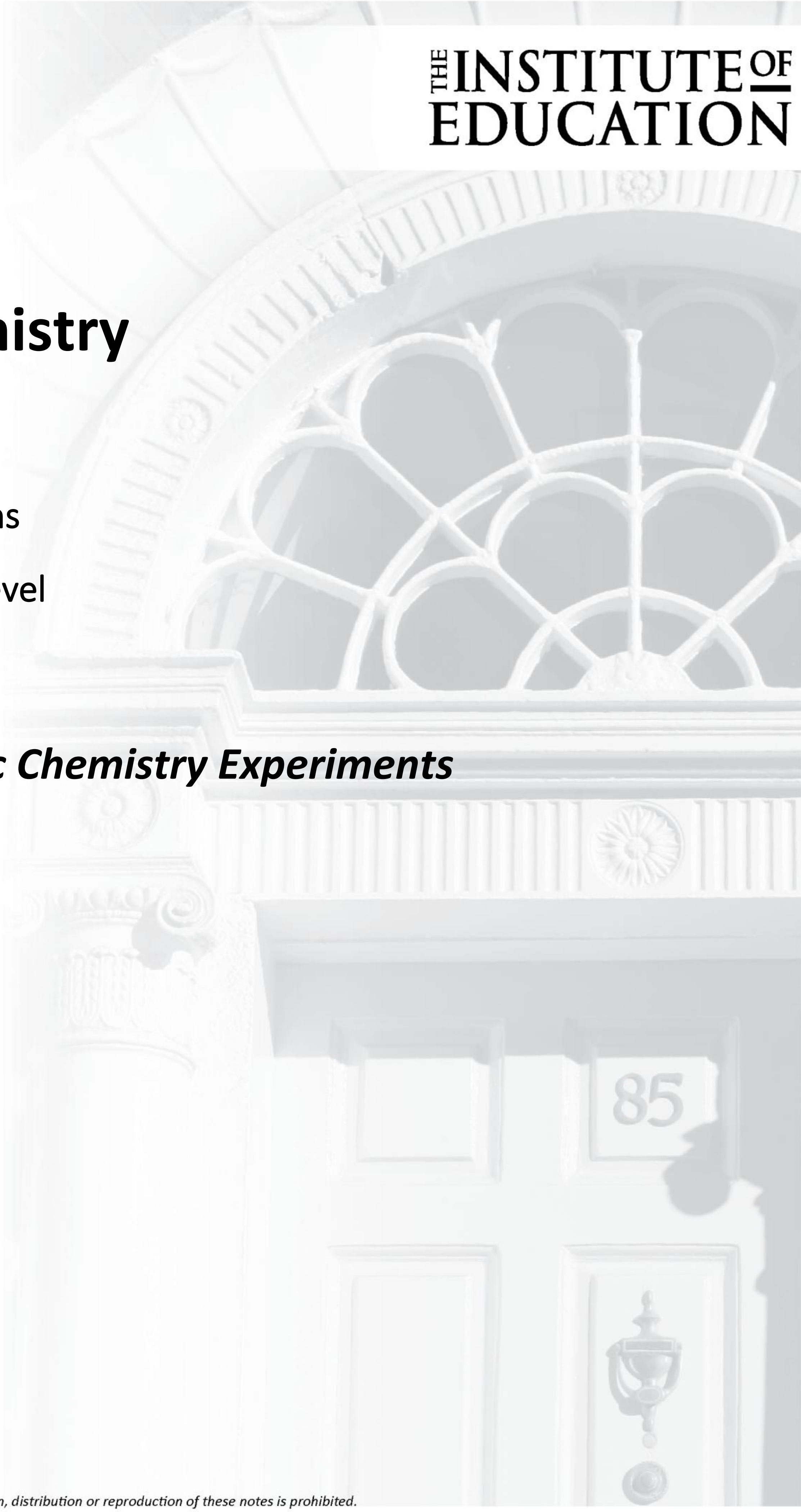


THE INSTITUTE OF EDUCATION

Chemistry

Tara Lyons

Higher Level

2020-21

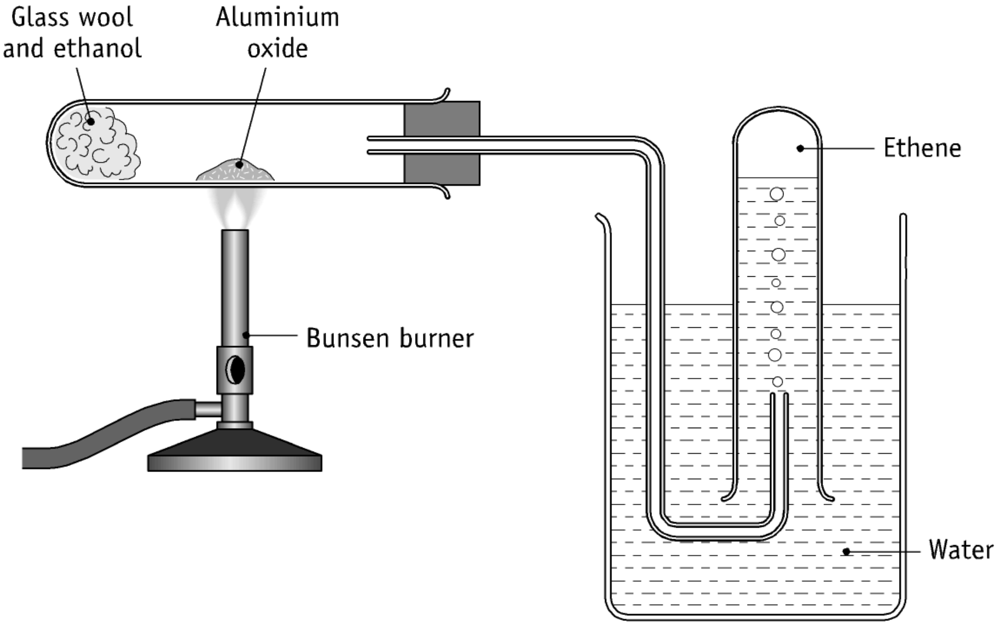
Organic Chemistry Experiments

ORGANIC CHEMISTRY EXPERIMENTS – Question 2 every year and sometimes Question 3 also. Practice and be prepared!!! **Tara Lyons**

ETHENE

Ethene is a very important organic compound as it is the 'building block' from which many plastics are made.
In the laboratory, ethene is prepared by a **dehydration reaction** of **ethanol.** Dehydration means removing water.
This is an example of an **elimination reaction**.

EXPERIMENT – THE PREPARATION OF ETHENE



Equation - $C_2H_5OH \xrightarrow[-H_2O]{Al_2O_3} C_2H_4 + H_2O$

Apparatus – boiling tube, retort stand, glass tubing, water bath, test tubes, Bunsen burner, rubber bung.

Materials – ethanol, aluminium oxide, water.

Method –

1. Some ethanol is poured into a boiling tube.
2. **Glass wool** is pushed down into the tube to (a) **soak up the ethanol** and (b) to **hold the ethanol in place** at the end of the tube during the experiment as it is a liquid.
3. The boiling tube was clamped in a horizontal position using the retort stand.
4. Some **aluminium oxide** (white powder) is placed half way along the tube. Aluminium oxide is the dehydrating agent/catalyst for the reaction.
5. The tube is stoppered with a rubber bung and glass tubing is attached through the rubber bung into the water bath.

6. A test tube is filled with water and placed in the water bath as shown in the diagram.
7. A Bunsen burner is placed under the aluminium oxide and is lit to give a blue flame.
8. The heat from the Bunsen burner vaporises the ethanol and as it passes over the aluminium oxide the ethanol is dehydrated to produce ethene.
9. The first test tube collected will have a low yield of ethene, as it will mainly consist of displaced air from the boiling tube.
10. Collect 5 more test tubes of ethene and stopper them.
