|  |  |  |
| --- | --- | --- |
| **Sr. No.** | **Name of Experiment** | **Page No.** |
| 1. | To perform Limit Test for chloride |  |
| 2. | To perform Limit Test for sulphate |  |
| 3. | To perform Limit Test for Iron |  |
| 4. | To perform Limit Test for Heavy metals |  |
| 5. | To determine the melting point of given organic compound |  |
| 6. | To determine the boiling point of given organic compound |  |
| 7. | To perform identification and test for purity of Aspirin |  |
| 8. | To perform identification and test for purity of Caffeine |  |
| 9. | To perform identification and test for purity of Paracetamol |  |
| 10. | To perform identification and test for purity of Sulphanilamide |  |
| 11. | Determination of Acid Value |  |
| 12. | Determination of Saponification Value |  |
| 13. | Determination of Iodine Value |  |
| 14. | Preparation of Benzoic acid from Benzamide |  |
| 15. | Preparation of Picric acid from Phenol |  |
| 16. | Synthesis of Benzanilide from Aniline |  |
| 17. | Synthesis of phenyl benzoate from phenol |  |
| 18. | Synthesis of Acetanilide from Aniline |  |
| 19. | Synthesis of 2, 4, 6-Tribromoaniline from aniline |  |
| 20. | Synthesis of Para bromoacetanilide from acetanilide |  |
| 21. | Synthesis of 5-Nitrosalicylic acid from salicylic acid |  |
| 22. | Synthesis of Meta di nitro benzene from nitrobenzene |  |
| 23. | Synthesis of Nitrobenzene by Nitration Reaction |  |
| 24. | Synthesis of Benzoic acid from Benzyl chloride by Oxidation Reaction |  |
| 25. | Synthesis of Benzoic acid from Ethyl Benzoate by Base Hydrolysis |  |
| 26. | Synthesis of Salicylic Acid from Alkyl salicylate by Hydrolysis Reaction |  |
| 27. | Synthesis of 1-Phenylazo-2-naphthol from Aniline by Diazotisation and Coupling Reaction |  |
| 28. | Synthesis of Benzil from Benzoin |  |
| 29. | Synthesis of Dibenzalacetone from Benzaldehyde by Claisen Schmidt reaction |  |
| 30. | Synthesis of Cinnamic Acid from Benzaldehyde by Perkin Reaction |  |
| 31. | Synthesis of P-Iodo benzoic acid from P-Amino benzoic acid |  |
| 32. | Synthesis of Phenytoin from Benzil |  |
| 33. | Synthesis of Paracetamol from para amino phenol |  |
| 34. | Synthesis of Benzilic acid from Benzoin |  |
| 35. | Synthesis of 2 Phenylindole from Phenyl hydrazine |  |
| 36. | Synthesis of Benzocaine from p-nitro benzoic acid |  |
| 37. | Synthesis of 7-Hydroxy-4-Methyl Coumarin from Resorcinol |  |

**General Instructions**

The study of chemistry is an observation-based field. In theory papers you will learn principles and theories and in laboratory you will have opportunity to experience these principles and theories in laboratory. There are a few safety precautions to keep in mind when moving to the chemistry lab to complete the experiment.

**Kindly follow the general behaviour listed:**

* No food or beverages will be permitted inside the lab.
* Always read the upcoming experiments carefully and thoroughly, being used to understand all of the directions before entering the lab.
* Determine the potential hazards and appropriate safety precautions before beginning any work.
* Be in and ready, promptly when the lab begins.
* Do not utilize fume hoods for evaporations and disposal of volatile solvents.
* Always read the labels of the reagents and never use a reagent from an unlabelled bottle.
* Never smell a chemical straight out of the container.
* Use equipment only for its designated purpose.
* Never pour a waste chemical into drain or put in the garbage.
* Never pick broken glassware with your bare hands, regardless of the size of the piece. Please place all broken glassware in the appropriate broken glassware bucket.
* Wash your hand frequently during the lab and definitely wash your hand twice at the end of the lab.
* All equipment should be regularly inspected for wear or deterioration.
* Do not use mouth suction for pipetting or starting a siphon.
* Avoid adding solids to hot liquids.
* Never leave containers of chemicals open.
* Know emergency exit routes.
* Minimize all chemical exposures.

**Experiment No: 01 Aim: To perform Limit Test for chloride**

**Requirements**

**Apparatus:** Nessler’s cylinder, measuring cylinder, glass rod, stand, beaker, pipette, volumetric flask

**Chemicals:** Sodium chloride, dilute nitric acid, silver nitrate solution

**Principle:** The principle is based on the reaction between soluble chloride ions (Cl-) with silver nitrate (AgNO3) in the presence of dilute nitric acid. In which, reaction forms silver chloride (AgCl) which produce as solid particles or precipitate (off white flakes). This silver chloride (AgCl) precipitates produces opalescence in solution. The opalescence produced in sample solution is compared with standard. If the opalescence in the sample is less than the standard, it passes the test. If it is more than the standard it fails the test.

**Reaction**



**Preparation of chloride standard solution (25ppm)**

Prepare a 0.0824% w/v solution of sodium chloride (NaCl). Take 5ml of 0.0824% w/v solution of sodium chloride and dilute it upto 100ml with water. It gives standard chloride solution of 25ppm.

**Procedure**

|  |  |  |
| --- | --- | --- |
| **Sr. No.** | **Test or sample solution** | **Standard solution** |
| 1 | Specific quantity of test substance dissolve in water or solution is prepared as directed in monograph and take it into 50ml Nessler’s cylinder. | Place 1ml chloride standard solution and 5ml of water in 50ml Nessler’s cylinder. |
| 2 | Add 10ml of dil. Nitric acid. | Add 10ml of dil. Nitric acid. |
| 3 | Dilute upto 49ml with water. | Dilute upto 49ml with water. |
| 4 | Add 1ml of silver nitrate (AgNO3) solution. | Add 1ml of silver nitrate (AgNO3) solution. |
| 5 | Stir immediately with glass rod. Keep aside for 5 minutes. | Stir immediately with glass rod. Keep aside for 5 minutes. |
| 6 | Observe the opalescence and compare with standard solution. | Observe the opalescence and compare with test/sample solution. |

**Observation:** If the opalescence produces in the sample solution is less than the standard solution, the sample will pass the limit test for chloride. If the opalescence produces in the sample solution is more than the standard solution, the sample will fail the limit test for chloride.

**Result:** The opalescence of both solutions is compared. The given compound passed/did not pass the limit test for chloride.

**Important Note:** 1. Nitric acid is added in the test to make solution acidic and helps silver chloride to precipitate and produces opalescence.

2. Nitric acid also dissolves impurities like sulphates, carbonates etc.

**Viva voce Questions**

1. What is limit test?

2. What is principle and reaction of limit test for chloride?

3. Write down reaction involved in the limit test for chloride.

4. Name the apparatus in which test and standard solution are compared?

**Experiment No: 02 Aim: To perform Limit Test for sulphate**

**Requirements**

**Apparatus:** Nessler’s cylinder, measuring cylinder, glass rod, stand, beaker, pipette, volumetric flask

**Chemicals:** Barium sulphate, ethanol, barium chloride,

**Principle:** The principle is based on the reaction of soluble sulphate ions (SO4-) with barium chloride (BaCl2) in presence of dilute hydrochloric acid (HCl) or acetic acid (CH3COOH). Which forms barium sulphate (BaSO4) appears as solid particles or precipitates (off white flakes). Barium sulphate (BaSO4) precipitates produces turbidity in the sample solution which is compared with the standard solution. If the turbidity in the sample is less than the standard, it passes the test. If it is more than the standard it fails the test.

**Reaction**



**Preparation of Standard Potassium Sulphate Solution**

Weigh accurately 0.1089gm of potassium sulphate was taken and the volume was made upto 100ml with water.

**Preparation of Barium Sulphate Reagent**

Dissolve 12gm of barium chloride in 1000ml water to make 0.05M barium chloride solution. To 15ml of the prepared solution add 55ml of water, 20ml alcohol, 5ml of 0.0181%w/v potassium sulphate solution and makeup the volume upto 100ml.

**Procedure**

|  |  |  |
| --- | --- | --- |
| **Sr. No.** | **Test or sample solution** | **Standard solution** |
| 1 | Specific quantity of test substance dissolve in water or solution is prepared as directed in monograph and take it into 50ml Nessler’s cylinder. | Place 1ml of 0.1089% w/v solution of potassium sulphate in 50ml Nessler’s cylinder. |
| 2 | Add 2ml of dil. HCl. | Add 2ml of dil. HCl. |
| 3 | Dilute to 45ml with water and add 5ml of barium sulphate reagent. | Dilute to 45ml with water and add 5ml of barium sulphate reagent. |
| 4 | Stir immediately and allow to stand for 5 minutes. | Stir immediately and allow to stand for 5 minutes. |
| 5 | Observe the turbidity developed and compare with that of the standard. | Observe the turbidity developed and compare with that of the test. |

**Observation:** If the turbidity produces in the sample solution is less than the standard solution, the sample will pass the limit test for sulphate. If the turbidity produces in the sample solution is more than the standard solution, the sample will fail the limit test for sulphate.

**Result:** The turbidity of both solutions is compared. The given compound passed/did not pass the limit test for sulphate.

**Important Note:** 1. Acetic acid helps to make solution acidic, helps barium sulphate (BaSO4)to precipitate and produces turbidity in the solution.

2. Potassium sulphate is used to increase the sensitivity of the test by giving ionic concentration in the reagent.

3. Alcohol helps to prevent super saturation.

**Viva voce Questions**

1. What is limit test?

2. What is principle of limit test for sulphate?

3. Write down reaction involved in the limit test for sulphate.

4. Name the apparatus in which test and standard solution are compared?

5. Why do we add ethanol in sulphate limit test?

**Experiment No: 03 Aim: To perform Limit Test for Iron**

**Requirements**

**Apparatus:** Nessler’s cylinder, measuring cylinder, glass rod, stand, beaker, pipette, volumetric flask

**Chemicals:** 20%w/v iron free citric acid, thioglycolic acid, iron free ammonia solution, iron standard solution, ferric ammonium sulphate

**Principle:** The principle is based on the reaction of ferrous ions (Fe++) in ammonical solution with thioglycolic acid in the presence of citric acid, which forms iron thioglycolate complex which is pale pink to deep reddish purple in colour. The intensity of colour produced in sample solution is compared with the colour produced in standard solution.

**Reaction**



**Preparation of Iron standard solution (20 ppm Fe)**

Dilute 1ml of 0.1726 percent w/v solution of ferric ammonium sulphate in 0.05M sulphuric acid to 10ml with

**Procedure**

|  |  |  |
| --- | --- | --- |
| **Sr. No.** | **Test or sample solution** | **Standard solution** |
| 1 | Specific quantity of test substance dissolve in water or solution is prepared as directed in monograph and take it into 50ml Nessler’s cylinder. | Place 2ml iron standard solution in 50ml Nessler’s cylinder. |
| 2 | Add 2ml of 20% w/v citric acid solution. | Add 2ml of 20% w/v citric acid solution. |
| 3 | Add 0.1ml of thioglycolic acid and mix it. | Add 0.1ml of thioglycolic acid and mix it. |
| 4 | Add ammonia to the solution to make alkaline. | Add ammonia to the solution to make alkaline. |
| 5 | Dilute to 50ml with water and stir immediately with glass rod, allow to stand for 5 minutes. | Dilute to 50ml with water and stir immediately with glass rod, allow to stand for 5 minutes. |
| 6 | Observe the colour produced and compare with the standard solution. | Observe the colour produced and compare with the test/sample solution. |

**Observation:** If the intensity of colour produces in the sample solution is less than the standard solution, the sample will pass the limit test for iron. If the intensity of colour produces in the sample solution is more than the standard solution, the sample will fail the limit test for iron.

**Result:** The intensity of colour of both solutions is compared. The given compound passed/did not pass the limit test for iron.

**Important Note:** 1. Citric acid forms soluble or unstable complex with iron so prevents its precipitation by ammonia.

2. In alkaline solution ferric iron (III) oxidizes thioglycolate to dithioglycolate with the formation of iron (II). So, thioglycolic acid helps to reduces ferric ions (Fe+++) to ferrous ions (Fe++).

**Viva voce Questions**

1. What is limit test?

2. What is principle of limit test for iron?

3. Write down reaction involved in the limit test for iron.

4. Name the apparatus in which test and standard solution are compared?

5. Name the complex due to which solution becomes pink to purple coloured.

**Experiment No: 04 Aim: To perform Limit Test for Heavy metals**

**Requirements**

**Apparatus:** Nessler’s cylinder, measuring cylinder, glass rod, stand, beaker, pipette, volumetric flask

**Chemicals:** Dilute acetic acid, lead nitrate, dilute ammonia solution, hydrogen sulphide solution

**Principle:** The principle is based on the reaction between the heavy metal solution and a saturated solution of hydrogen sulphide. In the acidic medium it produces reddish/brown colour with hydrogen sulphide which is compared with standard solution.

**Reaction**



**Preparation of Standard Lead Solution**

10ml of the lead nitrate stock solution diluted to 100ml with water (20ppm of lead).

**Preparation of Lead Nitrate Stock Solution**

Dissolve 0.1598gm of lead nitrate in 100ml of water add 1ml of conc. nitric acid and dilute to 1000ml with water.

