PROJECT NO.-1

Aim

To prepare a sample of cuprammonium rayon threads from filter paper.

Apparatus Required

a) Conical flask (preferably 250 ml)
b) Funnel
c) Glass rod
d) Beaker (preferably 250 ml)
e) Water bath
f) Filter paper (Whatman paper or ordinary filter paper sheets. Preferably, Whatman)

Chemicals Required

a) $CuSO_4$
b) NaOH solution
c) Liquor ammonia solution
d) Dilute H_2SO_4
e) Whatman Paper
f) Distilled H_2O

Background

Rayon is a synthetic fiber produced from cellulose. Developed in an attempt to produce silk chemically, it was originally called **artificial silk** or **wood silk**. Rayon is a **regenerated fiber**, because cellulose is converted to a liquid compound and then back to cellulose in the form of fiber. For example, cuprammonium rayon is made by dissolving cellulose in an ammoniacal copper sulphate solution.
The characteristics of rayon fibers are:

- They are highly absorbent,
- Soft and comfortable,
- Easy to dye &
- Drape well.

Introduction

Cellulose is nature's own giant molecule. It is the fibrous material that every plant from seaweed to the sequoia makes by baking glucose

molecules in long chains; the chains are bound together in the fibers that give plants their shape and strength. Wood has now become the main source of cellulose. Since it contains only 40% to 50% cellulose, the substance must be extracted by '**pulping**'. The logs are flaked, and then simmered in chemicals that dissolve the tarry lignin, resins and minerals. The remaining pulp, about 93% cellulose, is dried and rolled into sheets-raw material for paper, rayon and other products.
It can be obtained in 2 ways:
1. **Viscose Process:** Cellulose is soaked in 30% caustic soda solution for about 3 hrs. The alkali solution is removed and the product is treated with CS_2. This gives cellulose xanthate, which is dissolved in NaOH solution to give viscous solution. This is filtered and forced through a spinneret into a dilute H_2SO_4 solution, both of which harden the gum-like thread into rayon fibers. The process of making viscose was discovered by C.F.Cross and E.J.Bevan in 1891.
2. **Cuprammonium Rayon:** Cuprammonium rayon is obtained by dissolving pieces of filter paper in a deep blue solution containing tetra-ammine cupric hydroxide. The latter is obtained from a solution of copper sulphate. To it, NH_4OH solution is added to precipitate cupric hydroxide, which is then dissolved in excess of NH_3.

Reactions:

$CuSO_4 + 2NH_4OH \rightarrow Cu(OH)_2 + (NH_4)_2SO_4$

Pale blue ppt

$Cu(OH)_2 + 4NH_4OH \rightarrow [Cu(NH_3)_4](OH)_2 + 4H_2O$

$[Cu(NH_3)_4](OH)_2$ + pieces of filter paper left for 10-15 days give a viscous solution called **VISCOSE**.

Procedure

A. Preparation of Schweitzer's Solution:

a) Way 20g of $CuSO_4.5H_20$.
b) Transfer this to a beaker having 100ml distilled water and add 15ml of dilute H_2SO_4 to prevent hydrolysis of $CuSO_4$.
c) Stir it with a glass rod till a clear solution is obtained. Add 11ml of liquor ammonia drop by drop with slow stirring.
The precipitate of cupric hydroxide is separated out.
d) Filter the solution containing cupric hydroxide through a funnel with filter paper.
e) Wash the precipitate of cupric hydroxide with water until the filtrate fails to give a positive test for sulphate ions with barium chloride solution.
f) Transfer the precipitate to a beaker that contains 50ml of

liquor ammonia or wash it down the funnel. The precipitate when dissolved in liquor ammonia gives a deep blue solution of tetra-ammine cupric hydroxide. This is known as SCHWEITZER'S SOLUTION.

B. Preparation of Cellulose material

a) After weighing 2g of filter paper divide it into very fine pieces and then transfer these pieces to the tetra-ammine cupric hydroxide solution in the beaker.

b) Seal the flask and keep for 10 to 15 days, during this period the filter paper is dissolved completely.

C. Formation of Rayon Thread

a) Take 50ml of distilled water in a glass container. To this add 20ml of conc H_2SO_4 drop by drop. Cool the solution under tap water. In a big glass container pour some of the solution.

b) Fill the syringe with cellulose solution prepared before.

c) Place the big glass container containing H_2SO_4 solution produced before in ice (the reaction being spontaneous results in excess release of energy in the form of heat which makes the fibers weak and breaks them).

d) Immerse the tip of the syringe in the solution and press gently. Notice the fibers getting formed in the acid bath. Continue to move your hand and keep pressing the syringe to extrude more fibers into the bath.

e) Leave the fibers in solution till they decolorize and become strong enough.

f) Filter and wash with distilled water.