11. **N.B. - Before turning off the Bunsen burner it is very important to remove the glass tubing from the water bath to prevent 'suck back'**. If the tubing was left in the water bath, once the heat is removed the gases in the boiling tube would contract and this would cause water to be drawn back into the boiling tube to balance the drop in pressure which would cause the boiling tube to shatter.
12. **Other precautions include** – keep gas away from flames as gas is flammable and there is a risk of explosion, wear safety glasses, use of tongs when handling glass wool.

PROPERTIES OF ETHENE GAS

1. **Physical appearance** – colourless gas with a sweetish smell.
2. **Solubility in water** – the gas is insoluble in water as water is a polar solvent and ethene is non-polar therefore ethene does not form hydrogen bonds with water. This is why ethene can be collected by the displacement of water.
3. **Combustion** – when a lighted taper is placed in a test tube of ethene, **a luminous flame** is observed. A small amount of smoke may also be produced. If limewater is added after burning, the limewater goes 'milky'. What does this prove? Write a balanced equation for the combustion of ethene gas -

   ____

4. Tests for unsaturation:

(a) **Addition of bromine water** – bromine water is a red/brown colour. When bromine water is added to ethene an *ionic addition reaction* occurs. The bromine adds across the double bond to form 1,2-dibromo ethane. This colour change from **red-brown to colourless** proves that ethene is unsaturated.

Equation - $C_2H_4 + Br_2 \rightarrow C_2H_4Br_2$

(b) **Addition of dilute potassium manganate (VII)** – a dilute solution of potassium manganate (VII) is **light purple** in colour. When this solution is added to ethene gas and shaken the purple colour of the solution goes **colourless**. This test also proves the presence of the carbon – carbon double bond, thus proving that ethene is unsaturated.

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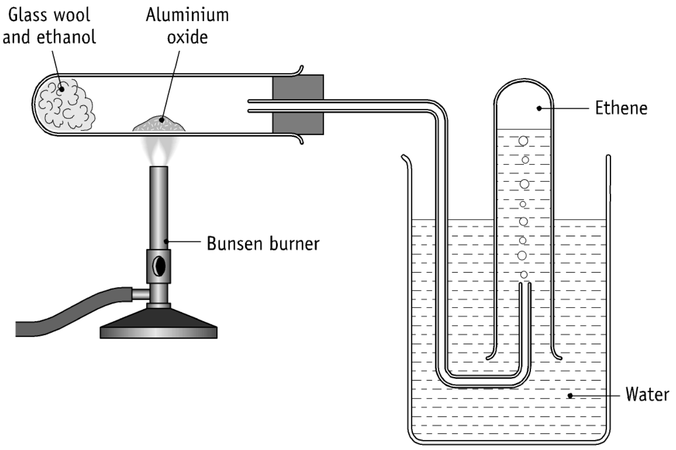
LEAVING CERT 2004

The diagram shows the experimental set up used by a group of students to prepare a sample of ethene from ethanol and to collect the ethene produced.