**Procedure**

|  |  |  |
| --- | --- | --- |
| **Sr. No.** | **Test or sample solution** | **Standard solution** |
| 1 | Specific quantity of test substance dissolve in 25ml water or solution is prepared as directed in monograph and take it into 50ml Nessler’s cylinder. | Place 2ml lead standard solution in 25ml of water in 50ml Nessler’s cylinder. |
| 2 | PH is adjusted between 3-4 by dilute acetic acid/ammonia solution. | PH is adjusted between 3-4 by dilute acetic acid/ammonia solution. |
| 3 | Dilute upto 35ml with water. | Dilute upto 35ml with water. |
| 4 | Add freshly prepared 10ml of hydrogen sulphide solution. | Add freshly prepared 10ml of hydrogen sulphide solution. |
| 5 | Dilute upto 50ml with water. Allow to stand for 5 minutes. | Dilute upto 50ml with water. Allow to stand for 5 minutes. |
| 6 | Observe intensity of colour produces and compare with the standard. | Observe intensity of colour produces and compare with the test. |

**Observation:** If the intensity of colour produces in the sample solution is less than the standard solution, the sample will pass the limit test for heavy metals. If the intensity of colour produces in the sample solution is more than the standard solution, the sample will fail the limit test for heavy metals.

**Result:** The intensity of colour of both solutions is compared. The given compound passed/did not pass the limit test for heavy metals.

**Viva Voce questions**

1. What is limit test?

2. What is principle of limit test for heavy metals?

3. Write down reaction involved in the limit test for heavy metals.

4. How do we prepare lead standard solution?

**Experiment No: 05 Aim: To determine the melting point of given organic compound**

**Requirements**

**Apparatus:** Capillary tube, Paraffin, Thermometer

**Chemicals:** Given organic compound

**Procedure**

1. Take a fine capillary of length 5-6cm. seal its one end by inserting the end of the capillary tube horizontally into the extreme edge of a small steady Bunsen flame for a few seconds, rotating the capillary meanwhile.

2. Take a small quantity of the compound whose melting point is to be determined on a porous plate and powder it with a spatula.

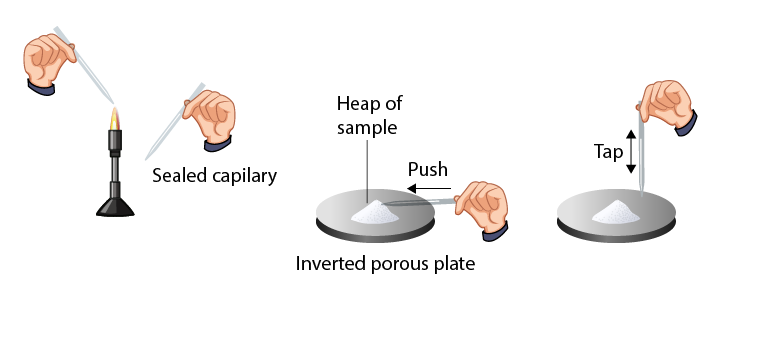
3. Introduce the powdered compound in the capillary tube by introducing the open end of the capillary tube into the powdered compound and gently rotating it. Gently tap the capillary tube against the porous plate so that the compound sinks into the closed end. Repeat the procedure of introducing and tapping three to four times.

4. Moisten the bulb of thermometer with Conc. Sulphuric acid or liquid paraffin and attach the capillary to the lower end of the thermometer.

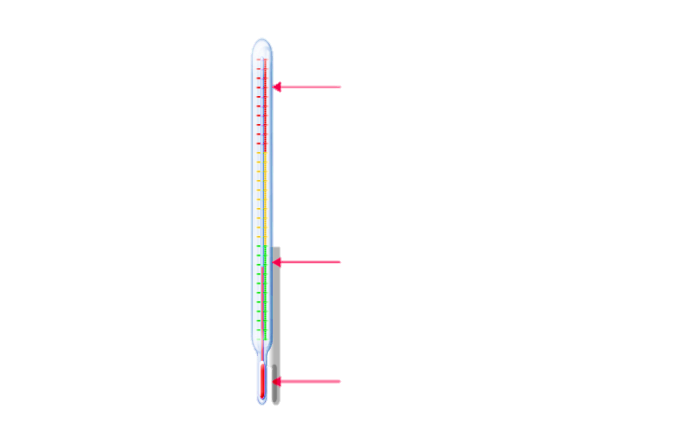
5. Place the thermometer with the capillary tube in the melting point apparatus containing at least two third of its volume liquid paraffin in such a way that the closed end of the capillary remains below the surface of Liquid paraffin.

6. Now heat the beaker gently and note down the temperature from time to time and finally note down the temperature at which the compound starts melting and completely melts.

7. Repeat the experiment with a new capillary tube and fresh quantity of the substance.



**Figure 1: Apparatus showing melting point determination**



Substance

Capillary

Sealed Capillary

**Figure 2: With the help of a thread, attach the capillary tube to a thermometer as shown in the given above figure**

**Observations**

**Melting Point**

1 ……………………0C

2 ……………………0C

3……………………0C

**Viva Voce questions**

1. What is melting point?

**Experiment No: 06 Aim: To determine the boiling point of given organic compound**

**Requirements**

**Apparatus:** Capillary tube, Paraffin, Thermometer

**Chemicals:** Given organic compound

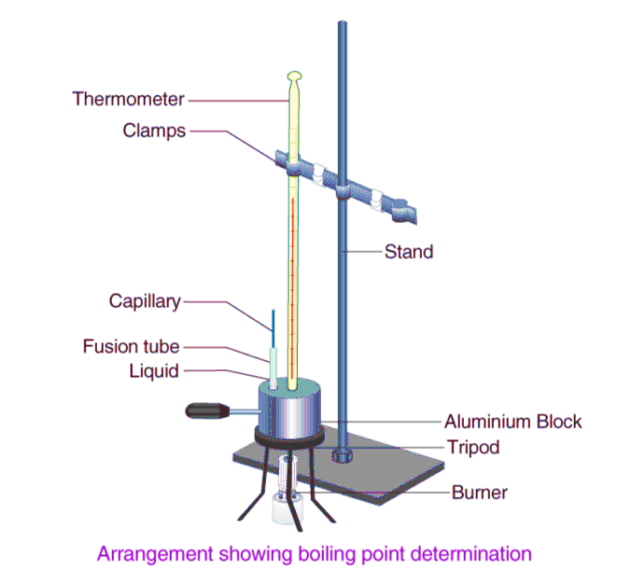
**Procedure**

1. Take a small amount of liquid in an ignition tube and place a capillary tube sealed at one of its end in an inverted position in the same ignition tube.

2. Attach the ignition tube with the thermometer by means of a rubber band in such a way that the lowers end of the ignition tube always remains in contact with the bulb of the thermometer.

3. Introduce whole of the arrangement into the beaker or boiling tube containing liquid paraffin in such a way that bulb of the thermometer dips in the liquid.

4. Heat the beaker gently with constant stirring until a stream of bubble gas goes outside the capillary tube rapidly. Note down the temperature.



**Figure 1: Apparatus showing Boiling Point Determination**

**Observations**

**Boiling Point**

1 ……………………0C

2 ……………………0C

3……………………0C

**Viva Voce questions**

1. What is boiling point?

**Experiment No: 07 Aim: To perform identification and test for purity of Aspirin**

**Requirements**

**Apparatus:** Test tube, measuring cylinder, glass rod, beaker, pipette, Buchner funnel

**Chemicals:** Sodium hydroxide, sulphuric acid, ferric chloride solution, methyl alcohol

**Theory**

**Chemical Structure**



Aspirin

**IUPAC Name:** 2-acetyloxybenzoic acid

**Other Name:** Acetylsalicylic acid

**Molecular Formula:** C9H8O4

**Molecular Weight:** 180gm/mol

**Appearance:** colourless to white crystalline solid

**Melting point:** 133-1350C

**Solubility:** water, ether, ethanol, and chloroform

**Procedure**

|  |  |  |  |
| --- | --- | --- | --- |
| **Sr. No.** | **Test** | **Observation** | **Inference** |
| 1. | Boil about 0.5gm of given sample with 10ml of 10%sodium hydroxide solution cool it, and add 10ml of dilute sulphuric acid. A white crystalline precipitate is produced and the odour of acetic acid is perceptible. Filter it, dissolve the precipitate in water and to this solution add ferric chloride solution. | Deep violet colour appears | Aspirin Present or Absent |
| 2. | Boil about 0.5gm of given sample with 10ml of 10%sodium hydroxide solution cool it, and add 10ml of dilute sulphuric acid. A white crystalline precipitate is produced and the odour of acetic acid is perceptible. Filter it, to the filtrate add 3ml of alcohol and 3ml of sulphuric acid and warm. | Odour of ethyl acetate is perceptible | Aspirin Present or Absent |
| 3. | Melting point | 133-1350C | Melting point of aspirin ………0C |

**Result**

The given sample is found to be of……………..with melting point…………….0C

**Uses**

1. It is a commonly used for the treatment of pain and fever due to various causes.

2. Acetylsalicylic acid has both anti-inflammatory and antipyretic effects.

**Cautions**

1. The synthesis should always be done under a working fume hood.

2. Avoid contact with your eyes, skin, and clothing. In case of contact, rinse with plenty of water.

3. The aspirin synthesized by unlicensed individuals (such as with lab experiments only) should not be ingested.

**Viva Voce Questions**

1. What are the uses of aspirin?

2. Give the IUPAC name of aspirin?

3. Give physical properties of aspirin.

**Experiment No: 08 Aim: To perform identification and test for purity of Caffeine**

**Requirements**

**Apparatus:** Test tube, measuring cylinder, glass rod, beaker, pipette, Buchner funnel, porcelain dish

**Chemicals:** Hydrochloric acid, potassium chlorate, ammonia, tannic acid, iodine

**Theory**

**Chemical Structure**



Caffeine

**IUPAC Name:** 1, 3, 7-Trimethylpurine-2, 6-dione

**Other Name:** Coffeine, trimethylxanthine

**Molecular Formula:** C8H10N4O2

**Molecular Weight:** 194gm/mol

**Appearance:** white powder

**Melting point:** 236-2380C

**Solubility:** water, chloroform

**Procedure**

|  |  |  |  |
| --- | --- | --- | --- |
| **Sr. No.** | **Test** | **Observation** | **Inference** |
| 1. | Add 10mg given sample, 1ml HCl, and 0.1gm of potassium chlorate in test tube. Evaporate on water bath. Expose the residue to dil. ammonia solution. | Purple colour appears | Caffeine Present or Absent |
| 2. | Add saturated solution of given sample, few drops of tannic acid in test tube. | White precipitate | Caffeine Present or Absent |
| 3. | Add 4 ml of saturated solution of given sample, 1.5ml of iodine and few drops of dilute hydrochloric acid in test tube. | Brown precipitate | Caffeine Present or Absent |
| 4. | Melting point | 236-2380C | Melting point of caffeine ………0C |

**Result**

The given sample is found to be of……………..with melting point…………….0C

**Uses**

1. Caffeine is used to treat tiredness and drowsiness, and to improve the effect of some pain relievers.

**Cautions**

1. Avoid Skin and Eye Contact.

2. Wearing protective gloves, lab coats, and goggles

**Viva Voce Questions**

1. What is the IUPAC name of caffeine?

2. Give one test for identification of caffeine.

3. Give the physical properties of caffeine.

**Experiment No: 09 Aim: To perform identification and test for purity of Paracetamol**

**Requirements**

**Apparatus:** Test tube, measuring cylinder, glass rod, beaker, pipette, Buchner funnel, porcelain dish, burner

**Chemicals:** Ferric chloride, pyridine, 4-nitrobenzyl chloride, conc. HCl, potassium dichromate

**Theory**

**Chemical Structure**



**IUPAC Name:** N-(4-hydroxyphenyl) acetamide

**Other Names:** Acetaminophen

**Molecular Formula:** C8H9NO2

**Molecular Weight:** 151 g/mol

**Appearance:** Colourless crystals or crystalline powder

**Melting Point:** 169 to 1710C

**Solubility:** Ethanol, water, acetone, chloroform

**Procedure**

|  |  |  |  |
| --- | --- | --- | --- |
| **Sr. No.** | **Test** | **Observation** | **Inference** |
| 1. | Add 0.1gm of given sample, 10ml distilled water, few ml of ferric chloride solution in test tube. | Violet colour is produced | Paracetamol Present or Absent |
| 2. | Add in test tube 0.25gm of given sample, 4ml pyridine, 0.5gm of 4-nitrobenzyl chloride. Boil the solution and then cool. Add mixture to 40ml of water and stir it. | Precipitate forms. | Paracetamol Present or Absent |
| 3. | Add in test tube 0.1gm of given sample, 1ml conc. HCl. Boil the solution. No precipitate formed. Then add 10ml water and cool. Then add few ml of potassium dichromate solution. | Violet colour is produced. | Paracetamol Present or Absent |
| 4. | Melting point | 169 to 1710C | Melting point of paracetamol ………0C |

**Result**

The given sample is found to be of……………..with melting point…………….0C

**Viva Voce Questions**

1. Give the uses of paracetamol.

2. Give the physical properties of paracetamol.

3. Write IUPAC name of paracetamol.

**Experiment No: 10 Aim: To perform identification and test for purity of Sulphanilamide**

**Requirements**

**Apparatus:** Test tube, measuring cylinder, glass rod, beaker, pipette, Buchner funnel, porcelain dish, burner

**Chemicals:** Dilute hydrochloric acid, β Naphthol solution, sodium acetate, ethanol, sulphuric acid, acetic acid

**Theory**

**Chemical Structure**



Sulphanilamide

**IUPAC Name:** 4-aminobenzene sulfonamide

**Molecular Formula:** C6H8N2O2S

**Molecular Weight:** 172 g/mol

**Appearance:** yellowish-white or white crystal or fine powder

**Melting Point:** 163 to 1650C

**Solubility:** Acetone

**Procedure**

|  |  |  |  |
| --- | --- | --- | --- |
| **Sr. No.** | **Test** | **Observation** | **Inference** |
| 1. | Add 50mg given sample in test tube, 2ml dilute hydrochloric acid, 2ml β Naphthol solution, 1gm sodium acetate. | Orange precipitate | Sulphanilamide Present or Absent. |
| 2. | Add 1gm of given sample, 5ml ethanol, 1ml sulphuric acid. | Ethyl acetate odour. | Sulphanilamide Present or Absent. |
| 3. | Add 50mg of given sample, heat for few minutes. | Oily liquid with acetamide odour. | Sulphanilamide Present or Absent. |
| 4. | Melting point | 163 to 1650C | Melting point of sulphanilamide ………0C |

**Result**

The given sample is found to be of……………..with melting point…………….0C

**Uses**

1. It is used to treat vulvovaginal candidiasis caused by Candida albicans.

2. Used to treat bacterial infections and some fungal infections.

3. They are widely used to treat different microbial infections.

**Cautions**

1. Avoid Skin and Eye Contact.

2. Wearing protective gloves, lab coats, and goggles.

**Viva Voce Questions**

1. What are the uses of sulphanilamide?

2. Give the physical properties of sulphanilamide.

3. Give one test for identification of sulphanilamide.

**Experiment No: 11 Aim: Determination of Acid Value**

**Definition:** The acid value is the number of milligrams of potassium hydroxide required to neutralize the free fatty acids present in one gram of substance.