Precautions

a) Addition of excess NH_3 should be avoided.

b) Before taking the viscose in the syringe make sure that it does not contain any particles of paper, otherwise, it would clog the needle of the syringe.

c) Addition of NH_3 should be done in a fume cupboard and with extreme care. The fumes if inhaled may cause giddiness.

d) Use a thick needle otherwise the fibers won't come out.

Bibliography

- *Chemistry (Part I) – Textbook for Class XII*; National Council of Educational Research and Training
- *Concepts of Physics 2* by H C Verma; Bharti Bhawan

PROJECT NO.-2

AIM

To analyse some fruits & vegetables juice for the contents present in them.

INTRODUCTION

Fruits and vegetable are always a part of balanced diet. That means fruits vegetables provide our body the essential nutrients, i.e. Carbohydrates, proteins, vitamins and minerals. Again their presence in these is being indicated by some of our general observations, like -freshly cut apples become reddish black after some time. Explanation for it is that iron present in apple gets oxidixed to iron oxide. So, we can conclude that fruits and vegetables contain complex organic compounds, for e.g., anthocin, chlorophyll, esters(flavouring compounds), carbohydrates, vitamins and can be tested in any fruits or vegetable by extracting out its juice and then subtracting it to various tests which are for detection of different classes of organic compounds. Detection of minerals in vegetables or fruits means detection of elements other than carbon, hydrogen and oxygen.

MATERIAL REQUIRED

- Test Tubes
- Burner
- Litmus paper
- Laboratory reagents
- Various fruits
- Vegetables juices

CHEMICAL REQUIREMENTS

- pH indicator
- Iodine solution
- Fehling solution A and Fehling solution B
- Ammonium chloride solution

- Ammonium hvdroxide
- Ammonium oxalate
- Potassium sulphocynaide solution

PROCEDURE

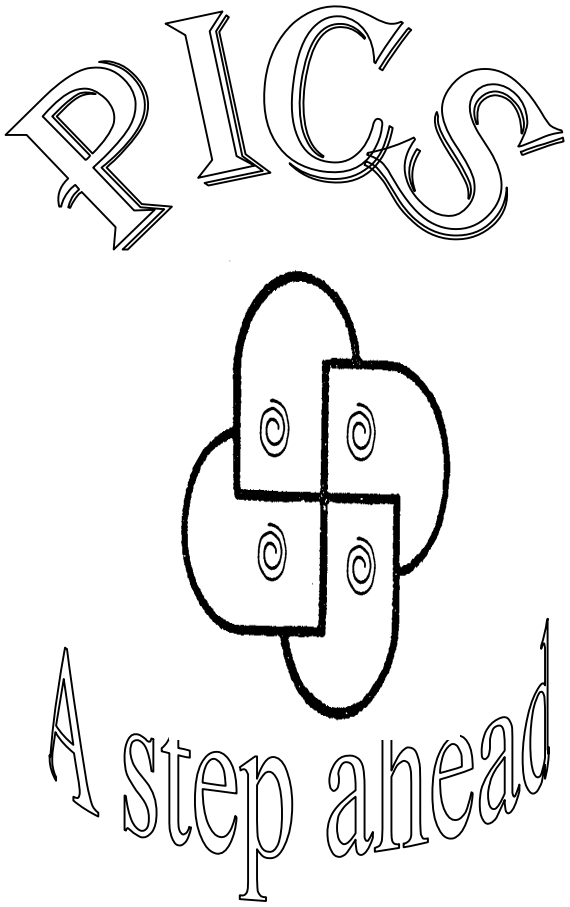
The juices are made dilute by adding distilled water to it, in order to remove colour and to make it colourless so that colour change can be easily watched and noted down. Now test for food components are taken down with the solution.