(a) What is the function of the glass wool?(5)

Identify the solid X and describe its appearance. (6)

(b) State and explain two safety precautions which should be observed when carrying out the student experiment. (12)

(c) Write a balanced equation for the reaction involved in this preparation. (6)

(d) If the ethene produced is bubbled through an acidified solution of potassium manganate (VII), the solution is decolourised showing that ethene is *unsaturated.* What is meant by the term *unsaturated*? Describe how you would carry out another test to confirm that ethene is unsaturated. (12)

(e) Describe the flame that would be observed when a combustion test is carried out on a sample of ethene gas. Write a balanced equation for the combustion of ethene in excess oxygen. (9)

LEAVING CERT 2005 Q7

Describe with the aid of a labelled diagram how the conversion of W (ethanol to ethene) may be carried out in a school laboratory and how a sample of the product may be collected. How would you test this product to show that it is unsaturated. (18)

LEAVING CERT 2006 Q9

Ethene may be made in a school laboratory using the arrangement of apparatus drawn.

(i) Give the name and formula of the solid A which is heated using the Bunsen burner. (6)
(ii) Identify the solid B which is used to keep the ethanol at the end of the test tube. (3)
(iii) What precaution should be observed when heating is stopped? Why is this necessary? (6)
(iv) Give one major use of ethene gas. (3)

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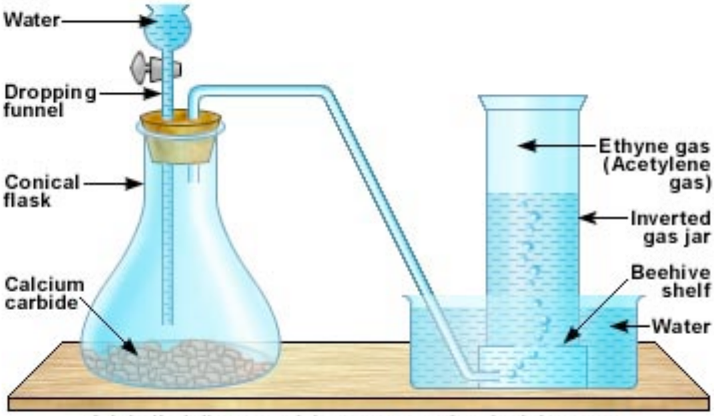
ETHYNE

Ethyne is used for cutting and welding metals as when burned in oxygen it can reach temperatures over 3000 ° C.

EXPERIMENT – PREPARATION OF ETHYNE



A labelled diagram of the apparatus for the laboratory preparation of ethyne or acetylene.

Equation - $CaC_2 + 2H_2O \rightarrow C_2H_2 + Ca(OH)_2$

Calcium dicarbide reacts with water to produce ethyne and calcium hydroxide.

Apparatus – dropping funnel, Buchner flask, glass tubing, conical flask, two holed rubber bung, test tubes, water bath.

Materials – calcium dicarbide, water, [acidified copper (II) sulfate solution.]

Method –

1. Set up the apparatus as shown in the diagram.
2. Do not touch the calcium dicarbide as sweat from hands may start the reaction.
3. Note that the **calcium dicarbide** is a **greyish solid.**
4. Slowly add the water from the dropping funnel.
5. Note **effervescence** (fizzing) as the water reacts with the calcium dicarbide.
6. Also note the production of calcium hydroxide in the Buchner flask. This appears as a white suspension (foamy).
7. The first test tubes collected will have a low yield of ethyne, as they will contain mainly displaced air from the Buchner flask. Discard these carefully.
8. Collect 4 test tubes of ethyne gas.

Important notes –

(a) Impurities are present in the calcium dicarbide. These solid impurities are calcium phosphide, Ca_3P_2 and calcium sulfide, CaS .

(b) These solid impurities give rise to gaseous impurities in the ethyne gas collected. The gaseous impurities are phosphine, PH_3 and hydrogen sulfide, H_2S.

(c) The gaseous impurities are removed by bubbling the impure ethyne gas through acidified copper (II) sulfate solution.

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PROPERTIES OF ETHYNE GAS

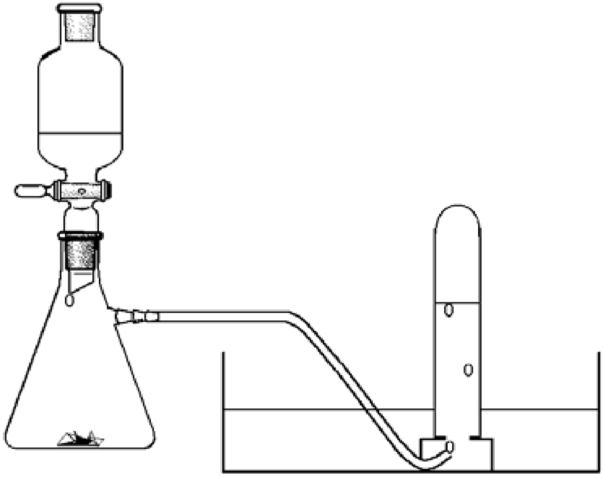
1. **Physical properties** – ethyne is a colourless gas with a sweetish smell if pure.
2. **Solubility in water** – ethyne is insoluble in water, as water is a polar solvent and ethyne being non-polar does not form hydrogen bonds with water. This is why ethyne can be collected by the displacement of water.
3. **Combustion** – N.B. very dangerous and must be done in a fume cupboard!! Ethyne burns with a **smoky luminous flame**. The smoke is due to unburned carbon (very sooty). To ensure no soot use an excess of oxygen when burning. Write a balanced equation for the combustion of ethyne:____
4. **Tests for unsaturation** –

