

Subject: Chemistry

TEACHER: Tara Lyons

COURSE: Leaving Certificate

ACADEMIC LEVEL: Higher Level

ACADEMIC YEAR: 2021 - 2022

***TOPIC: ORGANIC CHEMISTRY 8 – Base
hydrolysis of an ester, saponification.
Preparation of Soap experiment.***



CONTENTS

ESTERIFICATION	1
BASE HYDROLYSIS OF AN ESTER	1
PREPARATION OF SOAP	2
WORK SHEET	4



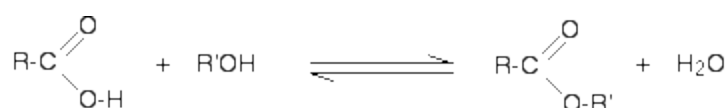
ESTERIFICATION

This type of reaction involves the reaction between a **carboxylic acid** and an **alcohol** to produce an **ester** and **water**. It is a reversible reaction and so at equilibrium it is affected by changes in temperature, and concentration (not pressure as it is not a gaseous system at room temp and pressure).

As an **ester** is a molecule that is formed when the hydrogen of the functional group of a carboxylic acid is replaced by an alkyl radical of an alcohol.

We should recall the reaction is also a **condensation reaction** as it involves the reaction of two different molecules to produce a more complex molecule, with the production of a smaller molecule such as water.

If we consider the reaction at equilibrium –



- The **forward reaction** is an **esterification** reaction.
- The **reverse** reaction is an **hydrolysis** reaction.

BASE HYDROLYSIS OF AN ESTER

Esters react with bases such as *KOH* or *NaOH* to form 2 products

- the alcohol and
- the salt of the carboxylic acid.

This reaction is used in the manufacture of soap, as the **salt of the carboxylic acid is soap**. As a result this reaction can also be called **SAPONIFICATION**.

SOAP

Soap is made when fats, which are natural esters are heated with bases such as potassium hydroxide or sodium hydroxide (potassium hydroxide is more soluble in ethanol than sodium hydroxide) to form the alcohol, propan-1,2,3-triol and the salts of a long chain carboxylic acid. These salts are soap.

- The fat used is the ester **glyceryl tristearate**, found in animal fat. Animal fat is saturated fat. Vegetable fat is unsaturated fat.
- The alcohol produced by base hydrolysis is called **propan-1,2,3-triol** or **glycerol**.
- The salts of the long chain carboxylic acid are called **potassium/sodium stearate**, soap!

CLEANING ACTION OF SOAP

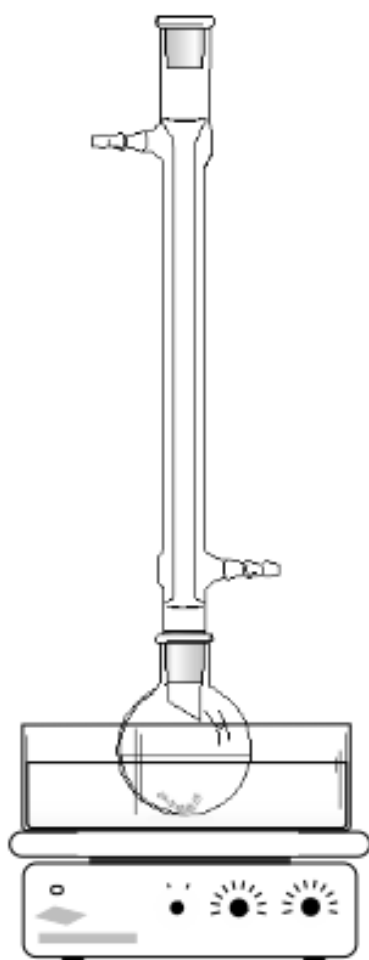
As soap consists of a large non-polar part ($\text{C}_{17}\text{H}_{35}$) and a smaller polar/ionic part ($\text{COO}^- \text{Na}^+$) we can see **how soap cleans our skin**. The oils that our skin produces dissolve in the non-polar carbon chain of the soap and the salts that our skin produces dissolve in the polar functional group.



EXPERIMENT – TO PREPARE A SAMPLE OF SOAP.

Apparatus – reflux apparatus, heating mantle, conical flask.