**Significance:**

1. This value indicates the degree of rancidity of the given fat sample. High acid values observed in rancified oils.

2. The decomposition of fats and other lipids by hydrolysis or oxidation is the rancidification process.

3. This rancidification can be reduced but not completely eliminated by storing fats and oils at low temperature.

**Principle:** The acid value is determined by directly titrating the oil/fat in an alcoholic medium against standard potassium hydroxide/sodium hydroxide solution.

**Chemical reaction:** The acid value can be determined by the amount of free fatty acids in oil by reacting the carboxylic group proton (COOH) with 3 moles of sodium hydroxide/potassium hydroxide to give a glycerol and 3 moles of soap in the presence of ethanol.



**Analytical Importance:** The value is a measure of the amount of fatty acids which have been liberated by hydrolysis from the glycerides due to the action of moisture, temperature or enzyme lipase.

**Requirements:**

**Apparatus:** Iodine flask, reflux condenser, burette, pipette, beaker, glass rod

**Chemicals:** Fixed oil, potassium hydroxide (0.1N potassium hydroxide solution), ether, ethanol phenolphthalein solution

**Procedure:**

1. Weigh about 10 gm of the substance being examined, add in an iodine flask.

2. Prepare 50 ml of equal volume of mixture of 95 percent of ethanol and ether, add 0.5ml of phenolphthalein solution and titrate against 0.1N potassium hydroxide solution to neutralize it.

3. Dissolved weighed quantity of substance in above neutralized solution if sample does not dissolve in solvent, attached the round bottom flask (RBF) with a reflux condenser and warm slowly, with frequent shaking, until the sample dissolves.

4. Add 1ml of phenolphthalein solution and titrate with 0.1N potassium hydroxide solution remains faintly pink after shaking for 30 seconds.

5. Calculate the acid value from the following equation,

Volume of 0.1N KOH used in ml

Acid Value = 5.6

Weight of the sample taken in gm

**Result:** The acid value of the given sample was found to be…………

**Uses:**

* 1. It is used as a determination of acid value of oils/fats, which may help to quality foods are serve to human being.

**Questions:**

1. What is the principle and reaction involved in acid value?

2. What is acid value?

**Experiment No: 12 Aim: Determination of Saponification Value**

**Definition:** The saponification value is the number of mg of potassium hydroxide required to saponified 1gram of oil/fat.

**Significance:**

1. It is used primarily for an identification to detect adulteration with unsaponifiable matter.

2. It is also used to distinguish between fatty oils and mineral oils.

3. It is also indicated to measure of molecular weight of fatty oils.

**Principle:** The oil sample is saponified by refluxing with a known excess of alcoholic potassium hydroxide solution. The alkali required for saponification is determined by titration of the excess potassium hydroxide with standard hydrochloric acid.

**Chemical Reaction:**



**Analytical importance:** The saponification value is an index of mean molecular weight of the fatty acids of glycerides comprising a fat. Lower the saponification value, larger the molecular weight of fatty acids in the glycerides and vice versa.

**Requirements:**

**Apparatus:** Iodine flask, reflux condenser, burette, pipette, beaker, glass rod

**Chemicals:** Fixed oil, 0.5M ethanolic potassium hydroxide solution, 0.5M hydrochloric acid solution, phenolphthalein solution

**Procedure:**

1. Weigh about 2 gm of the substance being examined add in iodine flask with reflux condenser.

2. Add 25 ml of 0.5M ethanolic potassium hydroxide solution and attached a reflux condenser on water bath for 30 minutes.

3. Remove the condenser, add 1ml of phenolphthalein solution and titrate immediately with 0.5M hydrochloric acid solution. Note the reading as ‘a’.

4. Repeat the procedure without the substance being examined. Note the reading as ‘b’

5. Calculate the saponification value from the following equation,

(b-a)

Saponification value = 28.05

W

Where, W= weight of substance in gm

**Result:** The saponification value of the given sample was found to be……..

**Uses:**

1. Soaps are essential to cleanse dirt and oil off the objects including skin surface. Soaps are widely used in bathing, cleaning, washing and in other household chores. Soaps are an integral part to maintain good health and hygiene of the individuals.

2. To extinguish cooking oils and fats used saponification reaction.

3. By using different types of alkali in the process the type of reaction product can be altered between hard and soft soaps. a. Using KOH: We can obtain soft soaps.

b. Using NaOH: We can obtain hard soaps.

**Questions:**

1. What is saponification value?

2. What are the significance involved in determination of saponification value?

3. What is the principle of saponification value?

**Experiment No: 13 Aim: Determination of Iodine Value**

**Definition:** The iodine value of an oil/fat is the number of grams of iodine absorbed by 100gm of the oil/fat.

**Significance:**

1. It gives idea about the proportion of unsaturated fatty acids present in free and combined esters.

2. It also used to indicate the composition of complex mixture as well as pure substances.

**Principle:** The oil/fat sample taken in carbon tetrachloride is treated with a known excess of iodine monochloride solution in glacial acetic acid. The excess of iodine monochloride is treated with potassium iodide and the liberated iodine estimated by titration with sodium thiosulfate solution.

**Analytical importance:** The iodine is a measure of the amount of unsaturation (number of double bonds) in a fat.

**Requirements:**

**Apparatus:** Iodine flask, reflux condenser, burette, pipette, beaker, glass rod

**Chemicals:** Fixed oil, carbon tetrachloride, iodine monochloride solution, potassium iodide solution, 0.1M sodium thiosulphate solution, starch solution

**Pre-experiment preparation:**

**1. Preparation of iodine monochloride solution:** Dissolve 8 gm of trichloride in 200 ml glacial acetic acid and separately dissolve 9 gm of iodine in 300 ml of dichloromethane. Mix the two solutions and dilute upto 1000 ml with glacial acetic acid.

**2. Potassium iodide solution:** 16.6 gm of potassium iodide dissolved in distilled water to produce 100 ml.

**3. 0.1M sodium thiosulphate solution:** Dissolve 24.8 gm of sodium thiosulphate, 0.2 gm of sodium carbonate in distilled water to produce 1000 ml.

**Procedure:**

1. Weigh 0.25 gm of substance being examined add in iodine flask.

2. To it add 10 ml carbon tetrachloride and dissolve it properly.

3. Add 20 ml of iodine monochloride solution, securely cork the stopper and allow to stand in dark for about 30 minutes with occasional shaking.

4. Add 15 ml of potassium iodide solution and 100 ml of distilled water.

5. Titrate with 0.1M sodium thiosulphate solution using starch as an indicator.

6. Note the reading as ‘a’.

7. Repeat the procedure without the substance being examined and record the reading as ‘b’

8. Calculate the iodine value from the following equation,

(b-a)

Iodine value = 1.269

W

Where, W= weight of substance in gm

**Result:** The iodine value of the given sample was found to be……..

**Uses:**

1. The iodine value in chemistry is the mass of iodine in grams that is consumed by 100 grams of a chemical substance.

2. Iodine numbers are often used to determine the amount of unsaturation in fatty acids.

**Questions:**

1. What are the analytical importance of iodine value?

2. Define iodine value.

3. What is the principle involved in the determination of iodine value.

4. What are the uses of iodine value?

**Experiment No: 14 Aim: Preparation of Benzoic acid from Benzamide**

**Requirements**

**Apparatus:** Round Bottom flask (RBF), measuring cylinder, glass rod, beaker, pipette, Buchner funnel

**Chemicals:** Benzamide, 10% sodium hydroxide solution, conc. HCl

**Principle:** The principle is based upon the presence of an acid like hydrochloric acid, the hydrolysis of benzamide with sodium hydroxide (NaOH) forms benzoic acid. Benzamide is first converted to sodium benzoate which is further acidified to benzoic acid.

**Reaction**



**Mechanism**



**Structure**



**IUPAC Name:** Benzoic acid

**Other Name:** Benzene carboxylic acid

**Molecular Formula:** C7H6O2

**Molecular Weight:** 122gm/mol

**Appearance:** White crystalline solid

**Melting point:** 120-1220C

**Solubility:** Alcohol, ether, benzene

**Procedure**

1. Add 5g of Benzamide and 75ml of 10% [sodium hydroxide solution](https://pharmacyinfoline.com/sodiumthiosulfate-standard-solution/) in 250 ml round bottom flask fitted with a reflux condenser. Add few pieces of porcelain into the reaction mixture.

2. Reaction mixture heat on a water bath for 30 minutes.

3. Cool the solution in ice cold water and then slowly add conc. Hydrochloric acid till the mixture is strongly acidic. A white product separates out. Cool the mixture in ice cold water and collect, filter the product at Buchner funnel at pump. Wash with cold water and drain.

4. Recrystallize by dissolving the product in minimum quantity of boiling water, filter the hot solution if necessary.

5. Report the percentage yield and melting point.

**Calculations:**

Molecular formula of Benzamide = C7H7NO

Molecular weight of Benzamide = 121 g/mole

Molecular formula of Benzoic acid = C7H6O2

Molecular weight of Benzoic acid = 122 g/mole

121 g of Benzamide is equivalent to 122 g Benzoic acid  
5 g of Benzamide is equivalent to X g Benzoic acid

X= (122 × 5)/121 = 5.041 gm

Theoretical Yield = 5.041 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/ 5.041 gm

= ……………….%

**Result:**

Benzoic acid was synthesized from Benzamide and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. Benzoic acid used to produce a wide range of products such as perfumes, dyes, topical drugs and insect repellents.

2. The salt of benzoic acid (sodium benzoate) used as a food pH adjuster and preservative, stopping microbes from developing to keep food healthy.

**Cautions:**

1. Wash hands thoroughly after handling.

2. Use only in a well-ventilated area.

3. Avoid contact with eyes, skin, and clothing.

**Viva Voce Questions**

1. What is principle involved in synthesis of benzoic acid from Benzamide?

2. Write down reaction involved in the synthesis of benzoic acid from Benzamide.

3. Give the uses of benzoic acid.

**Experiment No: 15 Aim: Preparation of Picric acid from Phenol**

**Requirements**

**Apparatus:** Conical flask, measuring cylinder, glass rod, beaker, pipette, Buchner funnel

**Chemicals:** Phenol, Conc. Sulphuric acid, Conc. Nitric acid

**Principle:** Phenols when treated with concentrated nitric acid in the presence of concentrated sulphuric acid, undergo nitration at both ortho and para position to give picric acid. When phenol is first converted to phenol sulphonic acid by treatment with sulphuric acid and then nitrated with concentrated nitric acid when the SO3H (sulphonic acid) groups are replaced by NO2 (nitro) groups.

**Reaction**



**Mechanism**



**Structure**



**IUPAC Name:** 2, 4, 6-trinitrophenol

**Other Name:** Picric acid

**Molecular Formula:** C6H3N3O7

**Molecular Weight:** 229gm/mol

**Appearance:** Pale yellow, odourless crystals

**Melting point:** 122-1240C

**Solubility:** Alcohol, water

**Procedure**

1. Weigh accurately about 4 g of phenol (or take 5.0 ml of liquefied phenol) place in a flask and add 5.0 ml Con. sulphuric acid and mix thoroughly. Reaction is exothermic, the mixture becomes warm.

2. Heat the flask in a water bath for 30 minutes to complete the formation of phenol sulphonic acid. Cool the flask thoroughly in an ice cold water.

3. After, cooling, add immediately 15ml of con nitric acid and mix it well by shaking for a few seconds. A vigorous reaction takes place and harmless red nitrous fumes come out from the flask.

4. After the reaction subsides, heat the flask in a boiling water bath for 1-2 hours with constant shaking. Initially, a heavy oily layer is formed, which gets converted to a crystalline mass.

5. After heating, add ice cold water and chill the mixture in ice-cold water.

6. Collect the product on Buchners funnel by vacuum filtration, wash thoroughly with cold water till free from acidity, and drain completely.

