Formula Weight of a Fatty Acid

INTRODUCTION

Saturated fatty acids, the focus of this experiment, have the general formula

$$CH_3(CH_2)_nCOOH$$

and consist of long carbon chains with a carboxyl group at one end. Unsaturated fatty acids must contain at least one double bond in the long carbon chain and therefore do not contain the maximum number of hydrogens. Saturated fatty acids do not contain a double bond in the long carbon chain and contain the maximum number of hydrogens as shown above. Fatty acids are rarely found in the "free" form in cells but are rather one part of more complex molecules such as triglycerides, waxes orphosphotriglycerides.

Free fatty acids find many uses in the manufacture of soaps, as additives to increase the viscosity of cosmetics, as chemicals for the treatment of textiles, and as additives to lubricants, rubbers, and dyes.

Examples of fatty acids from natural triglycerides, their names, their formulas and their melting points are listed in Table 1.

Because fatty acids are acidic, they will react with bases. The acidic hydrogen in a fatty acid is the hydrogen that is part of the carboxyl group. The reaction of an acid with a base is called a neutralization reaction. This neutralization reaction can be used to find the formula weight or molecular mass of fatty acid, or the average formula weight of a weight determined by titrating the unknown with an aqueous sodium hydroxide solution of known molarity.

DETERMINATION OF FORMULA WEIGHT OF FATTY ACIDS

Fatty acids have one acidic hydrogen per molecule. The neutralization reaction occurring between a fatty acid and sodium hydroxide is described by Equation 1 or in net-ionic form by Equation 2.

$$\underset{\text{Fatty acid}}{CH_3(CH_2)_nCOOH} + \underset{\text{base}}{NaOH} \rightarrow \underset{\text{soap}}{CH_3(CH_2)_nCOONa} + \underset{\text{water}}{H_2O} \qquad (1)$$

$$CH_3(CH_2)_nCOOH + OH^- \rightarrow CH_3(CH_2)_nCOO^- + H_2O \qquad (2)$$

Table 1 Some saturated fatty acids from natural fats and oils.

Common Name	**Formula**	**m.p. (°C)**	**Obtained by hydrolysis of glyceryl esters found in**
Butyric	$CH_3(CH_2)_2CO_2H$	-6	Butter
Caproic	$CH_3(CH_2)_4CO_2H$	-3	Butter and goat fat
Caprylic	$CH_3(CH_2)_6CO_2H$	16	Butter, goat fat, and coconut oil
Capric	$CH_3(CH_2)_8CO_2H$	31	Butter, goat fat, and coconut oil
lauric	$CH_3(CH_2)_{10}CO_2H$	44	coconut oil and palm oil
myristic	$CH_3(CH_2)_{12}CO_2H$	54	Butter, coconut oil, palm oil, beef tallow, and cod liver oil
palmitic	$CH_3(CH_2)_{14}CO_2H$	63	All of the above plus corn oil, lard, olive oil, peanut oil, and linseed oil.
stearic	$CH_3(CH_2)_{16}CO_2H$	70	All of the above.

When a sodium hydroxide solution is added to a solution of the fatty acid, the OH^- from the added base and the H^+ ions from the fatty acid combine to form water. When all the acid has been neutralized **at the equivalence-point** of the titration, additional base will **just** increase the hydroxide concentration ion concentration because H^+ ions are no longer present to combine with OH^-. Phenolphthalein, an indicator , reacts with these excess hydroxide ions and makes the solution purple-red. Phenolphthalein, before its reaction with OH^-, is, colorless. The appearance of the pink color therefore indicates the neutralization of the acid is complete and that **the endpoint** of the reaction has been reached.

When the molarity of the sodium hydroxide solution is known and the volume of the sodium hydroxide that was needed to reach the endpoint was carefully measured by dispensing the solution from a buret, the moles of sodium hydroxide consumed in the reaction can be calculated Equation 3.

$$\text{moles(NaOH)} = \text{M(molarity NaOH)} \times \text{Volume(of NaOH in liters)} \quad (3)$$

Because each mole of sodium hydroxide consumed in the neutralization reaction(Equation 1) requires one mole of fatty acid, the number of moles of sodium hydroxide calculated according to Equation 3 is equal to the number of moles of carboxylic acid neutralized.