TEST, OBSERVATION & INFERENCE

Test	Observation	Inference
ORANGE TEST:		
Test for acidity:		
Take 5ml of orange juice in a test tube and dip a pH paper in it. If pH is less than 7 the juice is acidic else the juice is basic.	The pH comes out to be 6.	Orange juice is acidic.
Test for Startch:		
Take 2 ml of juice in a test tube and add few drops of iodine solution. It turns blue black in colour than the starch is present.	Absence of blue black in colour.	Orange juice is acidic.
Test for Carbohydrates (FEHLING'S TEST):		
Take 2 ml of juice and 1 ml of fehling solution A & B and boil it. Red precipitates indicates the presence of producing sugar like maltose, glucose , fructose & Lactose.	No red coloured precipitates obtained.	Carbohydrates absent.
Test for Iron:		
Take 2 ml of juice add drop of conc. Nitric acid. Boil the solution cool and add 2-3 drops of potassium sulphocyanide solution .Blood red colours shows the presence of iron.	Absence of blood red colour.	Iron is absent.
Test for Calcium:		
Take 2 ml of juice add Ammonium chloride and ammonium hydroxide solution. Filter the solution and to the filterate add 2 ml of Ammonium Oxalate solution. white ppt or milkiness indicates the presence of calcium.	Yellow precipitate is obtained.	Calcium is present.

CONCLUSION

From the table given behind it can be conducted that most of the fruits & vegetable contain carbohydrate & vegetable contain carbohydrate to a small extent. Proteins are present in small quantity. Therefore one must not only depend on fruits and vegetables for a balance diet.



PROJECT NO.-3

PREPARATION OF ASPIRIN AND ACETAMINOPHEN

OBJECT

To synthesize some common pain relievers: aspirin and acetaminophen.
To determine the purity of the aspirin or acetaminophen.

THEORY

Aspirin, acetylsalicylic acid, was first synthesized in 1893 by Felix Hofmann, a chemist for the German firm of Bayer. This compound had the medicinal properties of salicylic acid, an extract of willow bark, without the unpleasant taste or the high degree of irritation of the mucous membranes lining the mouth, gullet, and stomach.
Aspirin is both an organic ester and an organic acid. It is used extensively in medicine as a pain killer (analgesic) and as a fever-reducing drug (antipyretic). When ingested, acetylsalicylic acid remains intact in the acidic stomach, but in the basic medium of the upper intestinal tract, it hydrolyzes forming the salicylate and acetate ions. However, its additional physiological effects and biochemical reactions are still not thoroughly understood.
Aspirin (molar mass of 180.2 g/mol) is prepared by reacting salicylic acid (molar mass of 138.1 g/mol) with acetic anhydride (molar mass of 102.1 g/mol). Aspirin is a weak monoprotic acid.
Acetaminophen is an amide, a compound that is a derivative of ammonia that has been reacted with an acidic substance, in this case, acetic acid. Acetaminophen acts as a fever reducer and pain reliever. It can be found in several analgesic preparations, such as Tylenol, some of which may contain other ingredients such as caffeine and buffers.
Qualitatively, the purity of an aspirin or acetaminophen sample can be determined from its melting point. The melting point of a substance is essentially independent of atmospheric pressure, but it is always lowered by the presence of impurities (a colligative property of pure substances). The degree of lowering of the melting point depends on the nature and the concentration of the impurities.

SAFETY PRECAUTIONS

Wear safety glasses or goggles at all times in the laboratory.
Acetic anhydride is corrosive and its vapor is irritating to the respiratory system. Avoid skin contact and inhalation of the vapors. In the event of skin contact, rinse well with cold water. If the vapors are inhaled, move to an area where fresh air is available.
Sulfuric acid is corrosive. Avoid skin contact. In the event of skin contact, rinse well with cold water.
p-aminophenol is harmful by inhalation and by contact with the skin. In the event of skin contact, rinse well with cold water. If the vapors are inhaled, move to an area where fresh air is available.

PROCEDURE

OPTION 1: THE PREPARATION OF ASPIRIN

Materials Needed

Salicylic acid
Acetic anhydride
Sulfuric acid, concentrated
Ethanol
Dropper
Erlenmeyer flask, 125-mL
Beakers, 2 400-mL, 100-mL, 10 or 20-mL
Graduated cylinders, 10-mL, 25-mL
Watch glass
Stirring rod
Vial to hold aspirin sample
Ring stand
Clamp (to hold 125-mL Erlenmeyer flask)
Buchner funnel
Filter paper to fit Buchner funnel
Vacuum filtration flask
Rubber tubing for vacuum flask

Ice

Thermometer, 110°C

Melting point capillary tube
Melting point apparatus
dropper
Disposable IR card
Optional: rubber gloves