7. Recrystallize the [product from the alcohol-water](https://pharmacyinfoline.com/cell-culture-microbiological-spoilage-mcq/) mixture. Filter off the crystalline product at the pump and dry by pressing between filter paper.

8. Report the percentage yield and melting point.

**Calculations:**

Molecular formula of Phenol = C6H6O

Molecular weight of Phenol = 94 g/mole

Molecular formula of Picric acid = C6H3N3O7

Molecular weight of Picric acid = 229gm/mol

94 g of Phenol is equivalent to 229 g Picric acid  
4 g of Phenol is equivalent to X g Picric acid

X= (229 × 4)/94 = 9.74 gm

Theoretical Yield = 9.74 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/ 9.74 gm

= ……………….%

**Result:**

Picric acid was synthesized from Phenol and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses**

1. It is also used in manufacturing coloured glass, in the leather industry, and in the synthesis of dyes.

2. Picric acid is both antibacterial and astringent.

3. It is also used in medical applications as a surface anesthetic ointment or solution, as well as burn ointments.

**Cautions**

1. Do not use a new bottle until the old picric acid is used completely.

2. Avoid contact with skin and eyes.

**Viva Voce Questions**

1. What is IUPAC name of picric acid?

2. Give principle of preparation of picric acid from phenol.

3. What are the uses of picric acid?

**Experiment No: 16 Aim: Synthesis of Benzanilide from Aniline**

**Requirements:**

**Apparatus:** Iodine flask, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Aniline, sodium hydroxide, benzoyl chloride

**Principle:** The synthesis of benzanilide is based upon schotten baumann reaction. Introduction of benzoyl moiety instead of an active hydrogen atom present in hydroxyl (OH), primary amino (NH2) or secondary amino group (NH) is known as benzoylation reaction. This particular reaction essentially bears a close similar to the phenomenon of acetylation except towards specific conditions of the reagent is benzoyl chloride reacts in the presence pyridine or 10% NaOH. The amines extensively soluble in acid chloride than in NaOH, the reaction carried out between benzoyl chloride and amines where, NaOH neutralizes the liberated HCl and also catalyze the reaction.

**Chemical Reaction:**



**Mechanism:**



**Chemical Structure**



**IUPAC Name:** N-Phenyl Benzamide

**Other Names:** N-Phenyl benzimidazole

**Molecular Formula:** C13H11NO

**Molecular Weight:** 197 g/mol

**Appearance:** White Crystalline Solid

**Melting Point:** 162 to 1640C

**Solubility:** Insoluble in water and soluble in 85 % acetic acid and ethanol

**Procedure:**

1. Add 2 ml (2.04 gm) of aniline, 30 ml of 10% NaOH solution in iodine flask and shake well at Room temperature.

2. Add 3 ml of benzoyl chloride slowly in iodine flask containing solution and shake well.

3. Cork the flask securely and shake again for further 15-20minutes or till the odour of benzoyl chloride can no longer be detected.

4. Dilute the reaction mixture with cold water, filter out the crude benzanilide with suction on a Buchner funnel, and wash with cold water.

5. Recrystallize from hot alcohol. Report the practical yield, percentage yield and melting point.

**Calculations:**

Molecular formula of Aniline = C6H7N

Molecular weight of Aniline = 93 g/mol

Molecular formula of Benzanilide = C13H11NO

Molecular weight of Benzanilide = 197 g/mol

93 gm of aniline is equivalent to 197 gm of benzanilide

2.04 gm of aniline is equivalent to X gm of benzanilide

X= (197 × 2.04)/93 = 4.32 gm

Theoretical Yield = 4.32 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/4.32gm

= ……………….%

**Result:**

Benzanilide was synthesized from aniline and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

* 1. Benzanilide used to prepare dyes, active pharmaceutical ingredients and perfumes.
  2. It is also used an amide model compound to study the reaction between amide and epoxy.
  3. Benzanilide is used as fungicide and acaricide (pesticide that kill ticks and mites).

**Cautions:**

1. Wear gloves when using chemicals.

2. Add the benzoyl chloride only in fuming hood.

3. Before removal stopper, check for completeness of reaction.

4. Release pressure during shaking by gently removing the stopper.

**Questions:**

1. What is starting material for synthesis of benzanilide?

2. What are the uses of Benzanilide?

3. What is the principle of synthesis of Benzanilide?

4. What is Benzoylation?

**Experiment No: 17 Aim: Synthesis of phenyl benzoate from phenol**

**Requirements:**

**Apparatus:** Iodine flask, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Phenol, sodium hydroxide, benzoyl chloride

**Principle:** Phenols reacts with an aromatic acid chloride in the presence of excess of NaOH at RT to form an ester this reaction is called as schotten baumann reaction. If phenol is shaken with benzoyl chloride, 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 phenoxide by dissolving it in sodium hydroxide solution so, this ion reacts more rapidly with benzoyl chloride than the original phenol does, but even so you have to shake with benzoyl chloride for about 15 minutes. Solid phenyl benzoate is formed.

**Reaction:**



**Mechanism:**



**Chemical Structure:**



**IUPAC Name:** 4-Butyl-1, 2-diphenyl-pyrazolidine-3, 5-Dione

**Other Names:** Butazolidine

**Molecular Formula:** C13H10O2

**Molecular Weight:** 198 g/mol

**Appearance:** White Solid Powder

**Melting Point:** 68 to 690C

**Solubility:** Insoluble in water and soluble in other organic solvent

**Procedure:**

1. Dissolve 1.0 g of phenol in 15 ml of 10% sodium hydroxide solution in a 250ml iodine flask and then add 2 ml benzoyl chloride to it.

2. Cork the flask properly shake the mixture vigorously until the smell of benzoyl chloride disappeared.

3. The phenyl benzoate which separates is filtered off, washed with cold water, dried and recrystallized from alcohol.

4. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Phenol = C6H6O

Molecular weight of Phenol = 94 g/mol

Molecular formula of Phenyl Benzoate = C13H10O2

Molecular weight of Phenyl Benzoate = 198 g/mol

94 gm of phenol is equivalent to 198 gm of phenyl benzoate

1.0 gm of phenol is equivalent to X gm of phenyl benzoate

X= (198 × 1.0)/94 = 2.11 gm

Theoretical Yield = 2.11 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/2.11gm

= ……………….%

**Result:**

Phenyl Benzoate was synthesized from Phenol and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. 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.

2. Phenyl benzoate based liquid crystals have excellent compatibility characteristics with other materials used in liquid crystal displays, such as biphenyl, phenyl cyclohexane, and bicyclohexane and fluorine types, especially at low temperatures.

**Cautions:**

1. Wear gloves when using chemicals.

2. Phenol is not only toxic but will cause severe burns.

3. Release pressure during shaking by gently removing stopper.

**Questions:**

1. Explain the mechanism and reaction of schotten-baumann reaction.

2. Which starting material used for the synthesis of phenyl benzoate?

3. What is the principle of synthesis of phenyl benzoate?

**Experiment No: 18 Aim: Synthesis of Acetanilide from Aniline**

**Requirements:**

**Apparatus:** RBF, reflux condenser, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Aniline, con. HCl, acetic anhydride, ethanol, charcoal powder, sodium acetate, ice

**Principle:** Acetanilide is synthesized from aniline by acetylating it with acetic anhydride in presence of glacial acetic acid. Aniline or phenyl amine is a primary amine and basic in nature, acetic anhydride, acts here as a source of acyl group. Aniline reacts with acetic anhydride to form acetanilide by nucleophilic substitution reaction and the reaction is called acetylation. In this reaction, aniline reacts acts as the nucleophile and acyl group from acetic anhydride acts as the electrophile. Here, the hydrogen atom of –NH2 group is replaced by the acyl group.

**Reaction:**



**Mechanism:**



**Chemical Structure:**



**IUPAC Name:** N-Phenyl acetamide

**Other Names:** N-Phenyl ethanamide

**Molecular Formula:** C8H9ON

**Molecular Weight:** 135 g/mol

**Appearance:** White Solid with flaky Powder

**Melting Point:** 113 to 1150C

**Solubility:** Insoluble in water and soluble in other organic solvent

**Procedure:**

1. Prepare a mixture of 10 ml of acetic anhydride and 10 ml of glacial acetic acid in beaker. Add 10 ml (10.3 gm) of aniline in a 250 ml Round Bottom Flask (RBF).

2. Carefully then add 20 ml of acetic anhydride and glacial acetic acid mixture (equal volumes). Fit a reflux water condenser to the flask and gently boil the mixture for 10 to 15 min. Then pour the hot liquid into 200ml of cold water with constant stirring. The acetanilide quickly crystallizes.

4. Filter yield a suction pump and wash the crude acetanilide well with water. Recrystallize from methylated spirit. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Aniline = C6H7O

Molecular weight of Aniline = 93 g/mol

Molecular formula of Acetanilide = C8H9ON

Molecular weight of Acetanilide = 135 g/mol

93 gm of aniline is equivalent to 135 gm of acetanilide

10.3 gm of aniline is equivalent to X gm of acetanilide

X= (135 × 10.3)/93 = 14.95 gm

Theoretical Yield = 14.95 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/14.95gm

= ……………….%

**Result:**

Acetanilide was synthesized from aniline and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. Acetanilide is used in the synthesis of penicillin and in other pharmaceuticals.

2. Acetanilide used for the production of 4-acetamidobenzenesulfonyl chloride, key intermediate for the manufacture of the sulfa drugs.

3. Acetanilide was the first aniline derivative found to possess analgesic as well as antipyretic properties.

**Cautions:**

1. Wear gloves when using chemicals.

2. Acetic anhydride is corrosive and must be handled carefully.

3. Do not inhale.

4. Avoid contact with skin

**Questions:**

1. What is Acetylation? Name any two acetylating agent.

2. What is nitrating mixture?

**Experiment No: 19 Aim: Synthesis of 2, 4, 6-Tribromoaniline from aniline**

**Requirements:**

**Apparatus:** Iodine flask, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Aniline, glacial acetic acid, bromine

**Principle:** Aniline undergoes nucleophilic substitution with bromine, the bromine atoms enter at the two ortho positions and the para position with the formation of 2, 4, 6-tribromoaniline. Here presence of bromine atoms in tribromoaniline, reduces the basic properties of the amino group, and salts even with strong acids are almost completely hydrolysed in presence of water.

**Reaction:**



**Mechanism:**





**Chemical Structure:**



**IUPAC Name:** 2, 4, 6 Tribromoaniline

**Other Names:** Benzenamine

**Molecular Formula:** C6H4Br3N

**Molecular Weight:** 329 g/mol

**Appearance:** White Yellow needle shape solid

**Melting Point:** 120 to 1220C

**Solubility:** Insoluble in water and soluble in alcohol

**Procedure:**

1. Take 5ml (4.66 gm) of aniline dissolve in 20 ml of glacial acetic acid in a flask.
2. Place the flask in ice bath and add to it 8 ml of bromine in 20 ml of glacial acetic acid drop wise with constant stirring.
3. Pour the reaction mixture in a beaker with excess of water.
4. Filter the product, wash with water and recrystallize from alcohol.
5. Report the Practical yield, Percentage yield and melting point.

**Calculation:**

Molecular formula of Aniline = C6H7O

Molecular weight of Aniline = 93 g/mol

Molecular formula of 2,4,6 Tribromoaniline = C6H4Br3N

Molecular weight of 2,4,6 Tribromoaniline = 329 g/mol

93 gm of aniline is equivalent to 329 gm of 2,4,6 Tribromoaniline

4.66 gm of aniline is equivalent to X gm of 2,4,6 Tribromoaniline

X= (329 × 4.66)/93 = 16.5 gm

Theoretical Yield = 16.5 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/16.5gm

= ……………….%

**Result:**

2,4,6 Tribromoaniline was synthesized from aniline and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is used in the organic synthesis of pharmaceuticals, agrochemicals, and fire extinguishing agents.

**Cautions:**

1. Glass ware should be clean and dry before being used.

2. Bromine must be handle with care. If the vapour is inhaled, relief may be obtained by soaking a handkerchief in a alcohol and holding it near the nose.

**Questions:**

1. What is the bromination reaction?

2. What is the principle of synthesis of 2,4,6 Tribromoaniline?

**Experiment No: 20 Aim: Synthesis of Para bromoacetanilide from acetanilide**

**Requirements:**

**Apparatus:** Iodine flask, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Acetanilide, bromine, sodium bisulphite, glacial acetic acid

**Principle:** The synthesis of p-bromoacetanilide is nuclear bromination. Bromination of acetanilide occurs at the para position due to the amine substituent. This substituent provides resonance stabilization to the carbocations created by ortho and para addition. Since the amine provides steric hindrance at the ortho position, bromination of acetanilide occurs at the para position. The function of the catalyst is to increase the electrophilic activity of the halogen.

**Reaction:**



**Mechanism:**





**Chemical Structure:**



**IUPAC Name:** N-(4-Bromophenyl) acetamide

**Other Names:** 4-Bromoacetanilide

**Molecular Formula:** C8H8BrNO

**Molecular Weight:** 214 g/mol

**Appearance:** White to light beige crystalline (white to off white) solid

**Melting Point:** 165 to 1670C

**Solubility:** Insoluble in cold water and soluble in benzene and chloroform and moderately soluble in alcohol

**Procedure:**

1. Dissolve 2.7 g of finely powdered acetanilide in 9 ml of glacial acetic acid in iodine flask.

2. In another small flask dissolve 1 ml of bromine in 5 ml of glacial acetic acid and transfer the solution to a separating funnel.

