

## Exp.1: Water pollution treatment

### Pollution impurities of water

- 1- **Dissolved impurities:** Consist of some inorganic salts, dissolved gases, and ions of some poisonous elements, also some dissolved organic materials which generates from the remains of died animals, humans and plants, which decomposes in water in addition to the wastes of houses and factories like dyes and detergents.
- 2- **Suspended impurities:** consist of suspended inorganic impurities like sludge, soils, and organic impurities like oil and crude oil drops spots or remains of died animals and plants and it's wastes.
- 3- **Bacterial impurities:** consist of different types of bacteria and microorganism.

### Water treatment:

**Waste water treatment:** Most of these wastes are organic and inorganic in addition to some suspended materials as mentioned before are undergo a systematical treatment to remove them from water, but in most cases the waste waters that drain from factories must be pretreated by equalizing acids and bases.

After these pretreatments there are three main treatments methods to collect these wastes.

- 1- **Mechanical method:** Consist of removal of large pieces which float and suspending by passing it on the large metal sieves then to sedimentation tanks finally filtering it in special tanks consist of layers of soil and stones.
- 2- **Chemical method:** Addition of some chemical compounds which helps in increasing sedimentation process like Alum (  $\text{Al}_2(\text{SO}_4)_3$  ) for chrome ion precipitant in leather tanning factories and lime  $\text{Ca}(\text{OH})_2$  for sedimentation of dissolved materials like phosphorus produce from detergents.
- 3- **Activated sludge method:** In this method an Aerobic bacteria is generated in sludge which digesting the organic wastes by using it as nutrition then oxidizing it in breathing to transfer to natural products like  $(\text{H}_2\text{O} \ \& \ \text{CO}_2)$  this process known as (Biological oxygen demand).



In this process gases like ammonia,  $\text{H}_2\text{S}$ ,  $\text{CH}_4$  were generated because of the bacterial decay of wastes by an aerobic bacteria, then  $\text{Cl}^-$  added to the water to kill and remove bacteria & microorganisms.

### **Practical experiment:**

#### **A) Determination of dissolved organic materials:**

- 1- Take a sample of 30g from the waste water.
- 2- Organic materials were extracted by 15ml of chloroform using separation funnel twice.
- 3- Evaporate the solvent gradually on a heating source inside the hood.
- 4- Weight the remaining organic materials.
- 5- Calculate the percentage of organic materials.

#### **B) Determination of suspended materials:**

- 1- Take six samples of waste water each one (5g).
- 2- Put them in the centrifuge for 5-10 minutes.
- 3- Discharge the clear water then dry the sediment in the oven at  $110^\circ\text{C}$ .
- 4- Weight each sample after drying then calculate the percentage.

#### **C) Semi-industrial method for waste water purification:**

##### **Procedure:**

- 1- Prepare chromatographic column with length 30cm and diameter 2.5cm.
- 2- Put inside it a mixture of activated charcoal and sand (1:3) percent and not more than 10cm length.
- 3- Add a specified volume and weight of waste water from the top of the column.
- 4- Take the discharged water from the bottom of the column.
- 5- Measure the refractive index for the waste water and discharged water and notice the difference.

## Exp 2: TESTING THE WATERS

*HOW GOOD IS THAT BOTTLED WATER AND  
HOW EFFECTIVE IS YOUR WATER FILTER?*

Bottled water is usually spring water from some natural spring. Generally, this water has been filtered through the earth and collects in underground reservoirs called aquifers, such waters will contain dissolved minerals that will contribute to its taste. If the water collects in aquifers where there is no decayed organic matter or nutrients, it is usually bacteria free. However, contaminants and bacteria can pass through the ground to contaminate the water.

One of the simplest, and the most common, water filtration systems is the water filtration pitcher. This type of pitcher consists of an upper reservoir with a filter cartridge attached at the bottom. The filter cartridge generally contains an ion exchange resin that removes most of the metallic elements in the water and activated carbon which removes chlorine and other constituents that may affect taste and odor.

**Natural water treatment:** The water comes from rivers or wells is a natural water it is treated by the following methods:

- 1) Water purification:** the process done by filtration, sedimentation and sterilization.
- 2) Hardness removal:** the process done by using different ion exchange resins or by using sedimentation processes by soda-lime (  $\text{Na}_2\text{CO}_3 + \text{Ca}(\text{OH})_2$  ) or by Phosphate treatment.
- 3) Silica removal:** This done by using Magnesium Oxide  $\text{MgO}$ .
- 4) Expulsion of Oxygen from water:** This process done by addition of antioxidant salts to water like chromate, silica, and phosphate salts or by addition of strong reduction agents like sodium sulfates which reacts with oxygen and remove it efficiently.

Most municipalities will clarify the water by sedimentation or filtration and treat the water with chlorine to kill any harmful bacteria. The normal processing does not remove dissolved minerals and salts.

In this experiment, you will compare the taste of some different waters and will determine the effectiveness of a water filtration system by **testing for dissolved solids, pH, hardness, iron, chlorine, lead and copper.**

**Determination of dissolved inorganic materials& Hardness:** This caused by dissolved minerals consist of Calcium and Magnesium salts mainly also Ferrate, Manganese ( $Mn^{2+}$ ) salts, strontium ( $Sr^{2+}$ ) and zinc ( $Zn^{2+}$ ), in small amounts these salts in water cause water hardness. The total dissolved solids and maximum contamination level is **500 mg per liter**.

Hard water will affect the action of soaps and will result in the buildup of white deposits and scale around water faucets and sinks.

**pH:** is an indication of acidity or alkalinity of the water. Ideally, water should be neutral with a pH of 7.

Acidic water will have a pH less than 7 and will have a sour taste as acidity increases. Basic or alkaline water will have a pH above 7 and will have a bitter taste as alkalinity increases. Neutral, or nearly neutral water has a pH of about 7 and should have no sour or bitter taste.

**Iron:** may be present in water as iron(II),  $Fe^{2+}$ , which can oxidize to iron(III),  $Fe^{3+}$ . It comes from natural deposits in the environment and from iron pipes in water systems. Iron will result in stains to laundry and porcelain and is more of a nuisance than a health hazard.

**Chlorine:** is the disinfectant most frequently used for water and wastewater treatment. Depending on the source of water in your locality, the amount of chlorine can vary from a slight amount that is hardly noticeable to a higher concentration that some people find objectionable. When chlorine is added to water, it forms **hydrochloric (HCl)** and **hypochlorous (HClO) acids**. The hypochlorous acid is responsible for the disinfectant action as well as a bleaching action.

Chlorine is also present in water combined with metals in the form of chlorides. When sodium is present in drinking water, chlorides in excess of 250 mg/L will result in a salty taste. The maximum allowable chloride concentration in drinking water is **250 mg/L**.

**Lead:** and its compounds are poisonous and accumulate in the bone structure when ingested. Accumulation of significant amounts of lead in the body may cause severe and permanent brain damage, convulsions, and death. Lead is not commonly found in surface waters as it is normally precipitated by many substances. There has been concern that

lead may be leached into drinking water from the tin-lead solder used in joining copper pipes.

**Copper:** concentrations in drinking water are usually very low and are not considered a health hazard. When copper concentrations exceed more than **1 mg/L**, it can impart a bitter taste to the water. Large oral doses of copper compounds can cause vomiting and eventually may cause liver damage.

**Arsenic:** and its compounds are poisonous. Arsenic compounds irritate the stomach severely and affect the heart, liver and kidneys. Arsenic is most likely found in well water rather than municipal water or bottled water and usually results from mining or metallurgical operations. The maximum amount of arsenic permitted in drinking water is **0.05 mg/L**.

### **Materials Needed**

Water to be used may be tap water, purified tap water, well water, or bottled spring water, and different brands of bottled water, pH meter with pH electrode.

### **Procedure**

It is imperative that you rinse all testing tubes, beakers, flasks, and bottles at least twice with distilled or deionized water.

#### **1-Testing for dissolved solids**

Obtain a clean, dry beaker. Using a graduated cylinder, measure 100 ml of the type of water you are testing into the beaker. Place the beaker of water on the hot plate and allow the water to evaporate.

After all the water has just evaporated, remove the beaker from the hot plate and allow it to cool.

Weigh the beaker and record its mass. (Note: If the beaker and residue weighs less than the empty beaker, clean out the beaker at the end of this procedure, dry it well, and reweigh it.)

Calculate the mass of the dissolved solids in mg. This is the **mass of dissolved solids in 100 ml of water**. Multiply by 10 to get the mass of dissolved solids per liter.

#### **2- Testing pH**

This procedure uses either a laboratory pH meter. The pH electrode must remain wet between tests to maintain its calibration.

Place approximately 20 mL of the type of water you are testing in the beaker.

### 3-Hardness

Fill the flask to the 100 ml mark with the type of water you are testing.

Add about 2 ml of Buffer Solution (Hydroxylamine hydrochloride, an organic amine, and buffered to a pH of 10.1) to the flask. Swirl to mix. The indicator is ero-chromoblak T, an organic dye, which reacts with calcium and magnesium ions to give a red colored complex. As the titrant, EDTA (ethylene diamine tetra acetic acid), is added, it reacts with the free calcium and magnesium ions, first, and then removing them from the dye. When the ions are removed from the indicator, the color changes from red to blue.

### 4-Chlorine

1-Prepare 0.1N of  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  in 250ml water.

2-Prepare 2.5% of KI.

3-Add 5 ml of KI to 50ml water.

4-Add 3 ml of 2N  $\text{H}_2\text{SO}_4$  to step 3.

5-Titrate the water sample against  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  solution.

6-Calculate the N and find the weight of chlorine.  $(N \cdot V)_{\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}} = (N \cdot V)_{\text{Cl}^-}$

### 5-Tasting water

All waters should be at the same temperature.

Obtain sufficient paper or plastic cups for the types of water being tasted. Test all the waters by drinking test then compare the tastes of each sample.