(a) **Addition of bromine water** – when bromine is added across the carbon-carbon triple bond the red-brown colour of the bromine goes colourless thus proving that the ethyne is unsaturated. An equation showing this reaction was asked in 2003.

(b) **Addition of dilute potassium manganate (VII)** – the purple colour of the manganate (VII) solution goes colourless also proving the presence of a multiple bond (carbon-carbon triple bond) hence ethyne is unsaturated.

LEAVING CERT 2003

The diagram shows an apparatus that can be used for the preparation of ethyne gas, C_2H_2. A liquid X is dropped onto the solid Y and the gas collected in test tubes as shown



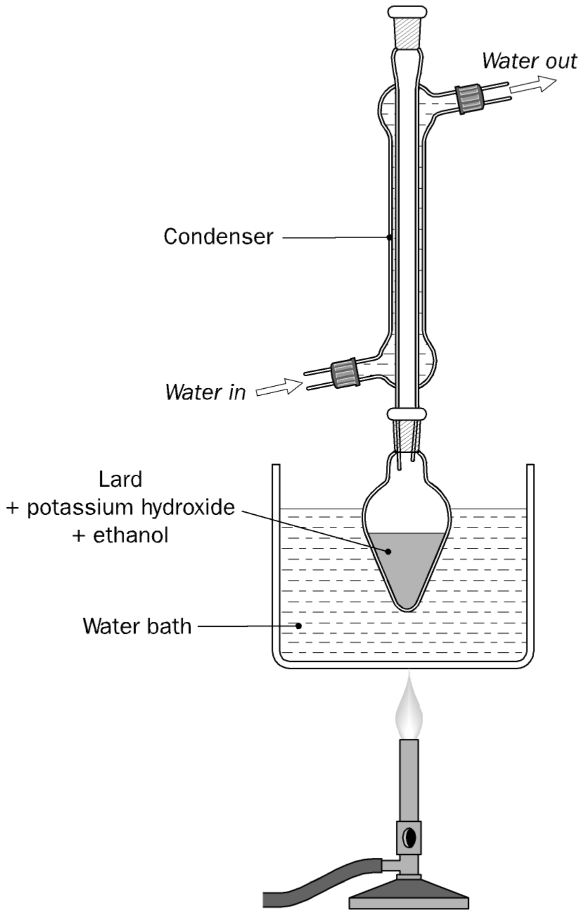
(a) Identify the liquid X and the solid Y. (8)
(b) Describe the appearance of solid Y.(3)
(c) Write a balanced equation for the reaction between X and Y producing ethyne. (6)
(d) What is observed when a sample of ethyne gas is burned in air? Write a balanced equation for the combustion of ethyne in oxygen.(9)
(e) Ethyne, C_2H_2, is described as an unsaturated hydrocarbon.
Describe a test you could carry out to show that ethyne is unsaturated.
Write an equation for the reaction taking place. Name the organic product (18)
(f) The common name for ethyne gas, C_2H_2, is acetylene. Give one major use of the gas. (6)

ORGANIC CHEMISTRY EXPERIMENTS – Question 2 every year and sometimes Question 3 also. Practice and be prepared!!! Tara Lyons

EXPERIMENT – TO PREPARE A SAMPLE OF SOAP.

Apparatus – reflux apparatus, heating mantle, conical flask.

Materials – lard or vegetable oil, sodium hydroxide pellets, ethanol, saturated sodium chloride solution, anti-bumping granules, water.



Method –

1. 5g of lard was placed in a pear-shaped flask.
2. To this about 4g of sodium hydroxide (excess) and 30 cm^3 of ethanol was added. **The ethanol acts as a solvent for the lard.**
3. Anti-bumping granules were added to the mixture.
4. The apparatus was set up for **reflux**. This ensures complete reaction without losing any volatile material.
5. The mixture was heated for about 30 min.
6. The flask was swirled from time to time to wash materials from the sides of the flask into the mixture.
7. After 30 min the heat was removed and the solution was allowed to cool.
8. The apparatus was rearranged for distillation and the ethanol solvent was distilled off. About 20 cm^3 of ethanol was collected. The ethanol is removed to isolate the soap as some soap may be dissolved in the ethanol and the soap would not precipitate fully to get a maximum yield.
9. The solution in the pear shaped flask was allowed to cool slightly.

10. This hot solution was then poured (decanted) quickly into a concentrated salt solution (brine). The **soap is insoluble** in the salt solution and so **precipitates out** of solution.
11. Using a Buchner funnel the soap was filtered through a weighed filter paper.
12. The soap was washed several more times with ice cold water to remove any sodium hydroxide solution clinging to the soap. This is important so that the soap does not burn the skin.
13. The soap was allowed to dry on a piece of filter paper. Reweigh and subtract to get the actual yield.