3. Add the bromine solution slowly with constant shaking to ensure thorough mixing; stand the flask in cold water. When all the bromine has been added, the solution will have an orange colour due to the slight excess of bromine; a part of the reaction product may crystallize out.

4. Allow the final reaction mixture to stand at RT for 30 minutes with occasional shaking.

5. Pour the reaction mixture into 200 ml of water; rinse the flask with about 50 ml of water.

6. Stir the mixture well and if it is appreciably coloured, add just sufficient sodium bisulphite.

7. Filter the crystalline precipitate with suction on a Buchner funnel, wash thoroughly with cold water and dry it. It is recrystallize from methanol.

8. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Acetanilide = C8H9NO

Molecular weight of Acetanilide = 135 g/mol

Molecular formula of Parabromoacetanilide = C8H8BrNO

Molecular weight of Parabromoacetanilide = 214 g/mol

135 gm of acetanilide is equivalent to 214 gm of Parabromoacetanilide

2.7 gm of acetanilide is equivalent to X gm of Parabromoacetanilide

X= (214 × 2.7)/135 = 4.28 gm

Theoretical Yield = 4.28 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/4.28 gm

= ……………….%

**Result:**

Parabromoacetanilide was synthesized from acetanilide and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is used as an inhibitor of hydrogen peroxide decomposition and is used to stabilize cellulose ester varnishes.

2. Used as an internal standard for phenyl urea and metabolic products in oysters.

3. It is also a precursor in the synthesis of penicillin and other pharmaceuticals.

**Cautions:**

1. Bromine is very toxic, corrosive and dangerous for the environment. Be careful while using it.

2. The concentrated acids are corrosives. Gloves should be worn when transferring these reagents.

**Questions:**

1. Identify what is the electrophile in this reaction?

2. What is the principle of Parabromoacetanilide?

3. Why glacial acetic acid is used in the bromination of acetanilide?

4. What are the uses of Parabromoacetanilide?

**Experiment No: 21 Aim: Synthesis of 5-Nitrosalicylic acid from salicylic acid**

**Requirements:**

**Apparatus:** Iodine flask, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Nitric acid, salicylic acid

**Principle:** Nitration on salicylic acid occurs by placing a nitro group on the aromatic ring system via an electrophilic aromatic substitution reaction. Here calcium nitrate is used as the nitrating agent in presence of acetic acid. Two groups –CO2H and –OH in salicylic acid complement each other since they both direct entering nitro group to the 5th position. The 5th position and the 3rd position are both electronically favored since the –CO2H group is meta directing and the –OH group is ortho-para directing. The nitro group is attached at the 5th position, and not at the 3rd position, due to steric effects. Also use anhydrous nitric acid or concentrated nitric acid and concentrated sulphuric acid as nitrating reagent.

**Reaction:**



**Mechanism:**



**Chemical Structure:**



**IUPAC Name:** 2-Hydroxy-5-nitrobenzoic acid

**Other Names:** 5-nitrosalicylic acid

**Molecular Formula:** C7H5NO5

**Molecular Weight:** 198 g/mol

**Appearance:** Yellow beige to orange brown solid

**Melting Point:** 228 to 2300C

**Solubility:** Soluble in cold water moderately soluble in alcohol

**Procedure:**

* 1. Take 2 g of salicylic acid in iodine flask and add small amount of hot distilled water in it.
  2. Add slowly 5 ml conc. Nitric acid with constant shaking of the flask,
  3. Heat the flask on boiling water bath for about 5 minutes till no more brown fumes come out from the reaction mixture.
  4. Cool the reaction mixture and pour into 50 ml ice cold distilled water with constant stirring.
  5. Filter the crude product using suction pump and wash with 2-3times with cold distilled water. Dry the crude product. Recrystallize the product using hot distilled water.
  6. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Salicylic acid = C7H6O3

Molecular weight of Salicylic acid = 138 g/mol

Molecular formula of 5 Nitro salicylic acid = C7H5NO5

Molecular weight of 5 Nitro salicylic acid = 198 g/mol

138 gm of salicylic acid is equivalent to 198 gm of 5 Nitro salicylic acid

2 gm of salicylic acid is equivalent to X gm of 5 Nitro salicylic acid

X= (198 × 2)/138 = 2.87 gm

Theoretical Yield = 2.87 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/2.87 gm

= ……………….%

**Result:**

5-Nitrosalicylic acid was synthesized from salicylic acid and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is an important raw material and intermediate used in organic synthesis, pharmaceuticals, agrochemicals and dyestuff.

2. It is also used as an important organic, pharmaceuticals intermediate.

**Cautions:**

1. Glass ware should be clean and dry before being used.
2. Use chemicals only in the hood and be sure the hood exhaust fan is on.
3. Wear gloves when using chemicals.
4. Be careful when using nitric acid.

**Questions:**

1. What type of reaction is the addition of salicylic acid to 5-Nitro salicylic acid?

2. Identify what is the electrophile in this reaction?

3. What are the uses of 5 nitro salicylic acid?

4. What is the principle involved in synthesis of 5 nitro salicylic acid?

**Experiment No: 22 Aim: Synthesis of Meta di nitro benzene from nitrobenzene**

**Requirements:**

**Apparatus:** RBF, Reflux condenser, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Nitrobenzene, conc. Nitric acid, conc. Sulphuric acid

**Principle:** Nitration is occurring on nitrobenzene. It is an electrophilic aromatic substitution in presence of NO2, which is a strong electron withdrawing group and it directs the incoming substituents to the meta position. Here nitronium ions act as the electrophile which is generated from fuming nitric acid in presence of conc. Sulphuric acid.

**Reaction:**



**Mechanism:**



58



**Chemical Structure:**



**IUPAC Name:** 1,3-Dinitrobenzene

**Other Names:** meta-dinitrobenzene

**Molecular Formula:** C6H4N2O4

**Molecular Weight:** 168 g/mol

**Appearance:** Yellow solid

**Melting Point:** 88 to 900C

**Solubility:** Insoluble in cold water and soluble in an organic solvent

**Procedure:**

1. Place 15 ml (22.5 gm) of nitric acid and 21 ml (37.5 gm) of conc. Sulphuric acid in a round bottomed flask (RBF) with some pieces of porcelain or glass to avoid bumping.
2. Attach reflux condenser and add 12.5 ml (15 gm) of nitrobenzene dropwise with constant stirring. Reaction mixture is heated on a boiling water bath for 15 minutes.
3. Allow the mixture to cool and pour it with constant stirring to 300 ml of cold water taken in a beaker.
4. Filter the product, wash with water and recrystallize from ethanol.
5. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Nitrobenzene = C6H5NO2

Molecular weight of Nitrobenzene = 123 g/mol

Molecular formula of Meta dinitrobenzene = C6H4N2O4

Molecular weight of Meta dinitrobenzene = 168 g/mol

123 gm of Nitrobenzene is equivalent to 168 gm of Meta dinitrobenzene

15 gm of Nitrobenzene is equivalent to X gm of Meta dinitrobenzene

X= (168 × 15)/123 = 20.48 gm

Theoretical Yield = 20.48 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/20.48 gm

= ……………….%

**Result:**

Meta dinitrobenzene was synthesized from nitrobenzene and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is used an organic drug synthesis.

2. Mainly uses as synthetic intermediates in pharmaceuticals.

3. It is used in making dyes, other chemicals and explosives.

**Cautions:**

1. Glass ware should be clean and dry before being used.
2. Be careful when using nitric acid.
3. Use chemicals only in the hood and be sure the hood exhaust fan is ON.

**Questions:**

1. What is the limiting reactant in the nitration of nitrobenzene?

2. What are the uses of Meta dinitrobenzene?

3. What is nitration?

**Experiment No: 23 Aim: Synthesis of Nitrobenzene by Nitration Reaction**

**Requirements:**

**Apparatus:** RBF, Reflux condenser, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Benzene, concentrated nitric acid, concentrated sulphuric acid, anhydrous calcium chloride

**Principle:** Nitration acts as a substitution reaction in which –NO2 group is substituted on aromatic ring. Aromatic hydrocarbons may be nitrated, where hydrogen atoms replaced by nitro group with concentrated nitric acid in the presence of concentrated sulphuric acid.

**Reaction:**



**Mechanism:**





**Chemical Structure:**



**IUPAC Name:** Nitrobenzene

**Other Names:** Nitrobenzol

**Molecular Formula:** C6H5NO2

**Molecular Weight:** 123 g/mol

**Appearance:** Yellowish, oily liquid

**Boiling Point:** 210 to 2120C

**Solubility:** Water insoluble pale yellow oil

**Procedure:**

1. Place 17.5 ml of concentrated nitric acid in round bottomed flask (RBF). To this add 20 ml of concentrated sulphuric acid. Keep the round bottom flask in ice bath and place a thermometer.
2. Try to maintain a temperature 50-550C. Introduction of 15 ml (12.5 gm) of benzene in portions of 1-2 ml. Shake well to ensure thorough mixing after each addition of benzene.
3. Do not allow temperature to rise above 55-600C.
4. When all the benzene is added fit a reflux condenser to the RBF.
5. Heat the flask in water bath maintained at 600C for 10-15 minutes.
6. Pour the content to the flask into 250 ml of cold water in a beaker. Stir it and allow to stand.
7. When nitrobenzene has settled to the bottom pour off the liquid as completely as possible.
8. Transfer the residual liquid into separating funnel and add 25ml of water shake well and allow to stand.
9. Separate the nitrobenzene and add into conical flask containing about 2.5 g of calcium chloride. Filter the product.
10. Report the Practical yield, Percentage yield and Boiling point.

**Calculation:**

Molecular formula of Benzene = C6H6

Molecular weight of Benzene = 78 g/mol

Molecular formula of Nitrobenzene = C6H5NO2

Molecular weight of Nitrobenzene = 123 g/mol

78 gm of Benzene is equivalent to 123 gm of Nitrobenzene

12.5 gm of Benzene is equivalent to X gm of Nitrobenzene

X= (123 × 12.5)/78 = 19.71 gm

Theoretical Yield = 19.71 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/19.71 gm

= ……………….%

**Result:**

Nitrobenzene was synthesized from benzene and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Boiling Point | ………..0C |

**Uses:**

1. It is used to manufacture a chemical called aniline.

2. It is also used to produce lubricating oils such as those used in motors and machinery.

3. Used in the manufacture of dyes, drugs, pesticides and synthetic rubber.

**Cautions:**

1. Nitrobenzene is toxic. If it is accidentally spilled on the skin, it should remove by washing with a little methylated spirit, followed by soap and warm water.
2. Wear gloves when using chemicals.

**Questions:**

1. What is nitrating mixture?

2. What is the principle and reaction involved in synthesis of nitrobenzene.

**Experiment No: 24 Aim: Synthesis of Benzoic acid from Benzyl chloride by Oxidation Reaction**

**Requirements:**

**Apparatus:** RBF, Reflux condenser, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Benzyl chloride, anhydrous sodium carbonate, potassium permanganate, sodium sulfite, Hydrochloric acid

**Principle:** If oxidation occurs to an aromatic compound having an aliphatic side chain then fission of the side chain occurs between the first and second carbon atom from the benzene ring. The oxidation process is carried out with a mixture of potassium permanganate and sodium carbonate in aqueous solution or with dilute nitric acid. The reaction is quite slow if the side chain is a simple alkyl group. The side chain containing alkyl group is more susceptible to oxidation. Hence in comparison to toluene, benzoyl chloride more rapidly oxidizes in presence of an aqueous oxidizing agent. Here benzyl chloride is first hydrolyzed to benzyl alcohol and then undergoes oxidation of a primary alcohol to the corresponding carboxylic acid.

**Reaction:**



**Chemical Structure:**



**IUPAC Name:** Benzene carboxylic acid

**Other Names:** Carboxy benzene

**Molecular Formula:** C7H6O2

**Molecular Weight:** 122 g/mol

**Appearance:** Colourless crystalline solid

**Melting Point:** 119 to 1210C

**Solubility:** It is soluble in organic solvents such as 95 % of ethanol but slightly soluble in water

**Procedure:**

1. Place about 5 ml (5.5 gm) benzyl chloride into a round bottomed flask equipped with a reflux condenser.
2. Then add 5 g sodium carbonate (Na2CO3) , 4.5 g potassium permanganate, 50 ml distilled water and a few excess boiling chips to it.
3. Boil the reaction mixture gently for 2 hours).
4. Allow to cool, acidify with 20 ml conc. Hydrochloric acid and add 20% aqueous solution of sodium sulfite (Na2SO3. 7H2O) with shaking to dissolve MnO2 completely and make sure the complete precipitation of benzoic acid.
5. Filter the precipitation and wash it with cold water.  Recrystallize from boiling water, cool and filter it. Dry the crystallized product.
6. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Benzyl chloride = C7H7Cl

Molecular weight of Benzyl chloride = 140 g/mol

Molecular formula of Benzoic acid = C7H6O2

Molecular weight of Benzoic acid = 122 g/mol

140 gm of Benzyl chloride is equivalent to 122 gm of Benzoic acid

5.5 gm of Benzyl chloride is equivalent to X gm of Benzoic acid

X= (122 × 5.5)/140 = 4.78 gm

Theoretical Yield = 4.78 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/4.78 gm

= ……………….%

**Result:**

Benzoic acid was synthesized from Benzyl chloride and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is used as a topical agent with salicylic acid to treat the skin problem such as irritation and inflammation which may causes by burns, insect bites and fungal infection.

2. Benzoic acid is most found in industrial settings to manufacture a wide variety of products such as perfumes, dyes, topical medications, and insect repellents.