### 6- Refractive index values

Test all the waters by taking the refractive index of them and then compare the value of each sample.

## Exp.3: Extraction of plants oil

Plants seeds usually contains some amounts of oils in different percent which is useful in several fields like soap and paints industry or in cooking oils, mainly these oils extracted by one or both of the two methods (press method or solvent extraction), like sun flower seeds, cotton seeds, soybeans, peanut seeds, sesame, etc.

Historically, such oils have been extracted by wrapping seeds in cloth, and then using devices operated by stones and levers to exert pressure on them.

Because most press or expeller processes overheat the meal and leave too much of the high value oil in the seed cakes, methods of extracting the oil with solvents were

developed. Seeds (like soybeans) with low oil content are processed by solvent methods alone. In other cases, presses are used first to extract part of the oil; then solvents extract the oil that remains in the seeds. Because of their efficiency.

The basic technology of solvent extraction is simple, but great care should be taken in deciding whether and where it can be used. Solvent extraction of vegetable oils, which recovers more oil than earlier methods and leaves more usable meal, begins to be economically attractive where large quantities of seed can be processed (at least 200 tons per day for continuous-feed processes).

For laboratory purposes solvent extraction is done by using Soxhlet apparatus. Soxhlet extractor is a piece of laboratory apparatus invented in 1879 by Franz von Soxhlet. It was originally designed for the extraction of a lipid from a solid material. However, a Soxhlet extractor is not limited to the extraction of lipids. Typically, a Soxhlet extraction is only required where the desired compound has only a limited solubility in a solvent, and the impurity is insoluble in that solvent. If the desired compound has a high solubility in a solvent then a simple filtration can be used to separate the compound from the insoluble substance.

A schematic representation of a Soxhlet extractor:

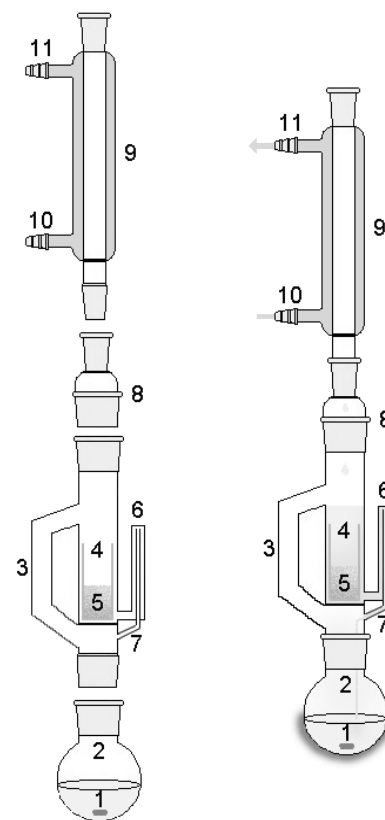
- 1: Stirrer bar    2: Still pot    3: Distillation path    4: Thimble    5: Solid  
6: Siphon top    7: Siphon exit    8: Expansion adapter  
9: Condenser    10: Cooling water in    11: Cooling water out

Normally a solid material is placed inside a thimble made from thick filter paper, which is loaded into the main chamber of the Soxhlet extractor. The Soxhlet extractor is placed onto a flask containing the extraction solvent.

The Soxhlet is then equipped with a condenser.

The solvent is heated to reflux. The solvent vapour travels up a distillation arm, and floods into the chamber housing the thimble of solid. The condenser ensures that any solvent vapour cools, and drips back down into the chamber housing the solid material.

The chamber containing the solid material slowly fills with warm solvent. Some of the desired compound will then



dissolve in the warm solvent. When the Soxhlet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle may be allowed to repeat many times, over hours or days. During each cycle, a portion of the non-volatile compound dissolves in the solvent. After many cycles the desired compound is concentrated in the distillation flask. The advantage of this system is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled.

After extraction the solvent is removed, typically by means of a rotary evaporator, yielding the extracted compound. The non-soluble portion of the extracted solid remains in the thimble, and is usually discarded.

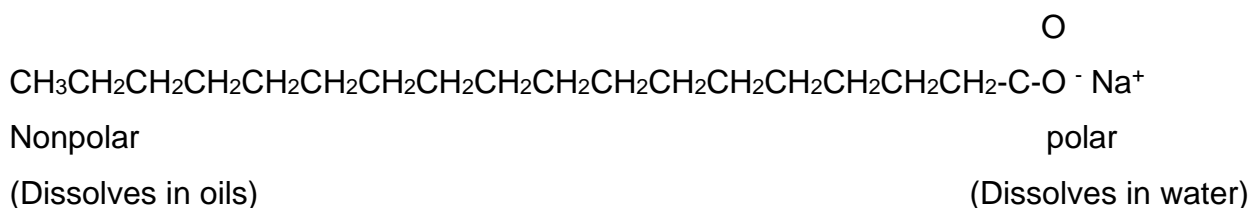
In this experiment any kind of mentioned seeds is used to extract its oil using solvents like (ether, petroleum ether, or chloroform).

### Procedure:

- 1- Weight around bottom flask and put 200ml of the petroleum ether (80-60) inside it.
- 2- Put 5g of the seeds in a thimble and put it inside the soxhlet chamber .
- 3- Fix the soxhlet apparatus as in the figure.
- 4- Heat the flask in a water bath or heater to a boiling point for a 60-75 minute and notice the solvent color change.
- 5- Distillate the solvent or evaporate the solvent then weight the flask which contains the extracted oil then calculate the percentage of extracted oil.

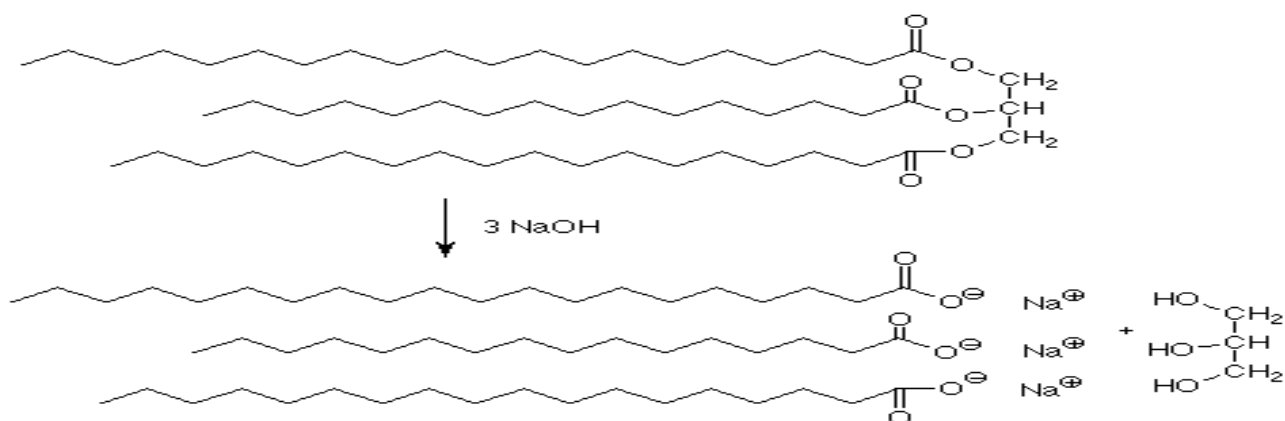
## Exp.4: The Synthesis and Properties of Soaps

A soap is the sodium or potassium salt of a long chain fatty acid. The fatty acid usually contains 12 to 18 carbon atoms. Solid soaps usually consist of sodium salts of fatty acids whereas liquid soaps usually are potassium salts of fatty acids. A soap, such as the sodium stearate you will be preparing in this experiment, consists of a polar end and a non-polar end.





Because like dissolves in like, the non-polar end (hydrophobic or water-fearing part) of the soap molecule can dissolve the greasy dirt, and the polar or ionic end (hydrophilic or water-loving part) of the molecule is attracted to water molecules. Therefore the dirt from the surface being cleaned will be pulled away and suspended in water. Thus soap acts as an emulsifying agent, a substance used to disperse one liquid (oil molecules) in the form of finely suspended particles or droplets in another liquid (water molecules). Treatment of fats or oils with strong bases such as lye (NaOH) or potash (KOH) causes them to undergo hydrolysis (saponification) to form glycerol and the salt of a long-chain fatty acid (soap).



Because soaps are the salts of strong bases and weak acids they should be slightly basic. If a soap is too basic it could cause damage to skin, surfaces to be cleaned, or clothes. We will test the basicity/ acidity of the soap you create.

Since the cleansing action of soaps depends upon the fact that they ionize readily in water, you can imagine what would happen if the ionic end lost its charge. The soap would no longer be attracted to water molecules and could no longer emulsify oil and dirt. This is just what happens in hard or acidic water. Hard water contains metal cations, such as  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ , which react with the charged ends of the soaps to form insoluble salts. The insoluble salts that  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  form with soap anions cause the gray precipitate commonly called **bathtub ring**.

In this experiment you will make a soap using a vegetable oil as your starting material. Then, you will compare the properties of the soap you made with those of a commercial soap and a commercial detergent.

**Purpose:** To synthesize a soap and compare its properties with the properties of a commercial soaps.

***Be careful with all glassware, they get very slippery when soapy and wet!***

**Procedure:**

**1- Preparation of the Sodium Soap:** Measure 12mL of vegetable oil or shortening into a 400mL beaker. Add 12mL of 5M NaOH. Stir the mixture **vigorously** with a glass rod and **gently** heat on a hot plate for 15 minutes or until it turns pasty.

***When the paste begins to form, stir very carefully to prevent frothing!***

The paste is a mixture of soap and glycerol.