LEAVING CERT 2002 Q2

Soap is produced by the hydrolysis of vegetable and animal fats.

(a) What is the principal chemical difference between vegetable and animal fats?

A sample of soap was prepared in a school laboratory as follows:
Approximately 3 g of lard (animal fat), 2 g of sodium hydroxide pellets (an excess), and 25 cm^3 of ethanol were placed in a round-bottomed flask. A condenser was fitted to the flask and the mixture was refluxed gently for 20 minutes (diagram 1).

Following the reflux, the apparatus was allowed to cool slightly and the arrangement of the apparatus was changed so that the ethanol could be removed by distillation (diagram 2).

The residue from the distillation flask was then dissolved in a minimum of hot water and the solution decanted into 75 cm^3 of brine. The soap was then isolated.

(b) Apart from the lard, sodium hydroxide and ethanol, what else should be added to the reaction flask prior to the reflux?
Why was the mixture refluxed?
Why was the ethanol added?

(c) Why was it desirable to remove the ethanol after reflux?

(d) Why was a minimum of hot water used to dissolve the residue from the distillation? What is brine?

(e) Describe how the soap could be isolated from the mixture of soap and brine. Give one precaution that helps to ensure that the soap is free of sodium hydroxide.

ORGANIC CHEMISTRY EXPERIMENTS – Question 2 every year and sometimes Question 3 also. Practice and be prepared!!! **Tara Lyons**

LEAVING CERT 2006

A sample of soap was prepared in the laboratory by refluxing a mixture of approximately 5g of animal fat, 2g of sodium hydroxide pellets (an excess) and 25 cm^3 of ethanol in an apparatus like that drawn below.

(a) Why was the reaction mixture refluxed? Name the type of reaction which occurs during the reflux stage of the preparation. (8)

(b) Complete and balance the equation below for the reaction between glyceryl tristearate, an animal fat and sodium hydroxide. (9)

(c) What is the purpose of the ethanol. Why is it desirable to remove the ethanol after reflux? Describe with the aid of a diagram how you would remove the ethanol after the reflux stage of the experiment. (12)

(d) Describe how a pure sample of soap was obtained form the reaction mixture. (9)

(e) at the end of the experiment what is the location

 (i) of the second product of the reaction,

 (ii) of the excess sodium hydroxide? (6)

(f) What would you observe, upon shaking, if a little of the soap prepared in this experiment is added to (i) a test tube containing deionised water,

 (iii) a test tube containing mineral water from a limestone region? (6)

ORGANIC CHEMISTRY EXPERIMENTS – Question 2 every year and sometimes Question 3 also. Practice and be prepared!!! Tara Lyons

EXPERIMENT – THE EXTRACTION OF CLOVE OIL FROM CLOVES USING STEAM DISTILLATION.

Theory behind the experiment – Cloves consist of unopened flowers that are picked when the heads are pinkish in colour (not ripe). It is at this point in their growth that the oil is present in its highest concentration. **Eugenol** is the main constituent of clove oil and it contains a benzene ring in its structure.

Steam distillation is used, as it is a method that uses the principle that when two immiscible liquids are together their boiling point is lower than either of the two pure compounds alone.

If clove oil was distilled directly from ground cloves the cloves would have to be heated to their boiling point and this would cause them to become charred (burned) and it would be impossible to extract the oil. When clove oil is steam distilled we can get two layers, the clove oil and water. The layers can be separated using a separating funnel.

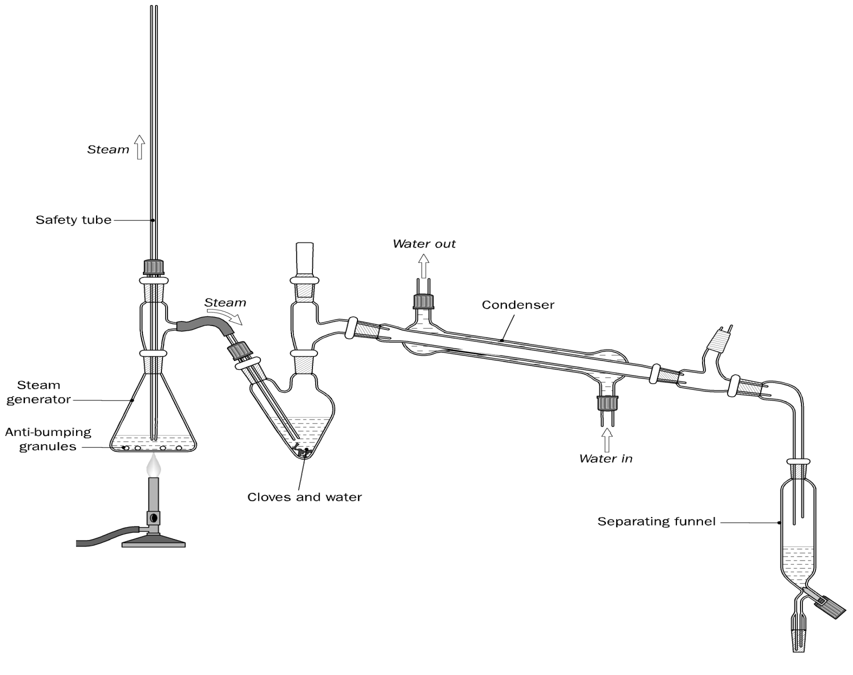
Solvent extraction

Usually instead of two separate layers the oil droplets can be dispersed throughout the water, this is called an **emulsion**. If this happens we can use an organic solvent such as cyclohexane that will dissolve the oil but not the water. The organic solvent is then separated from the water using a separating funnel and the organic solvent is allowed to evaporate off, leaving the oil behind. This is known as **solvent extraction.**

Apparatus – steam generator, safety tube, pear shaped flask, Liebeg condenser, heating mantle, rubber tubing, retort stands, beaker.