**Cautions:**

1. Glass ware should be clean and dry before being used.

2. Wear gloves when using chemicals.

**Questions:**

1. What type of reaction is the addition of benzoyl chloride to benzoic acid?

2. What is the principle and reaction involved in synthesis of benzoic acid?

3. What is oxidation reaction?

**Experiment No: 25 Aim: Synthesis of Benzoic acid from Ethyl Benzoate by Base Hydrolysis**

**Requirements:**

**Apparatus:** RBF, reflux condenser, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Ethyl benzoate, sodium hydroxide, hydrochloric acid

**Principle:** Acid or a base are used for hydrolysis of esters. Alkaline hydrolysis of ester is irreversible which is also called saponification. Acid hydrolysis of ester is a reversible reaction. Acid hydrolysis of esters can occur by the mechanism of a nucleophilic acyl substitution. Here ethyl benzoate on hydrolysis with sodium hydroxide gives benzoic acid and ethyl alcohol where OH- ion of sodium hydroxide act as a nucleophile.

**Reaction:**



**Chemical Structure:**



**IUPAC Name:** Benzene carboxylic acid

**Other Names:** Carboxy benzene

**Molecular Formula:** C7H6O2

**Molecular Weight:** 122 g/mol

**Appearance:** Colourless crystalline solid

**Melting Point:** 119 to 1210C

**Solubility:** It is soluble in organic solvents such as 95 % of ethanol but slightly soluble in water

**Procedure:**

1. A mixture of 2 ml (1.2 gm) ethyl benzoate and 15 ml 10% sodium hydroxide solution is refluxed in a round bottomed flask (RBF) fitted with a water condenser on a water bath for about 30 minute still the ester layer disappears.
2. Then the solution is cooled and acidified with HCl.
3. The resultant acidified solution is cooled in an ice bath.
4. The separated benzoic acid precipitate is filtered and recrystallised from hot water.
5. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Ethyl benzoate = C9H10O2

Molecular weight of Ethyl benzoate = 150 g/mol

Molecular formula of Benzoic acid = C7H6O2

Molecular weight of Benzoic acid = 122 g/mol

150 gm of Ethyl benzoate is equivalent to 122 gm of Benzoic acid

1.2 gm of Ethyl benzoate is equivalent to X gm of Benzoic acid

X= (122 × 1.2)/150 = 0.97 gm

Theoretical Yield = 0.97 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/0.97 gm

= ……………….%

**Result:**

Benzoic acid was synthesized from ethyl benzoate and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. Benzoic acid’s salt (sodium benzoate) is commonly used as adjustor and preservative in food, preventing the growth of microbes to keep food safe.

2. It is a sweet smelling, colourless, liquid used in perfumery under the name Essence de Niobe; in the manufacture of Peau d’Espagne; and as an artificial fruit essence.

**Cautions:**

1. Glass ware should be clean and dry before being used.

2. Wear gloves when using chemicals.

**Questions:**

1. What are the uses of benzoic acid?

2. Which starting material used for synthesis of benzoic acid by base hydrolysis reaction?

3. Which type of reaction involved in the synthesis of benzoic acid?

4. What is saponification reaction?

**Experiment No: 26 Aim: Synthesis of Salicylic Acid from Alkyl salicylate by Hydrolysis Reaction**

**Requirements:**

**Apparatus:** RBF, reflux condenser, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Methyl salicylate, sodium hydroxide, sulphuric acid

**Principle:** Alkaline hydrolysis of esters is called saponification and is an irreversible process. Here one mole of methyl salicylate reacts with two moles of sodium hydroxide to form sodium salicylate with methanol and water each of one mole. Sodium salicylate is reacted with sulfuric acid or hydrochloric acid to remove the sodium ion and forms salicylate acid with sodium sulphate as a byproduct.

**Reaction:**



**Mechanism:**



**Chemical Structure:**



**IUPAC Name:** 2-Hydroxybenzoic acid

**Other Names:** ortho-hydroxy benzoic acid

**Molecular Formula:** C7H6O3

**Molecular Weight:** 138 g/mol

**Appearance:** Colourless to white crystals

**Melting Point:** 158 to 1600C

**Solubility:** It is soluble in organic solvents such as ethanol but slightly soluble in water

**Procedure:**

1. Prepare freshly a solution of 25 ml 5M NaOH. Pour into 100 ml round bottomed flask (RBF) and add 3 ml (3.5) g of methyl salicylate containing the NaOH.
2. Heat the reaction mixture to boiling using a heating mantel for about 20 minutes and add 3-4 boiling porceline pieces to prevent bumping.
3. After 20 minutes reflux, transfer the reaction mixture to a beaker.
4. Add 50 ml of water to the reaction contents in your beaker and stir vigorously with a glass stirring rod as you add acid in the next step.
5. Carefully add enough 3M sulphuric acid to make the solution acidic to pH of 1.
6. After pH paper shows a pH of 1, add 1-2 ml more of the 3M sulphuric acid to ensure all the salicylic acid precipitate.
7. Cool the mixture in an ice bath to about 00C for about 15 min. while allowing the crystals to settle.
8. Collect the crystals by vacuum filtration, using a Buchner funnel and filter paper.
9. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Methyl salicylate = C8H8O3

Molecular weight of Methyl salicylate = 152 g/mol

Molecular formula of Salicylic acid = C7H6O3

Molecular weight of Salicylic acid = 138 g/mol

152 gm of Methyl salicylate is equivalent to 138 gm of Salicylic acid

3.5 gm of Methyl salicylate is equivalent to X gm of Salicylic acid

X= (138 × 3.5)/152 = 3.1 gm

Theoretical Yield = 3.1 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/3.1 gm

= ……………….%

**Result:**

Salicylic acid was synthesized from Methyl salicylate and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. Salicylic acid was one of the original starting materials for making acetylsalicylic acid (aspirin).

2. Salicylic acid is used as a food preservative, a bactericidal and an antiseptic.

3. Salicylic acid is used in the production of other pharmaceuticals, including 4-aminosalicylic acid, sandulpiride, and landetimide.

**Cautions:**

1. Glass ware should be clean and dry before being used.

2. Wear gloves when using chemicals.

3. The concentrated acids are corrosives. Gloves should be worn when transferring these reagents.

**Questions:**

1. What are the uses of salicylic acid?

2. Which are the starting material used in the synthesis of salicylic acid?

3. What type of reaction involved in the synthesis of salicylic acid?

**Experiment No: 27 Aim: Synthesis of 1-Phenylazo-2-naphthol from Aniline by Diazotisation and Coupling Reaction**

**Requirements:**

**Apparatus:** Conical flask, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Aniline, conc. HCl, sodium nitrite, 2-Naphthol, sodium hydroxide, glacial acetic acid

**Principle:** Phenyl diazonium chloride is obtained by the diazotization of aniline with nitrous acid which on coupling with beta naphthol in the presence of NaOH solution yields the desired coupled product phenyl-azo-beta naphthol. A mole of HCl is eliminated which instantly reacts with NaOH from the medium NaCl and water.

**Reaction:**



**Chemical Structure:**



**IUPAC Name:** 1-phenyldiazenylnaphthalen-2-ol

**Other Names:** Sudan 1

**Molecular Formula:** C16H12N2O

**Molecular Weight:** 258 g/mol

**Appearance:** Orange red solid

**Melting Point:** 131 to 1330C

**Solubility:** It is soluble in organic solvents such as ethanol but insoluble in water

**Procedure:**

1. Take a 100 ml conical flask and add 5 ml of aniline, 10 ml of cone. HCl and 20 ml of water. Cool this solution to 5°C by placing the conical flask in a trough containing ice- cold water.
2. In a 100 ml beaker dissolve 4 g of sodium nitrite in 20 ml of water and cool this solution also to 5°C. Now slowly add sodium nitrite solution to the solution of aniline in conc. HCl.
3. Dissolve 8 g of 2-naphthol in 60 ml of 10% NaOH solution taken in a 250 ml beaker and cool this solution to 5°C by placing in an ice bath. Some crushed ice may be added directly to fecilitate cooling.
4. Now add the diazotised solution very slowly to the naphthol solution with constant stirring. The mixed solutions immediately develop a red colour and the phenyl-azo-β- naphthol rapidly separates as orange-red crystals.
5. When the addition of diazo solution is complete, allow the mixture to stand in ice-salt mixture for 30 minutes, with occasional stirring. Filter the solution through a buchner funnel under suction from the pump. Wash the phenyl-azo-β-naphthol with water and dry the crystals obtained by pressing between the folds of filter paper.
6. Recrystallise the product from glacial acetic acid. Filter the crystals obtained at the pump. Wash with a few ml of ethanol to remove acetic acid. Phenyl-azo-β-naphthol is obtained as orange-red crystals. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Aniline = C6H7O

Molecular weight of Aniline = 93 g/mol

Molecular formula of 1-Phenylazo 2 naphthol = C16H12N2O

Molecular weight of 1-Phenylazo 2 naphthol = 258 g/mol

93 gm of Aniline is equivalent to 258 gm of 1-Phenylazo 2 naphthol

5.5 gm of Aniline is equivalent to X gm of 1-Phenylazo 2 naphthol

X= (258 × 5.5)/93 = 15.2 gm

Theoretical Yield = 15.2 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/15.2 gm

= ……………….%

**Result:**

1-Phenylazo 2 naphthol was synthesized from Aniline and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. Azo dyes are used to dye textile fibers, especially cotton, as well as silk, wool, viscose and synthetic fibers.

**Cautions:**

1. Glass ware should be clean and dry before being used.

2. Wear gloves when using chemicals.

3. Use chemicals only in hood and be sure the hood exhaust fan is ON.

**Questions:**

1. Which starting material are used in the synthesis of 1-Phenylazo 2 naphthol.

2. What is coupling reaction?

3. What is diazotization reaction?

**Experiment No: 28 Aim: Synthesis of Benzil from Benzoin**

**Requirements:**

**Apparatus:** Conical flask, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Benzoin, glacial acetic acid, conc. Nitric acid

**Principle:** In this synthesis, alcohol group of benzoin is oxidized to ketone group forming benzil in the presence of concentrated nitric acid. Nitration of aromatic ring is not occurring as sulphuric acid is totally absent in the whole process.

**Reaction:**



**Mechanism:**



**Chemical Structure:**



**IUPAC Name:** 1,2-diphenylethane-1,2-dione

**Other Names:** dibenzoyl

**Molecular Formula:** C14H10O2

**Molecular Weight:** 210 g/mol

**Appearance:** Pale yellow crystalline powder

**Melting Point:** 92 to 940C

**Solubility:** It is soluble in organic solvents such as ethanol, benzene and diethyl ether but insoluble in water

**Procedure:**

1. Heat a mixture of 1 g of benzoin and 25 ml of conc. Nitric acid in a conical flask boiling water bath for one hr. Then pour down the contents in 50 ml ice cold water with continuous stirring.
2. Filter the product wash the yellow solid with cold water and recrystallize with methanol.
3. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Benzoin = C14H12O2

Molecular weight of Benzoin = 212 g/mol

Molecular formula of Benzil = C14H10O2

Molecular weight of Benzil = 210 g/mol

212 gm of Benzoin is equivalent to 210 gm of Benzil

1 gm of Benzoin is equivalent to X gm of Benzil

X= (210 × 1)/212 = 1.0 gm

Theoretical Yield = 1.0 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/1.0 gm

= ……………….%

**Result:**

Benzil was synthesized from Benzoin and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. Benzil is used in the free-radical curing of polymer networks. Ultraviolet radiation decomposes benzil, generating free-radical species within the material, promoting the formation of cross-links.

2. It is used in the manufacture of glycollate pharmaceuticals as a benzillic acid, clidinium, Dilantin and flutropium.

**Cautions:**

1. Glass ware should be clean and dry before being used.
2. The fumes of nitric acid are very dangerous, take precaution while handling it.
3. Wear gloves when using chemicals.
4. Use chemicals only in hood and be sure the hood exhaust fan is ON.

**Questions:**

1. What is the principle and reaction involved in the synthesis of benzil?

2. What are the uses of benzil?

**Experiment No: 29 Aim: Synthesis of Dibenzalacetone from Benzaldehyde by Claisen Schmidt reaction**

**Requirements:**

**Apparatus:** Conical flask, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Benzaldehyde, acetone, sodium hydroxide, ethanol

**Principle:** When an ethanolic solution containing acetone and its two equivalents of benzaldehyde is made alkaline with sodium hydroxide rapid condensation occurs with the formation of Dibenzalacetone or dibenzylidene-acetone. This is a particular example of claisen reaction as clasien showed that aldehyde under the influence of sodium hydroxide condenses with (i) another aldehyde or (ii) a ketone with the elimination of water. Thus benzaldehyde condenses with (i) acetaldehyde to give cinnamic aldehyde and with (ii) one equivalent of acetone to give benzal acetone.