After all the paste has formed, set the beaker on your bench to cool. When the paste mixture is cool, add 50mL of saturated NaCl to the paste mixture and stir in thoroughly, breaking up the chunks as you stir. The NaCl solution provides  $\text{Na}^+$  and  $\text{Cl}^-$  ions that bind to the polar water molecules, and help separate the water from the soap. This process is called **salting out** the soap.

After stirring the NaCl solution through the soap paste, filter off the soap mixture by suction filtration and wash the collected soap precipitate with 15mL of ice water. Continue the suction for an additional 10 minutes to help dry the soap.

## **2- Testing the Properties:**

First, make separate test solutions of your prepared soap, and commercial soap by dissolving about 0.1g of each in 20mL of deionized water.

**pH:** Test the pH of each test solution by touching a clean glass rod to the solution and transferring a drop to a piece of both red and blue litmus paper. Record your results.

**Cleaning Ability:** Test the different solutions ability to clean fat off a piece of glassware. Compare your soap results with the commercial soap and deionized water and record your results.

**Behavior in acidic water:** Hydronium ions attach to one end of soap molecules, destroying their cleaning ability by reducing their attraction for water molecules. (Which end of the soap molecule will hydronium ions be attracted to?) To test this, place 10ml of each of your test solutions and distilled water in clean separate test tubes, add 5 drops (3M) HCl to each test tube, shake well, and record your observations. Next add 10 drops of oil to each test tube and shake well. Note your observations immediately on the recording sheet, observe again after the solutions have sat for 5 minutes.

**Behavior in Hard Water:** Before you begin the next part, put a 400mL beaker half full of water on a hot plate to heat up. In this part, you will test the effects of hard water on soap's ability to act. Place 10mL of each of your test solutions into separate clean test tubes. Add 2ml of the hard water solution to each test tube, shake well and immediately observe the nature of the contents, looking for cloudiness, precipitates, films, etc. Record your

observations. Allow for the test tubes to stand undisturbed for 5 minutes, observe their condition now and record these observations also. Repeat the procedure using distilled water in place of the hard water solution.

## Exp.5: Preparation of powder laundry detergent (Tide)

**Detergent:** is a compound, or a mixture of compounds, intended to assist cleaning.

The term is often used to differentiate between soap and other chemical surfactants used for cleaning purposes.

Synthetic detergents were developed to overcome these limitations of soaps. Detergents are similar to soaps in having an ionic end and a nonpolar end. They have different structures, however, which make them less susceptible to forming insoluble  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  salts. Many detergents and a few soaps contain phosphates, which serve as bases to neutralize acidic water and also to form insoluble salts with the  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions. This prevents the reaction with soap that forms bathtub ring. However, soluble phosphate salts, like sodium phosphate, when released into rivers and lakes can cause explosive growths of algae. This can cause decay or eventual death of the aquatic ecosystem due to deoxygenation from the decomposition of dead algae. Because of this, phosphates in detergents have been outlawed in many places.

Detergents and soaps are used for cleaning because pure water can't remove oily, organic soiling. Soap cleans by acting as an emulsifier. Basically, soap allows oil and water to mix so that oily grime can be removed during rinsing. Detergents were developed in response to the shortage of the animal and vegetable fats used to make soap.

**Chemical powder laundry detergent composition:** is a mixture of chemical compounds including alkyl benzene sulfonates and fatty alcohol sulfates as surfactant, and other materials like:

**1-Surfactants,** It lower the surface tension of water, and lower the interfacial tension between oil and water by adsorbing at the liquid-liquid interface, essentially making it 'wetter' so that it is less likely to stick to itself and more likely to interact with oil and grease they are wetting agents that lower the surface tension of a liquid, allowing easier spreading. They could be produced easily from petrochemicals. Many surfactants can also assemble in the bulk solution into aggregates.

Some commonly encountered surfactants of each type include:

## **Ionic**

**Anionic** (based on sulfate, sulfonate or carboxylate anions)

- ☐ Sodium dodecyl sulfate (SDS), ammonium lauryl sulfate, and other alkyl sulfate salts
  - ☐ Sodium laureth sulfate, also known as sodium lauryl ether sulfate (SLES)
  - ☐ Alkyl benzene sulfonate
- ☐ Soaps, or fatty acid salts

**Cationic** (based on quaternary ammonium cations)

- ☐ Cetyl trimethylammonium bromide (CTAB) a.k.a. hexadecyl trimethyl ammonium bromide, and other alkyltrimethylammonium salts
  - ☐ Cetylpyridinium chloride (CPC)
  - ☐ Polyethoxylated tallow amine (POEA)
  - ☐ Benzalkonium chloride (BAC) ☐ Benzethonium chloride (BZT)

**Zwitterionic** (amphoteric)

- ☐ Dodecyl betaine
- ☐ Dodecyl dimethylamine oxide
- ☐ Cocamidopropyl betaine
- ☐ Coco amphoteric glycinate

## **Non-ionic**

- ☐ Alkyl poly(ethylene oxide)
- ☐ Copolymers of poly(ethylene oxide) and poly(propylene oxide) (commercially called Poloxamers or Poloxamines)
  - ☐ Alkyl polyglucosides, including: (Octyl glucoside, Decyl maltoside)
  - ☐ Fatty alcohols (Cetyl alcohol, Oleyl alcohol)
  - ☐ Cocamide MEA, cocamide DEA, cocamide TEA

**2-Builders:** Builders are key detergent components which remove the calcium and magnesium ions presents in hard water and in soils, thus lowering the concentration of surfactants necessary to perform the detergative action. Some builders also prevent the deposition of calcium and magnesium salts on fabrics and washing machines. Typical builders are **sodium carbonate, or sodium phosphates, or zeolites**.

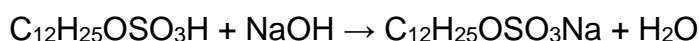
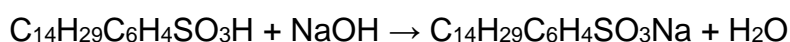
**3-Fillers:** these materials are added to enable the adjustment of the active matter in the detergent to the doses used. Filler products include, **sodium silicates, sodium citrates**.

**4-Bleaching agents:** Bleaching agents eliminate stubborn stains and ensure hygiene by killing bacteria through a chemical oxidation performed by a peroxygen generator, usually **sodium perborate**.

**5- Other additives:** These materials were added to the detergent for commercial purposes include color and perfume additives in addition to enzymes, abtibacterial, hand softeners, antioxidants, and activators to enhance the properties and activity of the detergent

**In this experiment we will:**

**Prepare alkyl benzene sulfonates and sodium lauryl sulfate as surfactants which react with sodium hydroxide as in the following equations:**



Then the builders and fillers are added as in the following procedure

**Procedure:**

1. Prepare 5ml of 25% NaOH and 5ml of 40% sodium meta silicate ( $\text{Na}_2\text{SiO}_3$ ).
2. In a small beaker put 1.25 ml of the sulfonic acid and 1.25g of sodium lauryl sulfate and mix them well.
3. Gradually add 4.5ml of the 25% NaOH and continue mixing until the mixture become in pasty shape.
4. Now dissolve 5g of sodium pyrophosphate and 1.25g of trisodium orthophosphate in a 12.5ml D.W and add it gradually to the paste with mixing until it become homogeneous.
5. weigh 4.5g of sodium carbonate and add it to the mixture with mixing, then add 2.5ml of the 40% sodium meta silicate to the paste and continue mixing.
6. After the paste become homogeneous and highly viscous, pour it in a petry dish and put it in the oven till it become solid and dry.
7. grind the product and test it's cleaning activity.

**Phosphates test:** To test for phosphates in your test solutions and deionized water, place 2mL of each solution into clean separate test tubes. Add 5 drops of 1M  $\text{HNO}_3$  and 2ml of 1% ammonium molybdate solution to each test tube. Warm the test tubes in a water bath but do not boil the solutions. A yellow solution or formation of a precipitate indicates the presence of phosphate. Record your results.

## **Exp.6: Extraction of essential oils from plants.**

### **Essential Oil**

Essential oils are natural chemicals that are extracted from the leaves, flowers, stems, roots or bark of plants. Essential oil is concentrated, hydrophobic liquid containing volatile aroma compounds from plants. They are also known as volatile oils.

Essential oil is a more or less volatile material isolated from an odorous plant of a single botanical species by a physical process. They are oxygenated derivatives of hydrocarbon terpenes such as aldehydes, ketones, alcohols, phenols, acids, ethers and esters.

### **Fragrance Oil**

Fragrance oils also called perfume oils, they are usually synthetic; chemists analyze the plants' components and reproduce their chemical compositions.

Essential oil blends (combinations of essential oils) are not synthetic but some suppliers call them fragrance or perfume oils.

### **Uses of Essential Oil**

Essential oils are used in a wide variety of consumer goods such as:

- Bath and household cleaning products like detergents, soaps.
- Toilet and cosmetics products.
- Pharmaceuticals. They have been used medicinally at different periods in history. Medical applications proposed by those who sell medicinal oils range from skin treatments to remedies for cancer.
- Perfumes.
- Food products, soft drinks, and hard drinks.
- Insecticides.
- The world production and consumption of essential oils and perfumes are increasing very fast. Production technology is an essential element to improve the overall yield and quality of essential oil.

### **Extraction methods**

There are several methods of extraction of essential oils.

- 1- Hydrodistillation (HD).
- 2- Steam distillation (SD).
- 3- Soxhlet Extraction.
- 4- Enfleurage.
- 5- Expression

## Extraction with volatiles solvents

This method is used for delicate flowers whose odors are damaged by the high heat needed to boil water. The oils are extracted using solvents which have lower boiling points than water. Various substances such as ether or high-grade petroleum, which evaporate rapidly, are used in modern perfumery to dissolve essential oils from fragrant plant and.

In this experiment the essential oil will be extracted using a micro-wave laboratory technique to accelerate the extraction in a minimum period then separation of solvent using dean –stark apparatus.