The mass of the fatty acid used in the titration is known because the sample was weighed on an analytical balance before it was titrated. The mass of the fatty acid and the number of moles of fatty acid allow the formula weight to be calculated (Equation 4).

$$\text{formula weight} = \frac{\text{mass in grams of fatty acid}}{\text{number of moles of fatty acid}} = \text{g/mole} \qquad (4)$$

When the formula weight is known, the “n” in $CH_3(CH_2)_nCO_2H$ can be calculated using equation 5.

$$n = \frac{\text{formula weight - 60.06}}{14.03} \qquad (5)$$

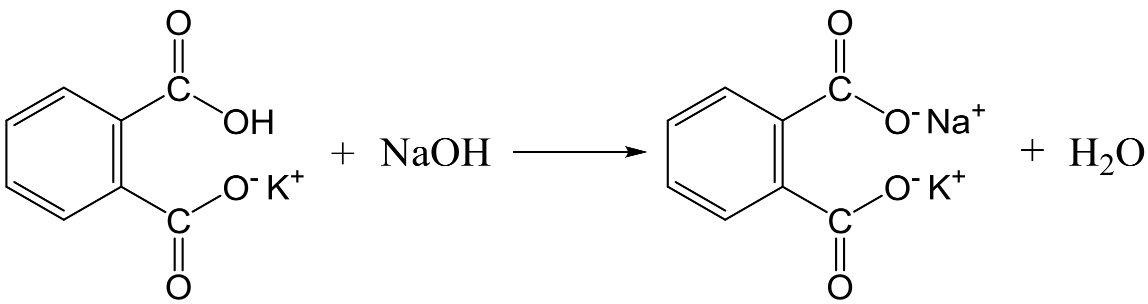
When you have identified “n”, you can write a formula for your unknown and use Table 1 to find the name for the fatty acid. Because errors are always associated with experimental work, the calculated "n" very likely will not be an integer. You will have to round your answer. When attempting to identify your unknown, keep in mind that natural fatty acids almost always have an even number of carbon atoms in the molecule.

STANDARDIZATION OF SODIUM HYDROXIDE

One additional question needs to be considered. Why must the sodium hydroxide concentration be accurately known or the solution standardized? Sodium hydroxide is a rather reactive substance. If you leave a pellet of sodium hydroxide in moist air, you will notice a liquid film forming on the surface of the pellet. Sodium hydroxide is deliquescent (it absorbs water from the air) and reacts with carbon dioxide present in the air. For these reasons, **sodium hydroxide is never pure when handled in air**. Therefore, sodium hydroxide solutions are prepared by weighing out an approximate amount of solid sodium hydroxide and dissolving it in an approximate volume of distilled water. Graduated cylinders serve well for measuring the volume. The use of a volumetric flask is not necessary. The exact molarity of this solution is determined by standardization with an acid that can easily be obtained in high purity, will not absorb water, and will not react with carbon dioxide or other constituents of the atmosphere, all desirable traits of a primary standard.

Potassium hydrogen phthalate (KHP is such a primary standard. The amount required for the standardization of a sodium hydroxide solution is carefully weighed on an analytical balance. The sample is dissolved in hot water, phenolphthalein is added, and sodium hydroxide solution added until the endpoint is reached. At this point equal moles of acid and base have reacted (Equation 6).

$$KHC_8H_4O_4\ (aq)\ +\ NaOH\ (aq)\ \rightarrow\ KNaC_8H_4O_4\ (aq)\ +\ H_2O\ (l) \quad (6)$$



$KHC_8H_4O_4$

KHP

Potassium hydrogen phthalate (KHP) as shown in this representation has a six-sided ring of single and double bonded carbon atoms representing the benzene-like portion of the compound. The ring has a carbon atom at each corner. A hydrogen atom is attached to each carbon atom that does not already have an attached oxygen atom or more than two carbon atoms. A very condensed formula of KHP is $C_8H_5O_4K$.

To calculate the molarity of a sodium hydroxide solution, M_{NaOH}, it must be first determined how much KHP, the standard, is used in the titration. This can be easily determined from the mass of KHP used per titration and the molar mass of KHP. Since the moles of KHP equals the moles of NaOH, the moles of NaOH are known. When the moles of NaOH are divided by the volume of the sodium hydroxide solution to obtain neutralization, the molarity of the NaOH is obtained.

Procedure

SAFETY AND WASTE DISPOSAL

Wear approved eye protection at all times in the laboratory. Sodium hydroxide is a strong base; handle it carefully and avoid contact with your skin. If contact has occurred, wash with plenty of tap water. Use caution when heating ethanol, a flammable liquid.

ACTIVITIES

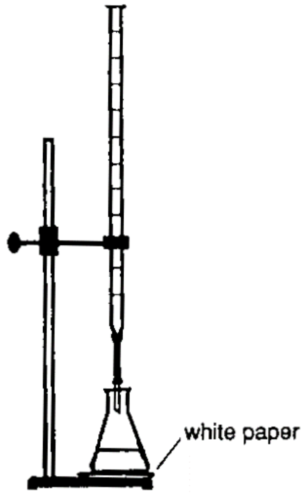
1. Prepare 300 mL of a sodium hydroxide solution of approximately 0.1 molar concentration.
2. Standardize the sodium hydroxide solution using potassium hydrogen phthalate.
3. Determine the formula weight of an unknown fatty acid.
4. Identify the fatty acid.