**Reaction:**



**Mechanism:**



**Chemical Structure:**



**IUPAC Name:** 1,5-Dphenylpenta-1,4-dien-3-one

**Other Names:** Dibenzylideneacetone

**Molecular Formula:** C17H14O

**Molecular Weight:** 234 g/mol

**Appearance:** Pale yellow crystalline solid

**Melting Point:** 110 to 1120C

**Solubility:** It is soluble in organic solvents such as ethanol and diethyl ether but insoluble in water

**Procedure:**

1. Place 5 g of sodium hydroxide in conical flask. Add 50 ml of water and 40 ml of ethanol. Cool the flask in ice cold water.
2. Add 5 ml (5.2 gm) of Benzaldehyde and 2 ml of acetone into it with swirling of the contents.
3. Shake frequently and maintain the temperature at 20-250C for 15 minutes by immersion of the flask in ice cold water.
4. Filter off the precipitated dibenzalacetone at the pump.
5. Wash it with cold water to eliminate the alkali.
6. Recrystallize from hot rectified spirit.
7. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Benzaldehyde = C7H6O

Molecular weight of Benzaldehyde = 106 g/mol

Molecular formula of Dibenzalacetone = C17H14O

Molecular weight of Dibenzalacetone = 234 g/mol

106 gm of Benzaldehyde is equivalent to 234 gm of Dibenzalacetone

5.2 gm of Benzaldehyde is equivalent to X gm of Dibenzalacetone

X= (234 × 5.2)/106 = 11.4 gm

Theoretical Yield = 11.4 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/11.4 gm

= ……………….%

**Result:**

Dibenzalacetone was synthesized from Benzaldehyde and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. Dibenzalacetone used as a component in sunscreens, and some industrial organometallic compounds because it bonds to metals and helps form a stable chemical structure.

**Cautions:**

1. Glass ware should be clean and dry before being used.
2. Wear gloves when using chemicals.

**Questions:**

1. Dibenzal acetone preparation is based on which naming reaction.

2. What is condensation reaction?

3. Which starting material involved in the synthesis of dibenzalacetone?

**Experiment No: 30 Aim: Synthesis of Cinnamic Acid from Benzaldehyde by Perkin Reaction**

**Requirements:**

**Apparatus:** RBF, reflux condenser, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Benzaldehyde, acetic anhydride, anhydrous potassium carbonate, sodium hydroxide

**Principle:** The reaction between aromatic aldehyde and an aliphatic anhydride of providing an active methylene moiety in the presence of a basic catalyst, such as acetate ion and a hydronium ion, which yields an unsaturated carboxylic acid and a mole of acetic acid i.e. interaction between benzaldehyde and acetic anhydride in presence of acetate ion and a hydronium ion yields, cinnamic acid acetic acid.

**Reaction:**



**Mechanism:**

94



**Chemical Structure:**



**IUPAC Name:** 3-Phenylprop-2-enoic acid

**Other Names:** Cinnamylic acid

**Molecular Formula:** C9H8O2

**Molecular Weight:** 148 g/mol

**Appearance:** White crystalline powder

**Melting Point:** 131 to 1330C

**Solubility:** It is soluble in organic solvents such as ethanol but slightly soluble in water

**Procedure:**

1. Place 5 ml (5.2 gm) of benzaldehyde, 8.2ml of acetic anhydride and 7g of anhydrous potassium carbonate in RBF.

2. Attach a condenser and thermometer. Heat the flask in air bath gradually.

3. Much foaming occurs at 1000C. When foaming has subsided, maintain the temperature at 170-1800C for about 90 minutes.

4. Allow the reaction mixture to cool. Add 40ml of water followed by 10% NaOH solution until alkaline.

5. Extract the clear solution with two 25ml of portions of ether to remove any unreacted benzaldehyde.

6. Treat the aqueous layer with conc. HCl until acid to Congo red with stirring.

7. Filter of the precipitated acid at pump. Wash with water and recrystallize from hot water.

8. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of Benzaldehyde = C7H6O

Molecular weight of Benzaldehyde = 106 g/mol

Molecular formula of Cinnamic acid = C9H8O2

Molecular weight of Cinnamic acid = 148 g/mol

106 gm of Benzaldehyde is equivalent to 148 gm of Cinnamic acid

5.2 gm of Benzaldehyde is equivalent to X gm of Cinnamic acid

X= (148 × 5.2)/106 = 7.2 gm

Theoretical Yield = 7.2 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/7.2 gm

= ……………….%

**Result:**

Cinnamic acid was synthesized from Benzaldehyde and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. Cinnamic acids is compose a relatively large family of organic acids which appear to have antibacterial, antifungal and anti-parasitical activities.

2. It is also used in the perfume production, the food industry, pharmaceuticals, medicine and technical applications, cinnamic acids are synthesized on a commercial scale.

**Cautions:**

1. Glass ware should be clean and dry before being used.
2. Wear gloves when using chemicals.

**Questions:**

1. Describe Perkin reaction.

2. What catalysts may be used in the Perkin reaction?

3. What is the principle and reaction involved in the synthesis of Cinnamic acid?

**Experiment No: 31 Aim: Synthesis of P-Iodo benzoic acid from P-Amino benzoic acid**

**Requirements:**

**Apparatus:** Conical flask, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** P-amino benzoic acid, hydrochloric acid, sodium nitrite, potassium iodide

**Principle:** It consists of diazotization of P-amino benzoic acid followed by reaction with potassium iodide.

**Reaction:**



**Mechanism:**



**Chemical Structure:**



**IUPAC Name:** 4-iodo benzoic acid

**Other Names:** P-iodo benzoic acid; Benzoic acid 4 iodo

**Molecular Formula:** C7H5IO2

**Molecular Weight:** 248 g/mol

**Appearance:** Off white to light brown colour powder

**Melting Point:** 271 to 2730C

**Solubility:** It is soluble in organic solvents such as ethanol and ether but slightly soluble in water.

**Procedure:**

1. Place 7 g of p-aminobenzoic acid (PABA) into a conical flask. Add 10 ml 3M HCl.

2. Warm gently while stirring until Para Amino Benzoic Acid dissolves. Dissolve 4.4 ml of NaNO2 in 10 ml water. Cool both solutions in ice baths until both are below 5ºC. Add sodium nitrite solution to conical flask, keep below 10ºC.

3. Test with starch-iodide paper, add minute amounts of urea to give a negative test.

4. Dissolve 9.4 ml of KI solution in 100 ml water.

5. Pour diazonium salt into beaker with the KI solution, stir it. Heat gently, pop foam with glass rod. Collect product with vacuum filtration, wash with cold water. Recrystallize from ethanol.

6. Report the Practical yield, Percentage yield and Melting point.

**Calculation:**

Molecular formula of P-amino benzoic acid = C7H7NO2

Molecular weight of P-amino benzoic acid = 137 g/mol

Molecular formula of P-iodo benzoic acid = C7H5IO2

Molecular weight of P-iodo benzoic acid = 248 g/mol

137 gm of P-amino benzoic acid is equivalent to 248 gm of P-iodo benzoic acid

7 gm of P-amino benzoic acid is equivalent to X gm of P-iodo benzoic acid

X= (248 × 7)/137 = 12.6 gm

Theoretical Yield = 12.6 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/12.6 gm

= ……………….%

**Result:**

P-iodo benzoic acid was synthesized from P-amino benzoic acid and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. 4-Iodobenzoic acid is used as anti-infective, contraceptive agent and x-ray contrast medium for diagnostic radiology.

**Cautions:**

1. Glass ware should be clean and dry before being used.
2. Wear gloves when using chemicals.
3. Use chemicals only in hood and be sure the hood exhaust fan is ON.

**Questions:**

1. Describe Sandmeyer reaction.

2. Explain principle and reaction involved in the synthesis of Para iodo benzoic acid.

3. What catalysts are used in the Sandmeyer reaction?

**Experiment No: 32 Aim: Synthesis of Phenytoin from Benzil**

**Requirements:**

**Apparatus:** Round Bottom Flask, Reflux, Condenser, beaker, measuring cylinder, Buchner funnel, filter paper, Magnetic stirrer, Melting point apparatus

**Chemicals:** Benzil, urea, 30% Sodium hydroxide solution, ethanol, Conc. HCl

**Principle:** Urea structural unit in 5, 5-diphenyl hydantoin and it constitutes one of the reagents in the synthesis process along with the benzil. The base catalysed reaction proceeds via intermediate heterocyclic pinacole which on acidification gives hydantoin as pinacole rearrangement. Where, pinacole-pinacolone rearrangement mechanism involves the conversion of pinacole-pinacolone under acidic conditions with a loss of water from 1, 2 diole shift by a 1, and 2 nucleophilic shift of an alkyl group or a hydride group.

**Chemical Reaction:**



**Mechanism:**



**Chemical Structure**



**IUPAC Name:** 5, 5-di(phenyl)imidazolidine-2,4-dione

**Other Names:** Solantin

Dilantin

Alepsin

**Molecular Formula:** C15H12N2O2

**Molecular Weight:** 252 g/mol

**Appearance:** Fine white or almost white crystalline powder

**Melting Point:** 295 to 2970C

**Solubility:** Ethanol, Dimethyl sulphoxide, Dimethyl formamide

**Procedure:**

1. Take 5.3 gm of benzil, 3.0 gm of urea, 15 ml of aqueous sodium hydroxide solution (30%) and 75 ml of ethanol in a 250 ml capacity round bottomed flask.

2. Attach reflux condenser with the flask and boil using an electric heating mantle or water bath for at least 2 h. Cool to room temperature, pour the reaction mixture into 125 ml of water and mix carefully.

3. Allow the reaction mixture to stand for 15 min and then filter the product under suction to remove an insoluble by-product.

4. Render the filtrate strongly acidic with concentrated hydrochloric acid, cool in ice-water and immediately filter off the precipitated product under suction.

5. Report Percentage yield and Melting point.

**Calculations:**

Molecular formula of Benzil = C14H10O2

Molecular weight of Benzil = 210 g/mol

Molecular formula of Phenytoin = C15H12N2O2

Molecular weight of Phenytoin = 252 g/mol

210 gm of Benzil is equivalent to 252 gm of Phenytoin

5.3 gm of Benzil is equivalent to X gm of Phenytoin

X= (252 × 5.3)/210 = 6.36 gm

Theoretical Yield = 6.36 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/6.36 gm

= ……………….%

**Result:**

Phenytoin was synthesized from Benzil and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is used an antiepileptic drug.

2. Used as an to control certain type of seizures, and to treat and prevent seizures that may begin during or after surgery to the brain or nervous system

**Cautions:**

1. Phenytoin may cause heart problems, including a slow heartbeat. Check with your doctor right away if you have chest pain, dizziness, or tiredness.

**Experiment No: 33 Aim: Synthesis of Paracetamol from para amino phenol**

**Requirements:**

**Apparatus:** Conical flask, beaker, measuring cylinder, Buchner funnel, filter paper, Magnetic stirrer, Melting point apparatus

**Chemicals:** Para amino phenol, acetic anhydride, concentrated sulphuric acid

**Principle:** The synthesis is based upon the acetylation reaction involving acetylation of primary amino group to acetamido group. Acetic anhydride undergoes rearrangement to give acetylating group and acetic acid. While, acetylation of para amino phenol involves two steps, first step is fast and second step is slow and is the rate determining step and involves the addition of nucleophile. Primary amino group are easily acetylated and hence, it is possible to acetylate this group easily in compounds containing both an amino group and a hydroxyl group.

**Chemical Reaction:**

**Mechanism:**



**Chemical Structure**



**IUPAC Name:** N-(4-hydroxyphenyl) acetamide

**Other Names:** Acetaminophen

**Molecular Formula:** C8H9NO2

**Molecular Weight:** 151 g/mol

**Appearance:** Colourless crystals or crystalline powder

**Melting Point:** 169 to 1710C

**Solubility:** Ethanol, water, acetone, chloroform

**Procedure:**

1. Place 6 g of [p-aminophenol](https://labmonk.com/synthesis-of-p-aminophenol-from-nitrobenzene) to a 100 ml cleaned and dried conical flask. Then, add to the flask 6.5 ml of [acetic anhydride](https://labmonk.com/synthesis-of-5-nitrosalisylic-acid-from-salicylic-acid) and 3–4 drops of concentrated [sulphuric](https://labmonk.com/synthesis-of-m-dinitrobenzene-from-nitrobenzene) acid carefully. The contents of the flask may be mixed thoroughly. Warm the mixture on a water-bath previously maintained at 60°C for about 20–25 min with constant stirring.

2. Allow the contents of the flask to attain room [temperature](https://labmonk.com/iodine-value-of-the-given-oil-or-fat), and pour it directly into a beaker having 100 ml of cold water (with a few chips of crushed ice) and stir it vigorously.

3. The crude product obtained is filtered onto a Buchner funnel using suction, wash it with cold water, drain well and dry the product and air-dry it or dry it in an electric oven maintained at 100°C. Report Percentage yield and Melting point.

**Calculations:**

Molecular formula of Para amino phenol = C6H7NO

Molecular weight of Para amino phenol = 109 g/mol

Molecular formula of Paracetamol = C8H9NO2

Molecular weight of Paracetamol = 151 g/mol

109 gm of Para amino phenol is equivalent to 151 gm of Paracetamol

6 gm of Para amino phenol is equivalent to X gm of Paracetamol

X= (151 × 6)/109 = 8.31 gm

Theoretical Yield = 8.31 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/8.31gm

= ……………….%

**Result:**

Paracetamol was synthesized from para amino phenol and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is used as an antipyretic and analgesic agent.

2. It is also found to be useful in diseases accompanied by pain, discomfort, and fever, for instance the common cold and other viral infections.

**Cautions:**

1. All apparatus must be perfectly dry.

2. Concentrated sulphuric acid should always be added with great caution.

**Experiment No: 34 Aim: Synthesis of Benzilic acid from Benzoin**

**Apparatus:** Conical flask, RBF, reflux condenser, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Benzoin, Conc. HNO3, Benzil, KOH, Ethanol, Conc. HCL, Benzene

**Principle:** In the first step alcohol group of benzoin is oxidized to ketone group forming benzil in presence of concentrated nitric acid. Nitration of aromatic ring is not occurring as sulphuric acid is totally absent in the whole process. In the second step on treatment with hydroxide ion molecular rearrangement occurs to 1,2-diketone forming potassium salt of α-hydroxy acid which on acidification yield α-hydroxy acid (benzilic acid).