### Procedure:

- 1- Put 10 g of the flowers or any plant parts in a 500ml beaker.
- 2- Add 100ml of solvent (water) to the beaker and close the beaker with watch glass.
- 3- Put them in the micro-wave apparatus for 2 minutes.
- 4- Bring out the beaker with a tong and transfer the liquid part to cylinder to separate solvent from the essential oil.

## Exp. 7: PREPARATION OF A SKIN CREAM

### OBJECTIVE:

To become familiar with the composition of a simple skin cream.

To deduce the purpose of some of the components in a skin cream.

### INTRODUCTION:

**Formulation:** *The formulation of something such as a medicine or a beauty product is the way in which different ingredients are combined to make it. You can also say that the finished product is a formulation.*

### The function of a skin cream

1. Protect the skin against harshness from the environment and any dry conditions of the skin.
2. It should aid the skin in carrying out its normal functions that is restoring moisture to dry skin.
3. Allow the elimination of waste matter through the pores.
4. Cooling of the body by evaporation of water (perspiration), thus aiding in the maintenance of the normal body temperature.

5. Skin creams contain ingredients for adding body.
6. Improving texture, improving the spread-ability, improving the odor.
7. Emulsifying the oil and water components.
8. Raising the melting point.
9. Softening the skin, and providing various medicinal properties.

If the cream clogs the pores of the skin with heavy, insoluble, inert material, it results in a thick sticky coating on the skin and prevents sufficient normal skin function, being detrimental to health.

### **The composition (ingredients) of a skin cream**

The primary ingredient that make lotion include both water soluble ingredients and oil soluble ingredients. To hold them together requires an emulsification system.

Skin creams contain a variety of ingredients that range from common, such as mineral oil, to the exotic, such as placenta extract. Some skin creams may contain small amounts of vitamins or other “nutrients”.

A basic and satisfactory skin cream can be prepared from stearic acid, lanolin, mineral oil, triethanolamine, and water.

### **The Process of Cosmetic Manufacturing**

Formulation of the product: Product formulation is the very first step that precedes the manufacturing process. This especially concerns the quality, efficacy, texture, aroma, color and general look of the product.

Manufacture: Manufacturing techniques keep changing at high speed these days, it is a priority for each company to keep up with the latest advancements and innovations.

Fill and pack: There are specialists responsible for the fill and pack of cosmetic products. The product should be filled into appropriate containers. When it comes to cosmetics for skin, body and hair care, packaging materials are mainly represented by jars, bottles, tubes, pumps etc. Each product is further packed into boxes or cartons for enhanced maintenance and safe storage.

Store and dispatch.

The purpose of this experiment to prepare a skin cream from these ingredients, and, then, by omitting one of the ingredients in subsequent preparations, to deduce the purpose of that particular ingredient.



## PROCEDURE:

Prepare 4 skin creams as described in the procedure below. Use the ingredients and amounts shown in: Table 1. Composition of Skin Cream Mixtures (Label each mixture).

### Ingredient Mixture 1 Mixture 2 Mixture 3 Mixture 4

Stearic acid	10 g	10 g	10 g	—
Lanolin	7 g	7 g	7 g	7 g
Mineral oil	10 g	10 g	—	10 g
Trietanolamine	2 ml	—	2 ml	2 ml
Water	48 ml	48 ml	48 ml	48 ml

Place a 150-ml beaker on a balance and weigh it. Weigh the quantities of stearic acid, lanolin, and mineral oil, called for in Mixture 1, in Table 1, above, into the 150-mL beaker. Heat the beaker in a water bath until all the ingredients have melted. (Cosmetic ingredients should not be melted over a direct flame or high heat because they may scorch or decompose if they are heated much above the boiling point of water.) Keep this mixture warm while heating the water-trietanolamine mixture in the next step.

Measure 48 mL of water into a 250-mL beaker. Measure the amount of triethanolamine, as called for in Mixture 1, to the water in the 250-mL beaker. Stir. Heat this mixture to a temperature of 80° to 90°C.

The mixture may be heated directly on a hot plate on medium heat or in a water bath.

After the water solution has reached a temperature between 80° and 90°C, remove it from the heat and slowly pour the melted stearic acid-lanolin-mineral oil mixture into the water a little at a time, stirring constantly. It may be helpful to hold the 250-mL beaker using a pair of beaker tongs. If you pour too fast or if you do not stir, your emulsion will be lumpy.

Continue stirring until you have a smooth, uniform paste. Label the beaker as “Mixture 1”, and set the skin cream aside to cool.

Repeat the procedure to make another skin cream using the quantities of materials called for in Mixture 2, 3&4

After the skin creams have cooled, compare the properties of each cream prepared. Note which ingredient is missing in each preparation. Based on the difference between the properties of a normal skin cream (Mixture 1) and those of each of Mixtures 2, 3 and 4, determine the function of the missing ingredient.

You may take your skin cream home. Commercial skin creams usually contain fragrance so they have a pleasant odour.

Add the perfume one or two drops at a time and stir well to blend it into the skin cream until the desired level of odour is obtain.

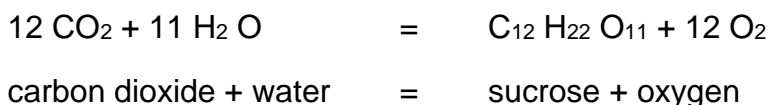
## **Exp8: Extracting sugar from sugar beet.**

### **SugarBeet**

Sugar beet is a temperate climate biennial root crop. It produces sugar during the first year of growth in order to see it over the winter and then flowers and seeds in the second year. It is therefore sown in spring and harvested in the first autumn/early winter. As for sugar cane, there are many cultivars available to the beet farmer. The beet stores the sucrose in the bulbous root which bears a strong resemblance to a fat parsnip.

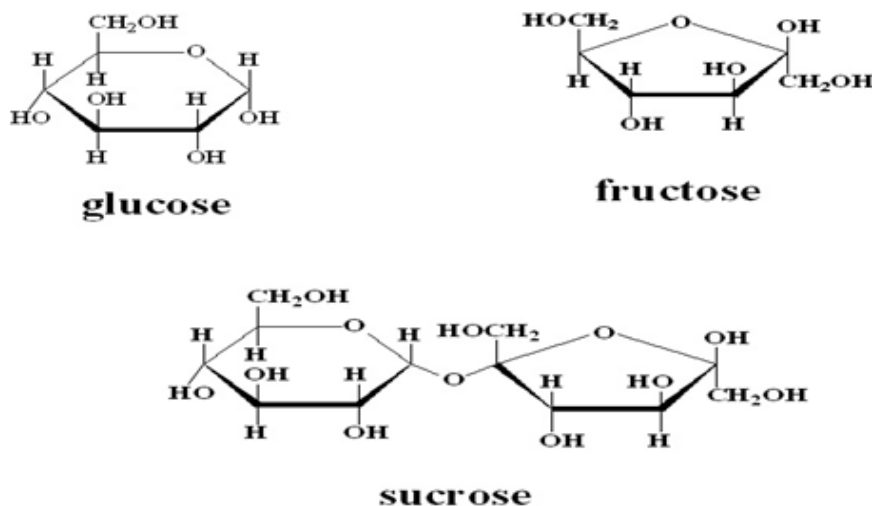
A typical sugar content for mature beets is 17% by weight but the value depends on the variety and it does vary from year to year and location to location. This is substantially more than the sucrose content of mature cane but the yields of beet per hectare are much lower than for cane so that the expected sugar production is only about 7 tons per hectare.

The process whereby plants make sugars is photosynthesis. The plant takes in carbon dioxide from the air through pores in its leaves and absorbs water through its roots. These are combined to make sugar using energy from the sun and with the help of a substance called chlorophyll. Chlorophyll is green which allows it to absorb the sun's energy more readily and which, of course, gives the plants' leaves their green colour. The reaction of photosynthesis can be written as the following chemical equation when sucrose is being made:



This shows that oxygen is given off during the process of photosynthesis. What we call sugar, the chemist knows as 'sucrose', one of the family of sugars otherwise known as saccharides in the grouping called carbohydrates. Carbohydrates, as the name implies, contain carbon and hydrogen plus oxygen in the same ratio as in water. The saccharides is a large family with the general formula  $\text{C}_n\text{H}_{2n}\text{O}_n$ . The simplest of the sugars is glucose,  $\text{C}_6\text{H}_{12}\text{O}_6$ , although its physical chemistry is not that simple because it occurs in two distinct forms which affect some of its properties. Sucrose,  $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ , is a disaccharide,

a condensation molecule made up of two glucose molecules [less a water molecule to make the chemistry work].



**Fig. 1- sugar molecule structure**

**Procedure:**

**Extraction of sugar from sugar beet.**

**Resources and equipment:**

- \_ Sugar beet (or parsnips, carrots, beetroot)
- \_ Chopping board and knife for slicing the sugar beet (or food processor)

**Practical tips**

**To slake quicklime** – heat a sample of quicklime (CaO) in a crucible for 15 minutes over a hot Bunsen flame. Test the sample by placing it in a beaker and adding a little water. If the water gets hot the lime is adequately slaked. To make the suspension continue to add water until the mixture has a consistency of emulsion paint. Use a safety screen while carrying this out.

**Experimental instructions**

**1. Preparation of the beet**

- \_ Wash the sugar beet and remove the stem of the beet.

- \_ Weigh it and record its mass.
- \_ Chop it up into long thin 'French fry' like pieces.- this will increase the surface area.
- \_ Put the sugar beet pieces into a beaker of boiling water and gently simmer for about half an hour, until it is soft. (Roughly 1.2 cm<sup>3</sup> water / g beet) – (to extract the sugar).
- \_ Separate the beet from the sugar by decanting. The impure solution can be stored for up to a week in a refrigerator.