Procedure

Weigh out 2.0 g of salicylic acid. Place it in a 125-mL Erlenmeyer flask.
Add 5 mL of acetic anhydride. Swirl the flask to wet the salicylic acid crystals. Add 5 drops of concentrated sulfuric acid, H_2SO_4, to the mixture.
Gently heat the flask in a boiling water bath for about 10 minutes.
Remove the flask from the hot water bath and add 10 mL of deionized ice water to decompose any excess acetic anhydride. Chill the solution in an ice bath until crystals of aspirin no longer form, stirring occasionally to

decompose residual acetic anhydride. If an "oil" appears instead of a solid, reheat the flask in the hot water bath until the oil disappears and again cool.
Set up a vacuum filtration apparatus. Wet the filter paper in the Buchner funnel with 1-2 mL of distilled water. Turn on the water aspirator. Decant the liquid onto the filter paper, minimizing any transfer of the solid aspirin. If some aspirin is inadvertently transferred to the filter, that will not cause any difficulty.
Add 15 mL of cold water to the flask, swirl, and chill again. Pour the liquid and the crystals of aspirin onto the filter paper. Repeat until the transfer of the crystals to the vacuum filter is complete. Wash the aspirin crystals on the filter paper with 10 mL of ice water.
Maintain the vacuum to dry the crystals as best possible.
If aspirin forms in the filtrate in the vacuum flask, transfer the filtrate and aspirin to a beaker, chill in an ice bath, and vacuum filter as before, using a new piece of filter paper. Dispose of the filtrate in the sink.
Determine the mass of the crude aspirin crystals.

Recrystallization of the Aspirin.

The major impurity in aspirin is salicylic acid. It can be removed by a recrystallization.
Place the aspirin crystals in a 100-mL beaker. Add 8 mL of ethanol and 25 mL of water.

Warm the mixture in a 60°C water bath (no flame, use a hot plate or a hot water bath). Warm the mixture until the aspirin dissolves. (If the solid does not dissolve after heating, consult with your instructor.)
Cover the beaker with a watch glass, remove it from the heat, and set it aside to cool slowly. Set the beaker in an ice bath. Beautiful needle-like crystals of acetylsalicylic acid form.
Collect the aspirin by vacuum filtration. Wash the crystals with two 10-mL volumes of ice water. Maintain the vacuum to air dry the aspirin. If time does not permit, place the filter paper and aspirin sample on a watch glass and allow them to air-dry. The time for air-drying the sample may require that it be left with your instructor until the next laboratory period.
Transfer the dry aspirin crystals to a pre-weighed sample container or vial. Determine the mass of the aspirin crystals.

Determine the Melting Point of the Aspirin Sample

Fill a capillary melting point tube to a depth of 0.2 cm with the recrystallized aspirin.
Place the capillary tube in the melting point apparatus. Determine its melting point. (Your instructor will demonstrate the use of this apparatus.)

Pure aspirin melts at 135°C.

The aspirin sample should be labeled with your name, the mass of the aspirin, the percent yield, and its melting point.
NOTE: Don't use your aspirin for a headache! Its purity is not assured.

Verification of Aspirin

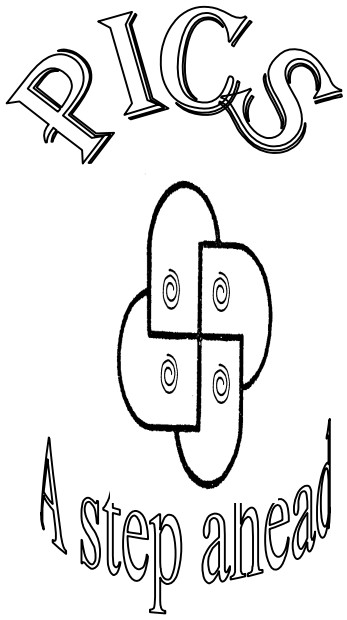
Place a small amount of your aspirin sample (about 0.10 g) in a 10-mL or 20-mL beaker. Add approximately 2 mL of ethanol. Stir or swirl to dissolve. If necessary, warm the mixture slightly on a hot plate to assist in solution.
CAUTION: Ethanol is flammable. Do not allow it to boil.
Place one or two drops of the acetaminophen solution on a disposable IR card. Allow the ethanol to evaporate.

Run an IR of your aspirin.
Compare your IR spectrogram with the standard IR spectra of salicylic acid and acetylsalicylic acid (aspirin). How do they agree?

