to be 8 gm and % yield to be 88.20%. The melting point of phenyl benzoate was found to be 300°C.

### Uses

1. Methyl orange is widely used as a PH indicator in titrations for acids (same as that of phenolphthalein).
2. It gives colour change at mid strength of acids.

### Precautions

1. Wear gloves and goggles throughout this experiment.
2. When working with keep the safety rules with special care.



3. Use them only in the fume hood and be sure the hood fan is on! If you spill a lot of either of these, **wash it off with lots of water** and **notify your instructor**.



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## About The Book

This book having the "Practical Lab Manual of Pharmaceutical Organic Chemistry - I" as per PCI regulation, written with an intention to benefit the B. Pharma students for easy understanding.

In Pharmaceutical Chemistry it is very important to have sensitive, accurate, precise, and selective methods of synthesis and characterization for crude drugs. A sincere attempt has been made to prepare the content simple, concise, and easy to understand. The content of each experiment is further arranged conceptually and logically to maintain the flow of information.

The present handbook could be beneficial not only to the pharmacy students, but it could be helpful to other chemistry-related courses also. We hope that students and teachers of pharmacy as well as pharmaceutical chemistry synthesis in the pharmaceutical industry find this book very useful. Particularly this book includes PCI New Syllabus for B. Pharmacy (2nd Semester) students to carry out the experiments of Organic Chemistry - I.



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