## **2. Purification of the liquid extract**

- \_ Heat the liquid extract to 80 °C.
- \_ Add about 30 cm<sup>3</sup> of a suspension of freshly slaked lime (Ca(OH)<sub>2</sub>) – to react with the acidic impurities. This should be made up fresh before the lesson.
- \_ Carefully bubble CO<sub>2</sub> through the solution as shown in Figure 3.
- \_ Regularly take samples with a pipette and test the pH, by dropping the sample on thymolphthalein paper or using a pH meter.
- \_ Stop adding CO<sub>2</sub> when the pH reaches 11.2. The indicator will be pale blue.
- \_ At pH 11.2, filter the extract under vacuum (see Figure 2).
- \_ Bubble CO<sub>2</sub> through the filtrate.
- Regularly take samples with a pipette and test the pH, by dropping the sample on Phenolphthalein drops on a porcelain tile or using a pH meter.
- \_ Stop adding CO<sub>2</sub> when the solution reaches pH 9 – the indicator will be light pink.
- \_ Filter under vacuum, using a fine filter paper. The straw coloured filtrate is called 'Thin juice' and maybe stored in a refrigerator for up to a week.

## **3. Concentration of the juice by vacuum evaporation-**

- Set up the apparatus (fig2) for distillation under vacuum, and evaporate sufficiently at 40 °C under high vacuum.
- Add the thin juice and continue evaporation until the juice has a consistency of 'Thick juice'.

-Turn off the vacuum.

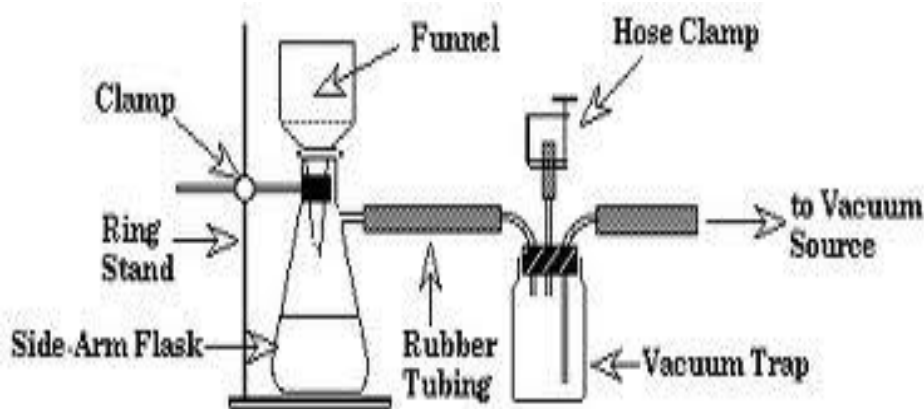
#### **4. Crystallisation**

\_ Remove the thick juice and add a little icing sugar, stirring slowly.

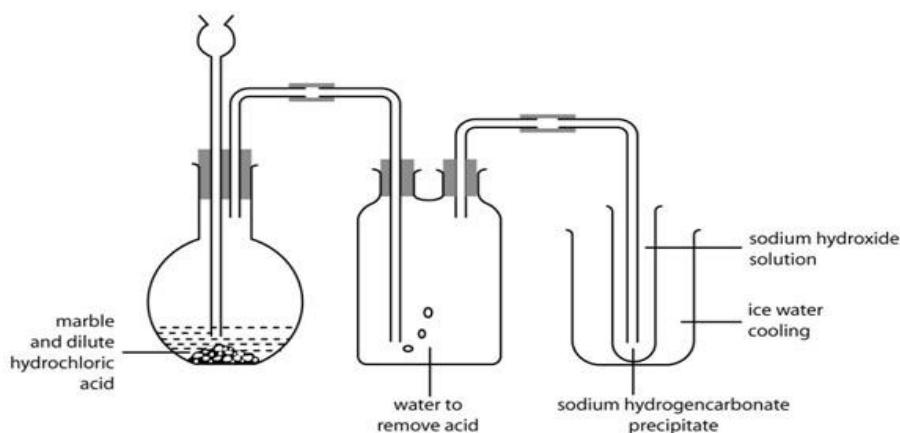
\_ Crystals will become visible in the juice, which can then be filtered off. The crystals will be a bright brown colour.

#### **Making sugar in the laboratory (a paper exercise)**

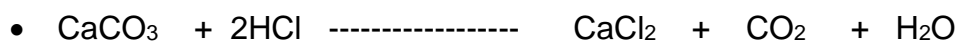
1. Sugar beet is chopped up to increase the surface area during the extraction process.
2. The sugar particles dissolve in the water.
3. Lime is added to remove the acidic impurities.
4. The pH of lime is about 14.
5. Carbon dioxide is a weak acid and it is neutralised by the lime. As the concentration of lime decrease the pH also decreases.
6. Using a pH meter or a suitable indicator.
7. A vacuum lowers the boiling point of a liquid. This will allow the water to evaporate off at lower temperature so that the newly formed sugar crystals will not decompose, and not leave a brown mess in the reaction vessel.
8. Label a diagram, showing how to filter by gravity or under vacuum.



**Fig. -2- Apparatus for vacuum filtration**



**Fig-3-(Apparatus for CO<sub>2</sub> bubbling)**



**Definitions of used term:**

**1. Sugar beet** – a root crop that stores sugar in its root. The root swells up as it grows beneath the ground. It looks like a large parsnip.

**Continuous** – the sugar beet extract flows directly from one place without stopping, until it reaches the end of the process.

**Crystallisation** – is the process by which crystals are formed from a saturated solution.

**Batches** – The reaction takes place in a big container. At the end of the reaction the product is emptied out and replaced with a new lot of reactant.

**Milk of lime** – a suspension of calcium hydroxide.

**Evaporators** – the place where water is evaporated to leave a concentrated solution.

**By-products** – extra products that are made during a reaction.

**Neutralise** – this happens when acids and alkalis react together.

## Exp-9: Preparation of Ammonium sulfate fertilizer

An inorganic chemical compound commonly used as a fertilizer. It contains 21% nitrogen as ammonia and 24% sulfur as sulfate. Its molecular formula is closely related to salt an ammonium sulfate analogue with an iron atom within the compound.

Ammonium sulfate is not soluble in alcohol or liquid ammonia. The compound is slightly hygroscopic and absorbs water from the air at relative humidity > 81% (at 20°C).

Ammonium sulfate is prepared commercially by reacting ammonia with sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). Ammonium sulfate is prepared commercially from the ammonia liquor of gas-works and is purified by recrystallization. It forms large rhombic prisms, has a somewhat saline taste and is easily soluble in water. The aqueous solution on boiling loses some ammonia and forms an acid sulfate.

It is used largely as an artificial fertilizer for alkaline soils. In the soil the sulfate ion is released and forms sulfuric acid, lowering the pH balance of the soil (as do other sulfate compounds such as aluminum sulfate), while contributing essential nitrogen for plant growth. It is also used as an agricultural spray adjuvant for water soluble insecticides, herbicides, and fungicides. There it functions to bind iron and calcium cations that are present in both well water and plant cells. It is particularly effective as an adjuvant for 2, 4-D (amine), glyphosate, and glufosinate herbicides. It is also used in the preparation of other ammonium salts. In biochemistry ammonium sulfate precipitation is a common method for purifying proteins.

This fertilizer can be prepared from different industrial methods like:

- 1- From sulfuric acid and ammonia by humid and dry method.
- 2- From coal gas and ammonia by direct, indirect and semi direct methods.
- 3- From calcium sulfate and ammonia.
- 4- From H<sub>2</sub>S and ammonia.

Reaction equation:  $2\text{NH}_4\text{OH} + \text{H}_2\text{SO}_4 \rightarrow (\text{NH}_4)_2\text{SO}_4 + 2\text{H}_2\text{O}$

### Procedure:

- 1- Calculate the amount of ammonium hydroxide and sulfuric acid which is needed for the reaction from the reaction equation.

- 2- Dilute the acid with D.W in percent (1:2) then add it to the ammonia solution gradually with mixing then put it on a sand bath.
- 3- Evaporate the solution until ammonium sulfates crystals forms, then dry it in the oven after that weight the product and calculate the percentage.

## Exp.10: Determination of alkalinity percent of urea

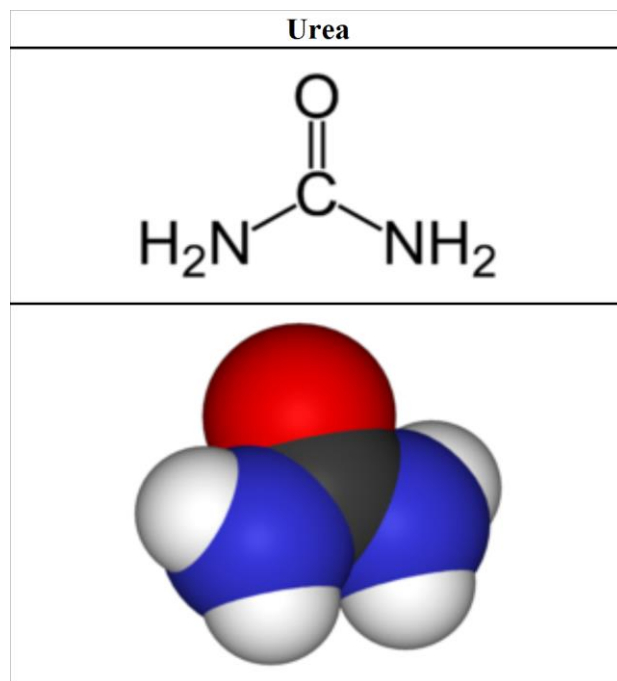
### Urea:

Urea is an organic compound with the chemical formula  $(\text{NH}_2)_2\text{CO}$ . Urea is also known as carbamide, Other names include carbamide resin, isourea, carbonyl diamide, and carbonyldiamine. It was the first organic compound to be artificially synthesized from inorganic starting materials.