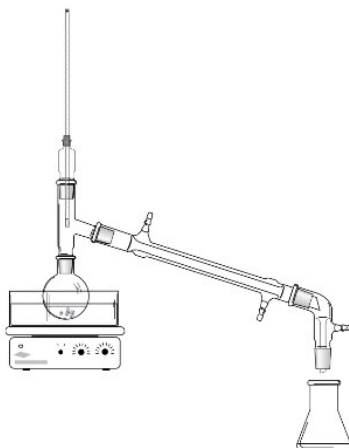
Materials – lard or vegetable oil, sodium hydroxide pellets, ethanol, saturated sodium chloride solution (BRINE), anti-bumping granules, water.



Method –

1. 5g of lard (limiting reactant) was placed in a pear-shaped flask.
2. To this about 4g of sodium hydroxide or potassium 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. These absorb heat from the reaction thus preventing violent shaking of apparatus.
4. **The apparatus was set up for reflux. This ensures (i) time to speed up the reaction (ii) for the complete saponification reaction (iii) without losing any volatile material.**
5. The mixture was heated under reflux for about 30 min.
6. The flask was swirled from time to time to wash materials (residue) from the sides of the flask into the mixture, or wash down the residue with a **minimum** of hot water. (Too much hot water will reduce the yield of soap as some will remain dissolved in it).
7. After 30 min the heat was removed and the solution was allowed to cool slightly. (list the contents of the reaction vessel at this point).
8. To isolate the soap the apparatus was rearranged for distillation and the roughly two-thirds ethanol solvent was distilled off. About 20 cm^3 of ethanol were 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. (same contents here but less ethanol, so more concentrated soap mixture).

Distillation apparatus-



9. The solution in the flask was allowed to cool slightly.
10. This hot solution was then decanted quickly into a concentrated salt solution (brine). The **soap is insoluble** in the salt solution and so **precipitates out** of solution. (antibumping granules stay behind as decanting.)
11. Using a Buchner funnel the soap was filtered. (weighed filter paper)
12. The soap was washed several more times using 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 or in a dessicator.
14. The soap is weighed and this is the actual yield. Use moles of lard to determine the theoretical yield. Calculate the % yield.

Questions –

1. How do you explain the cleansing action of soap?
2. Name the compound $\text{CH}_2(\text{OH})\text{CH}(\text{OH})\text{CH}_2(\text{OH})$
3. Name the acids $\text{C}_{17}\text{H}_{35}\text{COOH}$ and $\text{C}_{15}\text{H}_{31}\text{COOH}$

ANSWER - $\text{C}_{17}\text{H}_{35}\text{COOH}$ is stearic acid and $\text{C}_{15}\text{H}_{31}\text{COOH}$ is palmitic acid.

4. Glycerol is a by-product of the reaction in which soap is made. This does not distil over with the ethanol after the reaction is complete, and remains dissolved in the hot water when added to brine to precipitate the soap. Explain why. (hint - think water is a polar solvent and glycerol contains 3 polar –OH groups)



WORK SHEET

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.



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.

- Why was the reaction mixture refluxed? Name the type of reaction which occurs during the reflux stage of the preparation. (8)
- Complete and balance the equation below for the reaction between glyceryl tristearate, an animal fat and sodium hydroxide. (9)



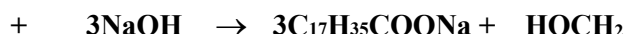
glyceryl tristearate

- 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)
- Describe how a pure sample of soap was obtained from the reaction mixture. (9)
- at the end of the experiment what is the location
 - of the second product of the reaction,
 - of the excess sodium hydroxide? (6)
- 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,
 - a test tube containing mineral water from a limestone region? (6)



LEAVING CERT 2010

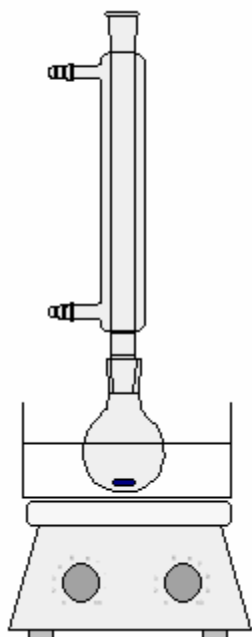
To prepare soap, a student refluxed 8.9 g of glyceryl tristearate (obtained from animal fat), 2 g of sodium hydroxide pellets and 30 cm³ of ethanol, together with a few anti-bumping granules, using the apparatus shown on the right. At the end of the experiment 7.0 g of pure, dry soap were isolated. The balanced equation for the reaction is as follows:



GLYCERYL TRISTEARATE

SODIUM STEARATE

GLYCEROL



(a) Write the systematic (IUPAC) name for glycerol. (5)

(b) Why was the reaction mixture heated under reflux?