Materials – whole cloves, water, organic solvent.

Note – gloves should be worn as Eugenol can burn the skin.



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Method –

1. The apparatus was set up as is in the diagram for steam distillation.
2. Water was added to the steam generator.
3. The safety tube was placed into the steam generator **ensuring that the bottom of the safety tube was well below the level of the water in the generator. The water should be topped up regularly. The safety tube releases pressure/avoids build up of steam/ avoids explosion.**
4. Some cloves were place in the pear shaped flask with some water.
5. The Bunsen burner was turned on and steam was allowed to pass through the flask for about 40 minutes.
6. The clove oil **emulsion** passed through the condenser to the receiver, which is a separating funnel in this diagram. The emulsion has **a milky colour**.
7. The emulsion was allowed to cool in the separating funnel.

SOLVENT EXTRACTION

8. Some organic solvent was added i.e. cyclohexane and the contents of the separating funnel were shaken. **Solvent extraction** is the technique being used here.
9. The lower aqueous layer was allowed to run off into a beaker.
10. The organic layer was run into a second beaker.
11. The aqueous layer is washed several times with cyclohexane to maximise yield of the oil.
12. Release pressure carefully by opening the separating funnel when upside down.
13. Pour into a conical flask and add anhydrous magnesium sulphate (to dry) until it stops forming clumps.
14. Filter and then heat the filtrate gently in a fume cupboard to allow the cyclohexane evaporate.
15. This leaves the clove oil behind.

Precaution – 'suck-back' may occur when heat is removed so care is needed. How could you prevent this from occurring?

USES OF CLOVE OIL

1. Flavouring – spiced beef, sweets, pickles.
2. Antiseptic – soap, perfumes.

LEAVING CERT 2008

Steam distillation, using an apparatus like that shown, is a technique used to isolate an organic substance from plant material. The principle of this technique is that the boiling point of a mixture of two *immiscible liquids* is below the boiling points of both pure liquids. This allows the organic substance to be isolated at temperatures below 100 C and avoids the delicate organic molecules being damaged at high temperatures.

(c) What is meant by the term *immiscible liquids*? (3)
(d) Name a substance you isolated by steam distillation in the school laboratory and the plant material from which it was extracted. (6)
(e) Explain the function of the tube labelled X. (6)

(f) Describe the appearance of the distillate collected. Name or describe briefly a technique that could be used to separate the organic substance from the water. (9)
(g) In a steam distillation experiment 20.0g of plant material were heated in the presence of steam. Only 0.250g of pure organic liquid was obtained. Calculate the percentage yield. (5)

CHROMATOGRAPHY

This is a technique that is used to **separate** different substances in a **mixture.** You can also then identify and measure the amount of these substances present in the mixture using other instruments.

THE PRINCIPLE

The basic principle is that a mobile phase i.e. a solvent, will carry a dissolved mixture through a stationary phase i.e. paper or aluminium oxide. The separation occurs, as some of the components of the mixture tend to be held (attracted to) by the stationary phase and move more slowly than those that mix well with the mobile phase.

EXPERIMENT – TO SEPARATE A MIXTURE OF INDICATORS (or INKS) USING CHROMATOGRAPHY.

1. USING PAPER CHROMATOGRAPHY

Theory behind the experiment – a small amount of the indicator mixture is placed on a strip of filter paper (or chromatography paper). This paper is placed so that it just touches a suitable solvent. The paper absorbs the solvent and the solvent moves up through the paper and the indicator mixture dissolves in the solvent. The degree to which the components of the mixture dissolves in the solvent differs. The components that do not dissolve easily in the solvent will come out of the solution and appear on the paper as a spot. Those that do dissolve well will be carried up the paper further and will eventually appear as spots on the paper at different intervals. In this way the components will be separated.
Results – these are obtained by recording the R_f values which equals

$$R_f = \frac{Dis\tan ce \text{ travelled by the component}}{Dis\tan ce \text{ travelled by the solvent front}}$$

These values may be used to tentatively identify the components in a mixture of indicators.

Apparatus – chromatography paper, gas jar, ruler, pencil, sellotape, glass rod, hair dryer, capillary tubes.

Materials – a mixture of indicators, water/ethanol/ammonia solution (solvent).