### Structure

Urea is highly soluble in water and is therefore an efficient way for the human body to expel excess nitrogen. Due to extensive hydrogen bonding with water (up to six hydrogen bonds may form - two from the oxygen atom and one from each hydrogen), it is very soluble and thus is also a good fertilizer.

The urea molecule is planar and retains its full molecular point symmetry, due to conjugation of one of each nitrogen's P orbital to the carbonyl double bond. Each carbonyl oxygen atom accepts four N- H-O hydrogen bonds, a very unusual feature for such a bond type.



### Production

Urea is a nitrogen-containing chemical product which is produced on a scale of some 100,000,000 tons per year worldwide. Urea is commercially produced from synthetic ammonia



and carbon dioxide. Urea can be produced as prills, granules, flakes, pellets, crystals and solutions.

Large quantities of carbon dioxide are produced during the manufacture of ammonia from coal or from hydrocarbons such as natural gas and petroleum derived raw materials. This allows direct synthesis of urea from these raw materials. The production of urea from ammonia and carbon dioxide takes place in an equilibrium reaction, with incomplete conversion of the reactants. The various urea processes are characterized by the conditions under which urea formation takes place and the way in which unconverted reactants are further processed. Unconverted reactants can be used for the manufacture of other products, for example ammonium nitrate or sulphate, or they can be recycled for complete conversion to urea in a total - recycle process.

Two principal reactions take place in the formation of urea from ammonia and carbon dioxide. The first reaction is exothermic:



While the second reaction is endothermic:



## Uses

- 1- **Agricultural use:** Urea is used as a nitrogen release fertilizer as it hydrolyses back to ammonia and carbon dioxide.
- 2- **Industrial use:** Urea has the ability to form 'loose compounds' with many organic compounds. The organic compounds are held in channels formed by interpenetrating helices comprising of hydrogen bonded urea molecules.
- 3- **Laboratory use:** Urea is a powerful protein denaturant. This property can be exploited to increase the solubility of some proteins.
- 4- **Drug use:** Urea is used in topical dermatological products to promote rehydration of the skin.
- 5- **Textile use:** Urea in textile laboratories are frequently used both in dyeing and printing as an important auxiliary which provides solubility to the bath and retains some moisture which is required for the dyeing or printing process.

In this experiment we appreciate the amount of ammonium hydroxide in the fertilizer sample which produced from the dissolution of ammonia in the humidity, and the percentage of ammonium hydroxide vary from one sample to another.

**Procedure:**

- 1- Prepare mixed indicator by dissolving 0.1g of methyl red in 50 ml of ethanol then add 0.05 g methyl blue then dissolve them and complete the volume to 100 ml by ethanol.
- 2- weigh 50g from urea fertilizer in a conical flask then dissolve it by adding 100ml of D.W.
- 3- add some drops from the mixed indicator and titrate the solution with 0.1M HCl until the color change.
- 4- Calculate the alkalinity percent by:  
$$\text{Alkalinity percent} = (\text{HCl volume} / \text{weight of used urea}) * 0.17$$

## **Exp.11: Corrosion & How much oxygen consumed during rusting?**

Corrosion generally has been defined as "the action of eating or wearing away by slow degrees", or it is a transfer of metals and their alloys to brittle materials because of chemical transfer begins from the surface to the inside of the metal.

In most cases there are two kinds of corrosion happens in the same time at the internal and external surfaces of the metal, where it is opened to the atmospheric circumstances which contains several corrosion causes like oxygen, humidity, and acid rains...etc., while the internal surface is subjected to corrosion causes during the chemical reactions like acids, bases, salts and the electrochemical actions.

Corrosion today is considered from the important problems which face the industry generally and the chemical industry specially, that's because most of the chemical industry contains one or more of the corrosion causes, so because of the importance of corrosion in the chemical industry, it becomes one of the major specialties in the chemical engineering nowadays where it becomes studied from all of its chemical, physical and technical sides.

There are two main types of corrosion:

**A) Physical corrosion** happens because of the environment conditions and its action is on the external surface of the metals, and it occurs as a result to the friction happens in the moving mechanical parts.

**B) Chemical corrosion** is the chemical homolysis of metals losing it's rigidity and transferred to a brittle materials because of the direct and indirect chemical actions, so there are two kinds of the chemical corrosion:

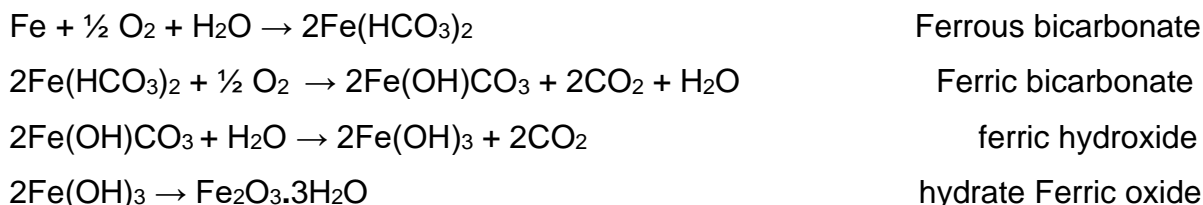
**1- Direct chemical corrosion:** this kind occurs as a result for the direct contact between the metal and corrosion cause as shown below:

**Metal + corrosion cause (Oxygen, Halogen, H<sub>2</sub>S, SO<sub>x</sub>, Acids (H<sup>+</sup>)) → reaction result (Metal oxide, Metal hydrides, Metal sulfate or Sulfides, Metal salts).**

The rate of corrosion is affected by water and accelerated by electrolytes, as illustrated by the effects of road salt on the corrosion of automobiles.

Some times occurs that the reaction results may become a thin layer protect the metal surface it can be useful in industry to protect metals from corrosion by increasing the formation of this thin layer which characterized by a good cohesion with the metal, an example of this layer is the protection of Aluminum by a thin layer of it's oxides or Lead oxide or Chromium oxide.

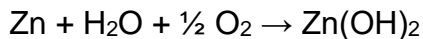
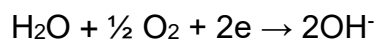
Ferrate and it's alloys are considered the most metals used in the industry and the most one which facing corrosion and the analysis for ferrate oxidation confirms that there is a part for CO<sub>2</sub> in the Ferrate oxidation process (Ferrate corrosion), also it is assured from occurrence of Ferrous and Ferric bicarbonate, Ferric hydroxide, Hydrate Ferric oxide (rust) as shown in the following equations:



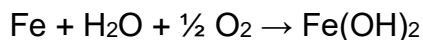
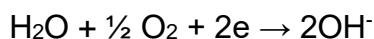
This kind of oxides considered brittle oxides not cohesive and swell so it permit the oxygen to reach the metal surface so it not protect the metal from corrosion.

**2- Indirect chemical corrosion:** This kind occurs as a result for contact between the metal surface and a liquid solution, generally it is due to the occurrence of some electrochemical reactions, i.e. corrosion happens when there is a transfer media to the chemical action between the metal and corrosion cause (usually this media is water), as a result for this the electrochemical cells will be made where the electrochemical reactions happens, for example if a

contact happens between Copper (it's potential difference is +0.35 v) and Zinc (it's potential difference is -0.77 v) in presence of water, so Zinc will lose it's electrons to copper and in the same time lose positive ions leaves it's surface to media (water), i.e. copper will be protected by Zinc as in the following equations:



To prepare a corrosion cell there is no need for two metals, just one metal and it's oxide for the potential difference to happen, this can be noticed between ferrate and it's oxide since there is a notable potential difference between the Ferrate metal which anode (corrosive) and the oxide which is cathode, so corrosion continues as in the following equations:



## Corrosion protection

To reduce corrosion effect we cannot theoretically prepare a material which absolutely resistant to corrosion, but there are different methods which were used to reduce the effect of corrosion, like:

- 1- coating or covering metal surface by thin layers of oxides and phosphates and metals or paints and organic paints.
- 2- Preventing or reducing the occurrence of chemical reactions which causes the corrosion, by using chemical compounds like silicates, borates, amines, alcohols, and others.
- 3- Preventing or reducing the occurrence of electrochemical reactions and corrosion cells, that's done by using anodic and cathodic protection.

We can make this experiment to take a look on corrosion and it's conditions which increase or reduce from corrosion rate or preventing it.

**A) Wrought Steel corrosion:** Steel is alloys of Iron with nickel or chromium or manganese or vanadium and other metals in different percents.

**Procedure:**

- 1- Take four pieces of wrought iron each one (3\*1) cm and smooth it carefully using smoothing paper No.1.
- 2- place the first piece in a test tube contains boiled water then close it by stopper then cover it by paraffin wax.
- 3- Put the second piece in a test tube then close it by a piece of cotton inside it then add a specified amount of non aqueous calcium chloride then close by a stopper.
- 4- Place the third piece inside a filter paper and close it tightly then put it inside an opened test tube.
- 5- Place the last piece inside an opened test tube.
- 6- Let the four test tubes for a one week then discuss the results with reasons.