Name the type of reaction that occurred during the reflux.

What was the purpose of the ethanol? (12)

(c) (i) Describe, with the aid of labelled diagrams, how the ethanol was removed after the reflux stage.

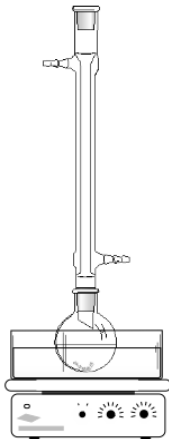
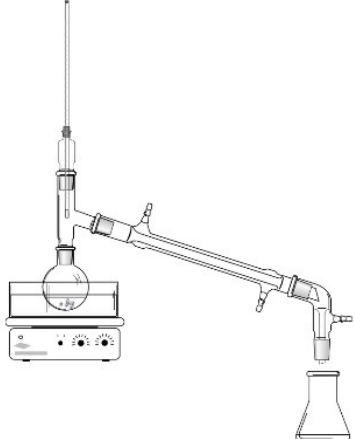
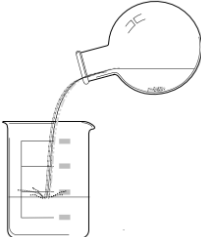
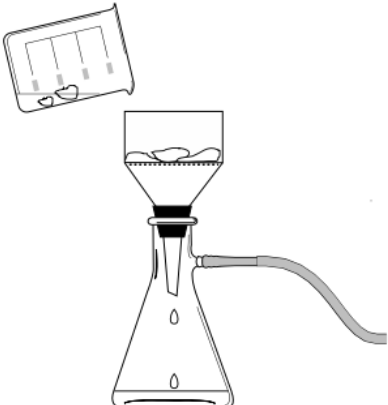
(ii) How was the soap isolated from the other substances left in the reaction mixture?

(iii) After isolating the soap, how was it purified and dried? (21)

(d) Given that the sodium hydroxide was in excess, calculate the percentage yield of soap (sodium stearate). (12)



Diagrams for Question 2 2014

Stage 1 Reflux	Stage 2 Distil	Stage 3 Decant	Stage 4 Filter
			

LEAVING CERT 2014

A student prepared a sample of soap in the school laboratory. The experiment was carried out in the four stages illustrated below. At Stage 1, using a water bath, the student refluxed for approximately 20 minutes 4.45 g of glyceryl tristearate (an animal fat) together with an excess of sodium hydroxide pellets, anti-bumping material and about 30 cm³ of ethanol. The reaction shown in the following balanced equation took place.



The apparatus was then allowed to cool and rearranged for Stage 2, distillation, again using a water bath. After distillation, the contents of the distillation flask were decanted or washed into a beaker containing brine – Stage 3. Filtration was used in Stage 4 to isolate the soap which was then thoroughly washed.

- (a) What is the purpose of refluxing in Stage 1 of the preparation? Name the type of reaction that occurred during this stage. (8)
- (b) What substance was removed by distillation in Stage 2? (3)
- (c) Explain the function of the brine in Stage 3. (6)
- (d) Why was it necessary to wash the soap thoroughly in Stage 4? How should the student have washed the soap? (6)
- (e) Draw the structure or give the name of the co-product of the reaction. Where was the co-product located at the end of the process? (9)
- (f) Given that the sodium hydroxide was in excess, calculate the maximum yield in grams of soap that could have been obtained in this preparation. (12)
- (g) Suggest, with reference to its structure, how a soap like sodium stearate can dissolve both the non-polar oils *and* the ionic salts in sweat from the skin. (6)



ADDITIONAL QUESTIONS

1. Write a balanced equation between methyl propanoate and sodium hydroxide. Name the products of the reaction. What term is used to describe this reaction/
2. Explain why an esterification reaction may also be referred to as a condensation reaction.