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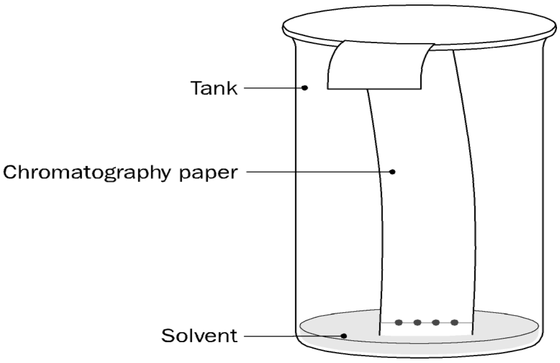


Method –

1. The solvent was added to the bottom of the gas jar to a depth of about 1 cm. This was allowed to stand for a while to allow the tank to become saturated with the solvent's vapour.
2. On the chromatography paper **a line** was made near the **top** of the paper with a pencil and **another line** near the **bottom**.
3. A small spot of the mixture of indicators was placed on the line at the bottom of the paper using the capillary tube. This was dried with a hair dryer to prevent the spot from spreading. This procedure was repeated several more times to build up **a small concentrated spot of the mixture.**
4. The paper with the 'spot side down' was then placed in the gas jar so that the end of the paper was just touching the solvent. **Do not immerse the spot.** Attach the top of the paper to a glass rod using the sellotape.
5. The chromatogram was allowed to run until the solvent reached the line at the top of the paper.
6. The paper was removed and dried.

Results –

Distance travelled by the solvent front –
Distance travelled by the fastest moving component of the mixture –
$R_f =$

Distance travelled by the second fastest moving component of the mixture –
$R_f =$

Distance travelled by the third fastest moving component of the mixture –
$R_f =$
Etc……………….

Questions on the experiment –

1. Why is the gas jar not used for a time after the solvent has been added?
2. Why are two lines drawn on the paper?
3. When is it possible to separate two components of a mixture using paper chromatography?
4. When two substances are found to have two different R_f values in an experiment carried out under the same conditions, what does this mean?

Answers –

1. To allow time for the tank to become saturated with solvent vapour.

2. One line is needed to indicate where the samples start from, and the other to indicate the distance travelled by the solvent front, which enables the R_f values to be calculated.
3. When one of the components is attracted to significantly different extents by the stationary phase and/or the mobile phase.
4. The two substances are not identical.

LEAVING CERT 2005

State the principle on which all chromatographic separation techniques are based. (10)

Describe with the aid of clearly labelled diagrams how you could carry out an experiment to separate a mixture of dyes (or indicators) using paper, thin-layer or column chromatography. (15)

EXPERIMENT - RECRYSTALLISATION OF BENZOIC ACID AND DETERMINATION OF ITS MELTING POINT.

Theory behind the experiment - this technique is used to **separate and purify** organic solids. It works on the **principle** that **more solute is soluble in a hot solvent than in a cold solvent**. The substance to be purified is dissolved in the minimum amount of hot solvent possible and then this is filtered to remove any insoluble impurities. This must be done quickly to prevent the solution from cooling. Then as the filtered solution cools, crystals appear. This is filtered and the crystals are dried using a desiccator or by leaving in a warm place and tested for purity by measuring their melting point.

Apparatus – beaker, Buchner funnel, vacuum pump, filter paper, melting point tubes, thermometer, Bunsen burner, clock glass.

Materials – benzoic acid, liquid paraffin.

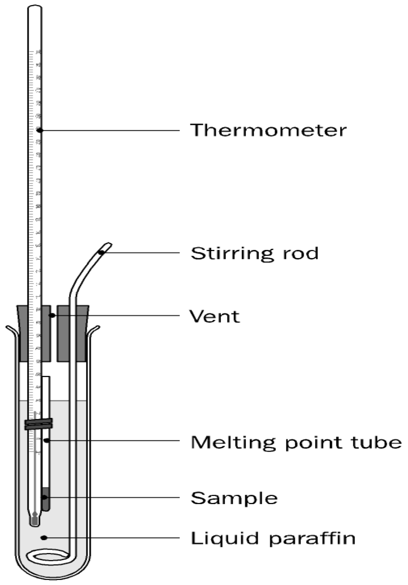
Method –

1. 5 g of benzoic acid was placed in a beaker.
2. This was dissolved in the **minimum** amount of hot water. **Minimum** so that the benzoic acid does not remain in solution on cooling to maximise the yield.
3. First filtration - This hot solution was filtered through a Buchner funnel (under suction, so, fast). Insoluble impurities remain on the filter paper.
4. The filtrate was allowed to cool slowly and fully to maximise the yield.
5. Second filtration - The excess solvent was filtered off and the crystals remain on the filter paper and were allowed to dry.

To determine the melting point of the benzoic acid crystals –

1. Some crystals were tapped into the open end of a melting point tube until there was about 0.5 cm of the crystals at the end of the tube.
2. Using a rubber band the tube was attached to a thermometer as shown below.
3. A boiling tube was half filled with liquid paraffin and the tube and thermometer were placed in the boiling tube.
4. This was placed over a Bunsen burner and heated. The liquid paraffin was stirred thoroughly throughout.
5. The temperature at which the crystals started to melt was noted and recorded.
6. The paraffin was allowed to cool about 10 C and a fresh melting point tube with crystals was put in the boiling tube.

7. The paraffin was heated again, more slowly this time and the temperature at which the crystals melted was again noted. Melting is indicated by the formation of a visible meniscus.



Note – if the crystals are **pure** then a **SHARP** melting point should be obtained. If the crystals are IMPURE then a melting point range will be obtained.