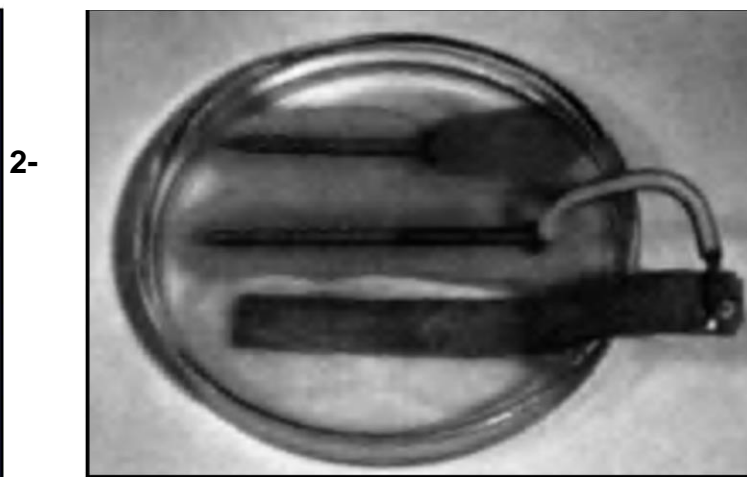
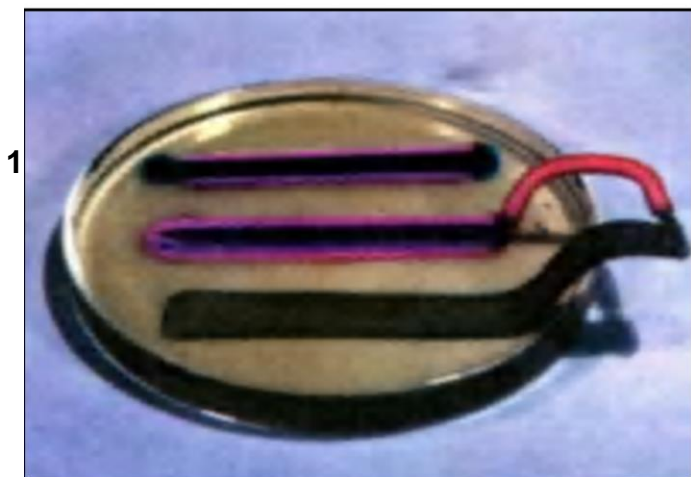
**B) Corrosion inhibiting by cathodic protection and sacrificial anodes:**

- 1- by using external current for protection of large metal composites for a long time by transferring the metal from anode to cathode by connecting it in the cathode of an external electric circuit and connecting the anode of the circuit to the anode buried in coal or gypsum in the corrosion environment to ensure the contact with the earth.
- 2- by using the sacrificial anodes which used in case of not presence of electric source, it provides a good protection for a specified period, where the metal which needs protection is connected with a more active metal directly or by wire so the corrosion will be concentrated on the active metal which is called the sacrificial anode and it's kinds are aluminum, zinc, magnesium, and their alloys.

**Procedure:**

- 1- Take four iron wires each one 4cm length then dry and weight them.
- 2- Place a wet filter paper in the bottom of a large flask then put the wires on it separately as follows:
  - One wire alone.
  - One wire with another one of zinc.
  - One wire with another one of aluminum.
  - One wire with another one of copper.

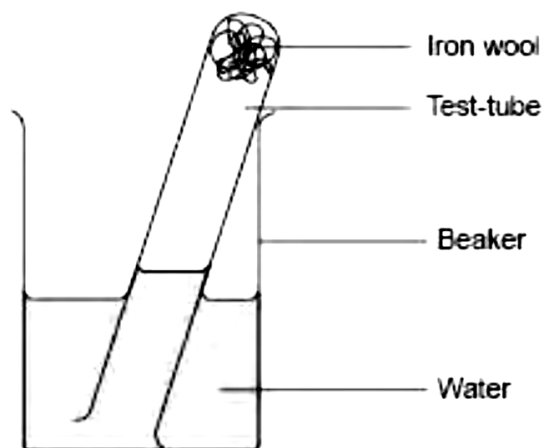
3- let them for a week then discuss the results. As in the following pictures:



### Rust:

is a general term for a series of iron oxides, usually red oxides, formed by the reaction of iron and oxygen in the presence of water or air moisture. Several forms of rust are distinguishable visually and by spectroscopy, and form under different circumstances. Rust consists of hydrated iron(III) oxides  $\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$  and iron(III) oxide-hydroxide ( $\text{FeO}(\text{OH})$ ,  $\text{Fe}(\text{OH})_3$ ). Rusting is the common term for corrosion of iron and its alloys, such as steel. Other metals undergo equivalent corrosion, but the resulting oxides are not commonly called rust. Given sufficient time, oxygen, and water, any iron mass eventually converts entirely to rust and disintegrates. The corrosion of aluminium is extremely slow because the resulting aluminium oxide forms a conformal coating, which protects the remaining aluminium. This process is known as passivation.

In this experiment Students set up iron wool to rust in a test tube full of air inverted in a beaker of water as in the following figure. As the iron wool reacts, rusts and removes the oxygen from the air, water is drawn up the tube. By observing the change in the volume of air in the tube, the percentage of oxygen in the air can be found.



**Procedure:**

1. Put about 2 g of iron wool into the test tube and wet it with water. Tip away excess water.
2. Put water into the beaker. Invert the test tube and place it in the beaker of water.

Measure the length of the column of air with the ruler ( $h_1$ ).

3. Leave for the next week.

4. Measure the new length of the column of air ( $h_2$ ), taking care not to lift the test tube out of the water.

5. Calculate the volume of oxygen consumed during the rusting process by the relation :

$$V = [\pi r^2 (h_2 - h_1)] \times 0.21$$

## Exp.12: Glass making

Glass is simply a mixture of oxides. Commercially, the main ingredient of glass is sand, or silicon oxide. This is heated with sodium carbonate at an extremely high temperature. This causes the oxides to melt. On cooling, a frozen super cooled liquid mixture is formed. This mixture is glass. Once it has been made, glass can be easily formed into new shapes by heating. The oldest method of preparing glass is by blowing. Glass-blowing has been practiced for many centuries - the Romans were well known for their skill in producing glass ornaments.

There are many different types of glass with different chemical and physical properties. Each can be made by a suitable adjustment to chemical compositions, but the main types of glass are:

### 1- Commercial Glass

Most of the glass we see around us in our everyday lives in the form of bottles and jars, flat glass for windows or for drinking glasses is known as commercial glass, Most commercial glasses have roughly similar chemical compositions of:

70% - 74%  $\text{SiO}_2$  (silica)

12% - 16%  $\text{Na}_2\text{O}$  (sodium oxide)

5% - 11%  $\text{CaO}$  (calcium oxide)

1% - 3%  $\text{MgO}$  (magnesium oxide)

1% - 3%  $\text{Al}_2\text{O}_3$  (aluminium oxide)

### 2- Lead Glass

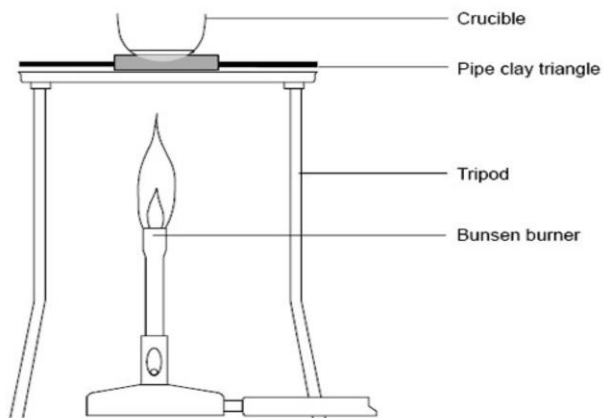
Commonly known as lead crystal, lead glass is used to make a wide variety of decorative glass objects. It is made by using lead oxide instead of calcium oxide, and potassium oxide instead of all or most of the sodium oxide. The traditional English full lead crystal contains at least 30% lead oxide ( $\text{PbO}$ ) but any glass containing at least 24%  $\text{PbO}$  can be described as lead crystal.

### 3- Borosilicate and Aluminosilicate Glass

Most of us are more familiar with this type of glass in the form of ovenware and other heat-resisting ware, better known under the trade name (Pyrex). Borosilicate glass, the third major group, is made mainly of silica (70-80%) and boric oxide (7-13%) with smaller amounts of the alkalis (sodium and potassium oxides) and aluminium oxide. aluminosilicate, contains 20% aluminium oxide (alumina- $\text{Al}_2\text{O}_3$ ) often including calcium oxide, magnesium oxide and boric oxide in relatively small amounts, but with only very small amounts of soda or potash. It is able to withstand high temperatures and thermal shock and is typically used in combustion tubes, gauge glasses for high-pressure steam boilers, and in halogen-tungsten lamps capable of operating at temperature as high as  $750^\circ\text{C}$ . Also there are many several types of glass their composition varies according to their use. In the laboratory, we didn't use silicon oxide as it melts at too high a temperature. Instead we used a mixture of lead oxide, boric acid and zinc oxide.

#### Procedure:

- 1- Weigh 3.25g of lead (II) oxide, 1.75g of boric acid and 0.25g of zinc oxide into the boiling tube and stopper firmly.
- 2- Shake the contents to ensure thorough mixing, and transfer into the crucible.
- 3- Heat carefully at and strongly with Bunsen flame, until the mixture becomes molten and runny.
- 4- Taking great care, remove the Bunsen flame from underneath the crucible, then use tongs to remove the lid and lift the crucible off the tripod. Pour one or two drops of the molten glass onto the heat resistant mat. Replace the crucible onto the tripod, and keep heating.
- 5- Allow the glass beads to cool on the mat for 5 minutes and then examine them.
- 6- Use the straightened paper clip to pick up a tiny speck of one of the metal oxides provided and stirr this into the remaining molten mixture. Do not add too much powder or you will produce a very dark piece of glass.
- 7- Remove the Bunsen flame, and use tongs to pour out one or two drops of the coloured glass from the crucible to form beads on the mat. Note the colour of the glass you have now produced. Place the crucible on the mat to cool.





## Exp.13: preparation of Indigo dye

**Indigo dye** is dye with a distinctive blue color, The chemical compound that constitutes the indigo dye is called indigotin. The ancients extracted the natural dye from several species of plant, but nearly all indigo produced today is synthetic. Historically, indigo played an important role in many countries' economies because natural blue dyes are rare. among other uses, it is used in the production of denim cloth for blue jeans.

A variety of plants have provided indigo throughout history, but most natural indigo is obtained from those in the genus *Indigofera*, which are native to the tropics.