OPTION 2: THE PREPARATION OF ACETAMINOPHEN

Materials Needed

p-aminophenol
Acetic anhydride
Phosphoric acid, concentrated
Ethanol
Dropper
Erlenmeyer flask, 125-mL
Beakers, 2 400-mL, 100-mL, 10 or 20-mL
Graduated cylinders, 10-mL, 25-mL
Watch glass
Stirring rod
Vial to hold aspirin sample
Ring stand
Clamp (to hold 125-mL Erlenmeyer flask)
Buchner funnel
Filter paper to fit Buchner funnel
Vacuum filtration flask
Rubber tubing for vacuum flask
Ice
Melting point capillary tube
Melting point apparatus
dropper
Disposable IR card
Optional: rubber gloves



Procedure

Fill a 400-mL beaker about half full with water. Place the beaker and water on a hot plate and bring to a boil.
Weigh out 1.5 g of p-aminophenol and transfer it into a 125-mL Erlenmeyer flask. (Avoid contact with skin. You may wish to wear gloves.)
Add 25 mL of water. Add 20 drops of concentrated phosphoric acid, H_3PO_4, and swirl the flask until all of the amine dissolves. If not, add a few more drops of phosphoric acid.
Turn off the hot plate. Place the flask in the hot water. Carefully add 2 mL of acetic anhydride to the flask. Leave the flask in the warm water for 10 minutes.

Remove the flask and place it an ice-water bath. Stir the mixture to crystallize the acetaminophen. You may need to scratch the walls of the flask to start the crystallization. If no crystals appear, add a small seed of acetaminophen to start the crystal formation. Allow the flask to stay in the ice-water bath for 30 minutes.
Collect the crystals in a Buchner funnel using vacuum filtration.
Wash the crystals with 10 mL of cold water. Allow the crystals to dry.
Determine the mass of the crude acetaminophen.

Recrystallization of the Acetaminophen

Place the crude acetaminophen in a 100-mL beaker. Add 20 mL of water and heat on a hot plate until the crystals dissolve. If the solution boils and crystals remain, add another 10 mL of water.
Remove the beaker and allow the solution to cool. When crystals begin to appear, place the beaker in an ice bath for 20 minutes. If no crystals appear, scratch the inside walls of the beaker.
Collect the crystals using the Buchner filtration apparatus. Wash with 10 mL of cold water.
Transfer the filter paper and crystals to a watch glass and let dry.
Determine the mass of the purified acetaminophen.

Determine the Melting Point of the Acetaminophen Sample

Fill a capillary melting point tube to a depth of 0.2 cm with the recrystallized acetaminophen.
Place the capillary tube in the melting point apparatus. Determine its melting point. (Your instructor will demonstrate the use of this apparatus.)

The melting point of acetaminophen is 169-171°C. p-aminophenol melts at 189-190°C.

The acetaminophen sample should be labeled with your name, the mass of the acetaminophen, the percent yield, and its melting point.

NOTE: Don't use your acetaminophen for a headache! Its purity is not assured.

Verification of Acetaminophen

Place a small amount of your acetaminophen sample (about 0.10 g) in a 10-mL or 20-mL beaker. Add approximately 2 mL of ethanol. Stir or swirl to dissolve. If necessary, warm the mixture slightly on a hot plate to assist in solution. CAUTION: Ethanol is flammable. Do not allow it to boil.

Place one or two drops of the acetaminophen solution on a disposable IR card. Allow the ethanol to evaporate. Run an IR of your acetaminophen.

Compare your IR spectrogram with the standard IR spectra of p-aminophenol and acetaminophen. How do they agree?

Report Sheet
PREPARATION OF ASPIRIN AND ACETAMINOPHEN

Name _____ Course/Section _____

Partner (If applicable) _____ Date _____

DATA

A. Preparation of Aspirin

Mass of salicylic acid _____ g

Mass of crude aspirin _____ g

Mass of purified aspirin _____ g

Theoretical yield of aspirin (show calculation below) _____ g

Percent yield of aspirin (show calculation below) _____ %

Melting point of aspirin _____°C

Does the infrared spectrum of your aspirin sample confirm the identity of your product? (Attach the IR spectrograph to your report sheet.)

Based on your melting point and infrared data, the aspirin sample is (check one)

[] crude [] purified

B. Preparation of Acetaminophen

Mass of p-aminophenol _____ g

Mass of crude acetaminophen _____ g

Mass of purified acetaminophen _____ g

Theoretical yield of acetaminophen (show calculation below) _____ g

Percent yield of acetaminophen (show calculation below) _____ %

Melting point of acetaminophen _____°C

Does the infrared spectrum of your acetaminophen sample confirm the identity of your product? (Attach the IR spectrograph to your report sheet.)

Based on your melting point and infrared data, the acetaminophen sample is (check one)

[] crude [] purified