LEAVING CERT 2004

(a) A sample of impure benzoic acid was recrystallised as follows: 2.5g of the impure benzoic acid was weighed out and dissolved in the minimum amount of hot water. The hot solution was filtered and the filtrate was allowed to cool and recrystallise. The recrystallised benzoic acid was isolated by filtration. After drying, 2.25g of purified acid were obtained.

- (i) Why is it important to use the minimum amount of hot water in the procedure? (5)
- (ii) Indicate clearly the stage of the recrystallisation procedure at which insoluble impurities were removed and how their removal was achieved. Indicate also the stage at which soluble impurities were removed and how their removal was achieved. (12)
- (iii) How could you have ensured that the recrystallisation was complete? (3)
- (iv) How could the crystals have been dried? (3)
- (v) What was the percentage yield of the purified benzoic acid? (3)

(b) Melting points of samples of the impure and recrystallised benzoic acid were taken and compared.

- (i) Describe with the aid of a labelled diagram how you would have measured the melting point of one of these samples. (15)

(ii) Give two ways in which you would expect the melting point of the impure benzoic acid to differ from that of the purified acid. (6)
(iii) State one use of benzoic acid and its salts. (3)

MANDATORY EXPERIMENT – PREPARATION OF BENZOIC ACID OXIDATION OF PHENYLMETHANOL (BENZYL ALCOHOL) TO BENZOIC ACID WITH POTASSIUM MANGANATE (VII) SOLUTION IN ALKALINE CONDITIONS.

Theory behind the experiment – an alcohol is oxidized in alkaline conditions to an aldehyde and this then is oxidized to become a carboxylic acid. The alcohol used is phenyl methanol (benzyl alcohol). The oxidising agent is potassium manganate (VII) and the alkaline conditions are provided by the use of sodium carbonate. Excess potassium manganate (VII) is used to ensure complete oxidation to the carboxylic acid; otherwise we will end up with phenyl methanal. The potassium manganate (VII) will itself be reduced to a brown precipitate of MnO_2. Sodium carbonate is used, as the rate of the reaction will increase in alkaline solution. Hydrochloric acid is now added to (i)precipitate the crystals and (ii)to neutralize the sodium carbonate used and the potassium hydroxide produced. It also (iii) protonates the benzoate anion to form benzoic acid as the solution cools.
To prevent the benzoic crystals from being contaminated by the brown precipitate of MnO_2, sodium sulfite solution is used as it reduces Mn(+4) to Mn (+2) which is colourless and in solution. This requires heat in a water bath.
Recrystallization of the crystals takes place and these are then isolated and purified. The purity can be then determined by finding the melting point of the crystals.

Equation for the reaction:

$$3C_6H_5CH_2OH + 4KMnO_4 \rightarrow 3C_6H_5COOH + H_2O + 4KOH + 4MnO_2$$

Method -

1. 1cm^3 of phenylmethanol, a colourless, oily liquid, was added to a conical flask.
2. Then 25cm^3 of excess (saturated) $KMnO_4$ and 0.5grams of sodium carbonate to the conical flask. Excess is needed to ensure complete oxidation to the carboxylic acid, benzoic acid. Gloves should be worn when using concentrated $KMnO_4$.
3. Sodium carbonate is used as the rate of reaction increases in alkaline conditions.
4. If complete oxidation does not happen the smell of almonds will be noticed, as the alcohol will only be oxidized to phenylmethanal.
5. The flask was placed in a water bath for 20 minutes. This aids full oxidation to benzoic acid.
6. Notice the colour change of purple (Mn +7) to dark brown precipitate (Mn+4).
7. If after 20 minutes there is still a smell of almonds, heat some more to complete the oxidation process.
8. 4cm^3 of HCl is added drop by drop in a fume cupboard (as HCl gas is given off). The HCl helps precipitate the crystals of benzoic acid, it neutralizes the excess sodium carbonate used at the beginning and the KOH that is produced.

9. A glass rod was dipped into the crystals and this was then touched with blue litmus paper. The paper should turn red due to presence of benzoic acid crystals. (If litmus doesn't turn red then aldehyde may be present instead).
10. To prevent contamination of crystals with brown MnO_2, the MnO_2 is dissolved in $10cm^3$ of 1.5M sodium sulfite solution. Heat if necessary to aid dissolving.
11. When all the MnO_2 has dissolved, the conical flask was placed on ice and the benzoic acid recrystallizes.
12. The crystals were filtered. They were washed with ice water to maximize the yield. (Warm water would dissolve the crystals and reduce yield).
13. The slower the cooling, the bigger the crystals and the faster the cooling the smaller the crystals.
14. Crystals are dried in a desiccator or warm oven.
15. The purity of the crystals is obtained by determining the melting point.

SAMPLE QUESTION

Phenylmethanol reacts in the presence of potassium manganate (VII) , to produce benzoic acid according to the following equation:

$$3C_6H_5CH_2OH + 4KMnO_4 \rightarrow 3C_6H_5COOH + H_2O + 4KOH + 4MnO_2$$

If 2 cm^3 of phenylmethanol (density = $1.04g/cm^3$) and 6.32 grams of $KMnO_4$ is used (i) show clearly that the potassium manganate (VII) is in excess and (ii) calculate the % yield given that 1.05 grams of benzoic acid was produced.