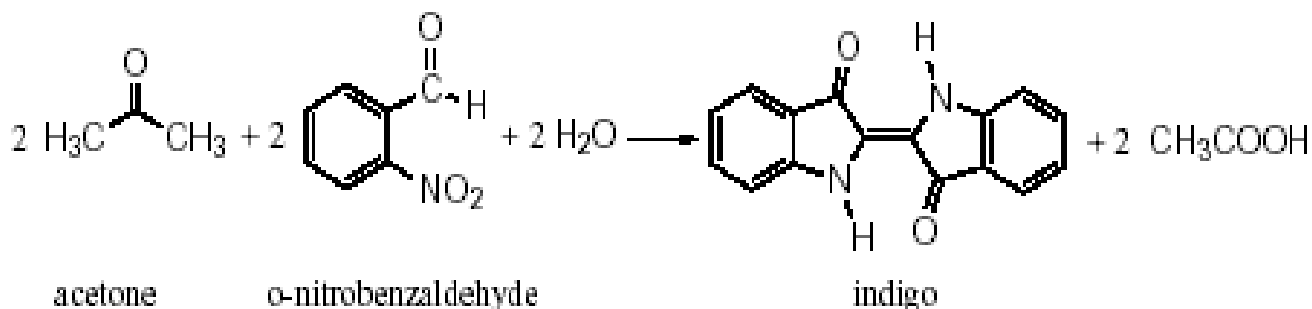
### Chemistry:

Indigo is a crystalline powder, insoluble in water, alcohol and ether with a melting point higher than 390°C. absorbs light in the yellow region of the spectrum (maximum at 602 nm), the indigo molecule is relatively small with molecular weight of 262.27 atomic units of mass.

Soluble in chloroform, nitrobenzene, or concentrated sulfuric acid. The chemical structure of indigo corresponds to the formula  $C_{16}H_{10}N_2O_2$ .

### Description of the Chemical Reactions

Acetone, base, and o-nitrobenzaldehyde are mixed and many reactions occur very rapidly and are accompanied by a color change.



The formulas for the compounds are as follows:

acetone:  $C_3H_6O$  o-nitrobenzaldehyde:  $C_7H_5NO_3$  Indigo:  $C_{16}H_{10}N_2O_2$

The blue, *water-insoluble* indigo is isolated and then treated with sodium dithionite, a chemical that adds an electron to each indigo molecule, to produce the pale yellow, *water-soluble* **leucoindigo**:

**Procedure:**

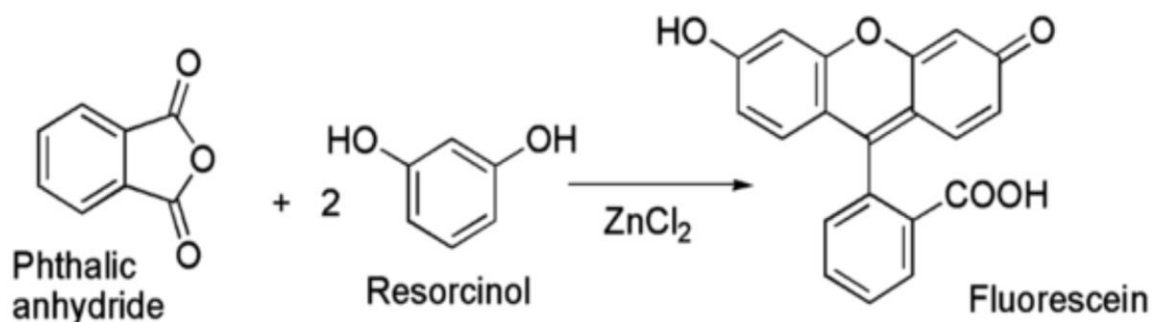
1. Weigh out about 0.5 g of o-nitrobenzaldehyde into a clean 50 ml beaker.
2. Place the beaker a hot plate in the fume hood. Add 5 ml of acetone and with a glass rod, stir gently until the o-nitrobenzaldehyde dissolves.
3. Add 5 ml of water and continue the stirring.
4. Add 2.5 ml of 1 M NaOH one drop at a time. The blue color of the indigo will appear.
5. Continue stirring the solution for about 10 min.
6. Prepare the filter paper and Buchner funnel.
7. When the reaction is complete, place the beaker in an ice bath for 5-10 minutes to cool the solution and form good crystals.
8. Start the vacuum, wet the filter paper with distilled water and pour the ice-cold indigo solution into the filter.
9. Wash the indigo with 10 ml of water by pouring the water into the beaker, swirling to dissolve any remaining solid indigo, and emptying the beaker into the filter paper.

## **Exp.14: Preparation of Fluorescein and Eosin dyes**

This kind of dyes classified under Xanthane dyes or pthaleins dyes because it prepared from the condensation of phenolic compounds with the Phthalic unhydride, to form a yellow and fluorophore green solutions called Fluorescein ( $C_{20}H_{12}O_5$ ).

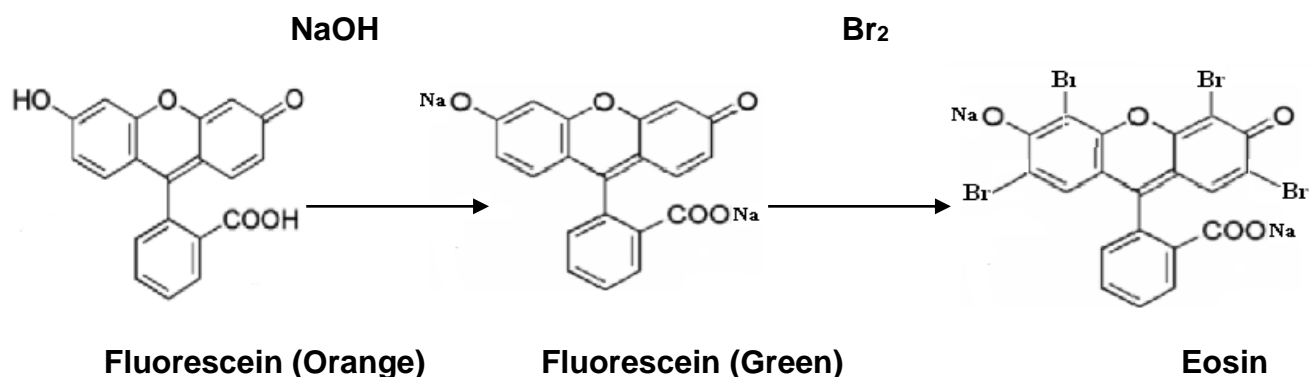
One of its more recognizable uses was in the River, where fluorescein was the first substance used to dye the river green in 1962. Other uses of fluorescein include using it as a water-soluble dye added to rainwater in environmental testing simulations to aid in locating and analyzing any water leaks. In cellular biology, the isothiocyanate derivative of fluorescein is often used to label and track cells in fluorescence microscopy applications.

Fluorescein can be prepared from phthalic anhydride and resorcinol in the presence of zinc chloride as follows:

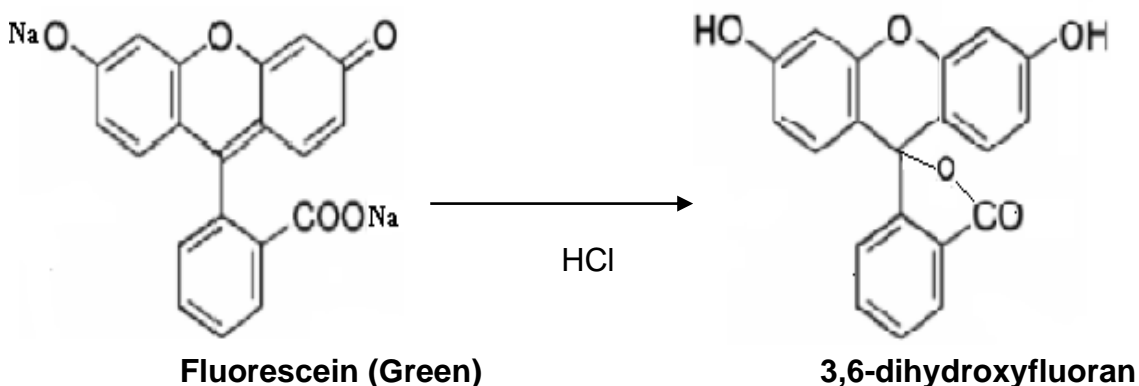


Eosin is a fluorescent red dye resulting from the action of bromine on fluorescein. It can be used to stain [cytoplasm], collagen and muscle fibers for examination under the microscope. Eosin is most often used as a counterstain to haematoxylin in H&E (haematoxylin and eosin) staining. H&E staining is one of the most commonly used techniques in histology.

Eosin can be prepared after treating the fluorescein with NaOH then halogenating it with bromine to produce the red Eosin dye as follows:



After treating with NaOH, when fluorescein is treated with HCl, it will give another derivative yellow dye called 3,6-dihydroxyfluoran as follows



In this experiment we will study these reaction briefly according to the following Procedure

- 1- Put (2.75g) resorcinol with (1.8g) phthalic anhydride in a round bottom flask equipped with condenser, thermometer and stirrer if possible.
- 2- Heat the flask in an oil bath to 180 °C, during heating grind 0.88g of Zinc chloride anhydrous then add it to the flask gradually with mixing and continue heating for one hour.
- 3- Cool the oil bath to 90 °C then add to the reaction flask 25ml D.W and 1.25ml of conc. HCl, continue heating with mixing to boiling, fix the temperature to 100 °C until all Zinc salts dissolved.
- 4- Filterate the undissolved precipitate (Fluorescein), wash it then dry and weigh it.
- 5- Dissolve the precipitate in NaOH and notice the color then take a part of it and add drops of conc. HCl and notice the change in color.
- 6- Weigh the second part of the precipitate and add bromine water to it which will form the red Eosin dye, then filterate, dry and weigh it. (if the red Eosin dye didn't appear when adding bromine, so you can add a small amount of icy acetic acid.