**Chemical Reaction:**

**Step I:** Preparation of Benzil



**Step II:** Preparation of Benzilic acid



**Mechanism**

**Step I:**



**Step II:**



**Chemical Structure:**



**IUPAC Name:** 2-hydroxy-2,2-diphenylacetic acid

**Other Name:** Benzylic acid

Diphenylglycolic acid

Benzilic acid

**Molecular Formula:** C14H12O3

**Molecular Weight:** 228 g/mol

**Appearance:** white

**Melting Point:** 148-152 °C

**Solubility:** Sparingly soluble in water

**Procedure:**

**Step 1: Preparation of Benzil from Benzoin**

Heat a mixture of [benzoin](https://labmonk.com/different-chemical-test-to-analyze-the-crude-drug-of-benzoin) (20 g) and conc. [nitric acid](https://labmonk.com/synthesis-of-m-dinitrobenzene-from-nitrobenzene) (100 ml) in a test tube on boiling [water](https://labmonk.com/turbid-metric-determination-of-chloride) bath for about 1.5 hour until the evolution of oxides of [nitrogen](https://labmonk.com/synthesis-of-m-nitrophenol-from-nitrobenzene) ceases. Then pour the contents into ice cold water (300 – 400 ml) with continuous shaking. Filter the product, wash with cold water, and recrystallize from [ethanol](https://labmonk.com/determination-of-alcohol-soluble-extractive-value-of-ginger), m.p. 94-96oC.

**Step 2: Preparation of Benzilic acid from Benzil**

Dissolve [KOH](https://labmonk.com/acid-value-of-given-oil-fat) (35 g) in water (70 ml) by heating on hot plate and the cool to room temperature. Add 90 ml [rectified spirit](https://labmonk.com/synthesis-of-p-bromoacetanilide-from-acetanilide) and 35 g [benzil](https://labmonk.com/synthesis-of-phenytoin-from-benzil-and-urea) and reflux the content on a water bath till blue colour disappears (10-15 min). Pour the contents of the flask into a [porcelain dish](https://labmonk.com/synthesis-of-m-dinitrobenzene-from-nitrobenzene) and cool in ice, collect the colorless needles of [potassium](https://labmonk.com/determination-of-potassium-by-flame-photometry) benzilate. Dissolve the solid in 350 ml hot water and acidify with [HCl](https://labmonk.com/determination-of-ash-value-of-given-sample) slowly. Collect the solid benzillic acid and recrystallize from hot benzene. Report the practical yield, percentage yield and melting point.

**Calculations:**

Molecular formula of benzoin = C14H12O2

Molecular weight of benzoin = 212 g/mole

Molecular formula of  benzilic acid = C14H12O3

Molecular weight of benzilic acid = 228 g/mole

212 g of Benzoin is equivalent to 228 g of Benzilic acid

20 g of Benzoin is equivalent to X g of Benzilic acid

X= (228 × 20)/212 = 21.5 gm

Theoretical Yield = 21.5gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/21.5gm

= ……………….%

**Result:**

Benzilic acid was synthesized from Benzoin and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is used in organic synthesis, as a base point for preparation of glycollate pharmaceuticals and some hallucinogenic (deliriant) drugs.

2. Used to determine zirconium and to get benzophenone.

**Cautions:**

1. If ingested, seek medical advice immediately and show the container or the label.

2. Avoid contact with skin and eyes. Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area.

**Experiment No: 35 Aim: Synthesis of 2 Phenylindole from Phenyl hydrazine**

**Requirements**

**Apparatus:** Conical flask, RBF, reflux condenser, water bath, beaker, measuring cylinder, Buchner funnel, filter paper

**Chemicals:** Acetophenone, Phenyl hydrazine, Glacial acetic acid, Ethanol, Phosphoric acid

**Principle:** Aryl hydrazones are formed from a condensation reaction of an aryl hydrazine and an aldehyde or ketone. Here phenyl hydrazine condenses with acetophenone to produce acetophenone phenyl hydrazone. Finally by Fischer indole synthesis the aryl hydrazone (acetophenone phenyl hydrazone) converts into the indole (2-phenylindole) in the presence of an acid catalyst.

**Chemical Reaction:**



**Mechanism**

**Step I**



**Step II**



**Chemical Structure:**



**IUPAC Name:** 2-phenyl-1H-indole

**Other Name:** 1H-Indole, 2-phenyl

2 Phenylindole

**Molecular Formula:** C14H11N

**Molecular Weight:** 193 g/mol

**Appearance:** Off-white to beige or slightly green

**Melting Point:** 188-190 °C

**Solubility:** almost transparency in Methanol

**Procedure:**

1. Prepare acetophenone phenyl hydrazone by boiling a mixture of 20 g (0.167 mol) of acetophenone and 18 g (0.167 mol) of phenyl hydrazine carefully with ethanol 60 ml and a few drops of glacial acetic acid.

2. Filter the cold reaction mixture, wash the solid with dilute hydrochloric acid followed by about 12 ml of cold rectified spirit.

3. Recrystallise a small portion from ethanol and thus obtain a sample of pure acetophenone phenyl hydrazone as a white solid, m.p. 106 °C.

3. Place 28 g of the crude phenyl hydrazone in a 250 ml beaker containing 180 g of polyphosphoric acid. Heat on a boiling water bath, stir with a thermometer and maintain at 100-120 °C for 10 min (the reaction is exothermic).

4. Add 450 ml of cold water and stir well to complete solution of the polyphosphoric acid. Filter at the pump and wash well with water. Boil the crude solid under the reflux along with 300 ml of rectified spirit, add a little decolourising charcoal and filter through a preheated Buchner funnel. Wash the residue with 40 ml of hot rectified spirit.

5. Cool the combined filtrates to room temperature, filter off the 2-phenylindole and wash it three times with 10 ml portions of cold alcohol. Report the practical yield, percentage yield and melting point.

**Calculations:**

Molecular formula of phenyl hydrazine = C6H8N2

Molecular weight of phenyl hydrazine = 108 g/mole

Molecular formula of 2-phenylindole = C14H11N

Molecular weight of 2-phenylindole = 193 g/mole

108 g of phenyl hydrazine is equivalent to 193 g 2-phenylindole  
18 g of phenyl hydrazine is equivalent to X g 2-phenylindole

X= (193 × 18)/108 = 32.17gm

Theoretical Yield = 32.17gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/32.17gm

= ……………….%

**Result:**

2-Phenylindole was synthesized from Phenyl hydrazine and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is used as a reactant in difluorohydroxylation reactions and Mannich-type reactions.

2. 2-Phenylindole used as reactant for preparation of organic light emitting diodes (OLEDs), anti-inflammatory agents, antibacterial, antifungal agents and fluorescent probes.

**Cautions:**

1. Wear protective clothing/eye protection/face protection.

2. If in eyes rinse cautiously with water for several minutes.

**Experiment No: 36 Aim: Synthesis of Benzocaine from p-nitro benzoic acid**

**Requirements:**

**Apparatus:** Round Bottom Flask, Reflux Condenser set, beaker, measuring cylinder, Buchner funnel, filter paper, Melting point apparatus

**Chemicals:** p-Nitrobenzoic acid, Conc. HCl,  Glacial acetic acid, p-amino benzoic acid, ethanol, Sodium carbonate

**Principle:** In step 1 involves the reduction of aromatic para-nitrobenzoic acid by tin and hydrochloric acid to para-amino benzoic acid and in step 2 esterification of para-amino benzoic acid by sulphuric acid and ethanol to give benzocaine called as Fischer esterification.

**Chemical Reaction:**

**Step 1:**



**Step 2:**



**Mechanism:**

**Step 1:**



**Step 2:**



**Chemical Structure:**



Benzocaine

**IUPAC Name:** Ethyl 4-aminobenzoate

**Molecular Formula:** C9H11NO2

**Molecular Weight:** 165 g/mol

**Appearance:** white to off-white crystalline powder

**Melting Point:** 90 to 920C

**Solubility:** Ethanol, chloroform, ethyl ether

**Procedure:**

**Step 1**: **Preparation of p-amino benzoic acid**

1. Take 15 g (0.09 mol) of p-nitrobenzoic acid in a round-bottomed flask (RBF) fitted with a reflux condenser.

2. Then addition of 35 g (0.295 mol) of powdered tin and 75 ml of concentrated hydrochloric acid. Heat the mixture gently until the reaction commences, and remove the flame.

3. After about 20 min, most of the tin will have reacted to get clear solution. Allow to cool at room temperature then add the liquid into a beaker; wash the residual tin by decantation with 15 ml of water, and add the washings to the contents of the beaker.

4. Add concentrated ammonia solution until the solution is just alkaline to litmus and heat it again the suspension of precipitated hydrated tin oxide on a water bath for 20 min. Add 10 g of filter-aid (‘Celite’), stir well, filter at the pump and wash with hot water. Transfer the filter cake to a beaker, heat on a water bath with 200 ml of water to ensure extraction of the product and filter it again get the solution.

5. Heat the filtrate mixture and washings until the volume has been reduced to get 150-200 ml: filter off the precipitates.

**Step 2**: **Preparation of ethyl p-aminobenzoate (esterification of p-aminobenzoic acid)**

**1.** Take 50-80 ml of ethanol in a 250 ml two-necked round bottom flask attached with a double surface reflux condenser. Add, 12 g (0.088 mol) of p-aminobenzoic acid and heat the mixture under reflux for 2 hours. Upon cooling, to get solid mass of ethyl p-aminobenzoate.

2. Pour the hot solution into cold water and add sodium carbonate carefully to get the clear solution until it is neutral to litmus. Filter off the precipitated product and dry it. Report percentage yield and melting point.

**Calculations:**

Molecular formula of p-nitrobenzoic acid = C7H5NO4

Molecular weight of p-nitrobenzoic acid = 167 g/mol

Molecular formula of Benzocaine = C9H11NO2

Molecular weight of Benzocaine = 165 g/mol

167 gm of p-nitrobenzoic acid is equivalent to 165 gm of Benzocaine

15 gm of p-nitrobenzoic acid is equivalent to X gm of Benzocaine

X= (165 × 15)/167 = 14.82 gm

Theoretical Yield = 14.82 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/ 14.82 gm

= ……………….%

**Result:**

Benzocaine was synthesized from p-nitrobenzoic acid and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is used as a local anesthetic agent.

2. used short term to relieve pain from minor mouth problems (such as toothache, canker sores, sore gums/throat, and mouth/gum injury).

**Experiment No: 37 Aim: Synthesis of 7-Hydroxy-4-Methyl Coumarin from Resorcinol**

**Requirements:**

**Apparatus:** Conical Flask, beaker, measuring cylinder, Buchner funnel, filter paper, Heating mantle, Melting point apparatus

**Chemicals:** Polyphosphoric acid, resorcinol, ethyl acetoacetate

**Principle:** This synthesis is an example of Beckmann’s rearrangement which involves reaction in between resorcinol with beta keto ester in the presence of acidic reagents like sulphuric acid, phosphorous pentachloride and thionyl chloride. The mechanism is transfer of proton from the acidic catalyst to the carbonyl group of beta keto ester which results in a reduction of electron density from the carbonyl carbon.

**Chemical Reaction:**



**Mechanism:**



**Chemical Structure:**



**IUPAC Name:** 7-hydroxy-4-methylchromen-2-one

**Other Names:** 4-methylumbelliferone

Hymecromone

**Molecular Formula:** C10H8O

**Molecular Weight:** 176 g/mol

**Appearance:**  White to light yellow to light beige

**Melting Point:** 240 to 2420C

**Solubility:** Ethanol, acetic acid, alkali solution, ammonia

**Procedure:**

1. Take 160 gm of Polyphosphoric acid to a solution of 1.1 gm of resorcinol in 1.3 gm of ethyl acetoacetate in conical flask or beaker. Stir immediately the mixture and heat at 70-800C.

2. Then pour the solution into ice water. Filter off the solid, wash with cold water, dry it. Report percentage yield and melting point.

**Calculations:**

Molecular formula of Resorcinol = C6H6O2

Molecular weight of Resorcinol = 110 g/mol

Molecular formula of 7-Hydroxy-4-Methyl Coumarin = C10H8O

Molecular weight of 7-Hydroxy-4-Methyl Coumarin = 176 g/mol

110 gm of Resorcinol is equivalent to 176 gm of 7-Hydroxy-4-Methyl Coumarin

2 gm of Resorcinol is equivalent to X gm of 7-Hydroxy-4-Methyl Coumarin

X= (176 × 2)/110 = 3.2 gm

Theoretical Yield = 3.2 gm

Practical Yield = ……...gm

Percentage yield = (Practical yield×100)/Theoretical yield

= (……gm × 100)/ 3.2 gm

= ……………….%

**Result:**

7-Hydroxy-4-Methyl Coumarin was synthesized from Resorcinol and submitted.

|  |  |
| --- | --- |
| Name of Compound | ……………….. |
| Molecular formula |  |
| Molecular Weight | ………..gm |
| Theoretical Yield | ………..gm |
| Practical Yield | ………..gm |
| Percentage yield | ………..% |
| Melting Point | ………..0C |

**Uses:**

1. It is commonly clinically used as a choleretic drug.

2.  It is used commercially as laser dye.

**Cautions:**

1. Wash face, hands and any exposed skin thoroughly after handling.

2. Wear protective gloves/protective clothing/eye protection/face protection.

3. Use only outdoors or in a well-ventilated area