1. PREPARATION OF 300 mL OF A 0.1 M NaOH SOLUTION

Using the dilution law, $M_1 \times V_1 = M_2 \times V_2$ determine the volume of stock sodium hydroxide solution provided to prepare to 300. mL of a 0.1 M solution. Check the volume with your lab instructor.

Take a clean, but not necessarily dry, 50- or 25-mL graduated cylinder to the reagent area and obtain the volume of stock sodium hydroxide calculated for the preparation of the needed ~0.1 M NaOH solution. Add the stock NaOH solution to a 500-mL beaker containing 100 mL distilled water. Swirl the flask carefully. Use 25 mL of water to rinse the graduated cylinder. Add this wash to the flask. Then add additional distilled water to the flask until a total volume of 300 mL is obtained. Mix thoroughly by carefully swirling. Label it "~0.1 M NaOH".

Check that your 50-mL buret is clean, free of grease, and draining properly. Rinse the buret three times with 5 mL of your -0.1 M NaOH solution. Then fill the buret with the NaOH solution above the O-mL mark. Drain some of the solution from the buret to expel air from the buret tip. Allow the liquid level to drop just below the zero mark.



2. STANDARDIZATION OF THE 0.1 M NaOH SOLUTION

Determine the mass of potassium hydrogen phthalate needed to neutralize 30 mL of a ~0.1 M NaOH solution. If you have doubts about your calculation, check with your instructor.

In the balance room tare the mass of a weigh boat on an analytical balance. Carefully add approximately this mass of potassium hydrogen phthalate needed to neutralize 30 mL of a 0.1 M NaOH solution. The mass actually obtained should not be exactly the amount you needed. The mass can be ±20.0 mg of the calculated amount. If the mass is outside this limit, add or remove some of the phthalate. Remember that the balance must be clean when you leave it. Record the mass of your sample.

Transfer the weighed phthalate sample into a clean 250-mL Erlenmeyer flask. Add approximately 100 mL distilled water to the flask. Carefully swirl and gently warm the flask until all the phthalate has dissolved. Then remove the flask from the hotplate.

Add three drops of phenolphthalein indicator to the phthalate solution in the flask and set it on a white paper under the buret.

To the nearest 0.01 mL, read and record the volume level of your ~0.1 M NaOH solution in the buret. Slowly add the ~0.1 M NaOH solution to the phthalate solution until a permanent pale pink endpoint is observed. Record the final buret reading. Clean the flask and rinse it with distilled water. Repeat the standardization if time allows.

3. DETERMINATION OF THE FORMULA WEIGHT OF A FATTY ACID

From your instructor obtain an unknown which should be placed into a clean, dry 100-mL beaker. This unknown could be the fatty acid prepared from soap or another solid carboxylic acid. Ask your instructor how much of your unknown you should use for a titration. Record the unknown code in your datasheet.

Weigh out the suggested amount of your unknown on the analytical balance to 0.1 mg using a weigh boat. The amount of unknown you use may deviate by ±20.0 mg from the suggested amount. Transfer the sample quantitatively into a clean 250-mL Erlenmeyer flask. Add 50 mL of 95- percent ethanol. Carefully heat the mixture on a hotplate until the unknown has dissolved. If the unknown has not dissolved when the ethanol boils, remove, add 20 mL of ethanol and reheat. **Caution: Ethanol is flammable!**

When all the unknown has dissolved, add three drops of phenolphthalein to the solution and titrate with your NaOH solution to a pale pink endpoint. Record the initial and final buret readings. Calculate the formula weight of your unknown. Repeat the titration with another sample of the same unknown.

If you titrated your unknown more than once, calculate the average formula weight of your unknown. On the basis of your average formula weight, suggest a formula for your unknown assuming that the unknown has the general formula $CH_3(CH_2)_nCOOH$Consult Table 1 for the formulas of possible fatty acids. Is your unknown identical to any of the compounds listed in Table 1? If more than one formula appears possible for your unknown, suggest experiments that would allow you to distinguish among them.

Before you leave lab, you should request that your instructor clarify any question you have on such topics as: standardization, primary standard, titration, titrant, aliquot, indicator, end point, equivalence point, acid base titration, etc.

Complete and hand in the data sheet before leaving the lab. Be sure to include all calculations and watch significant figures when reporting your final results.

NOTE:

Ensure that the burets are washed thoroughly after being filled with NaOH or the stopcocks will freeze and ruin the buret.

FATTY ACIDS QUIZ HINTS – KNOW HOW TO ANSWER QUESTIONS LIKE THE FOLLOWING:

1. Define in your own words: Molarity (no equations, please), Acid, Base, Titration, equivalence point, endpoint, indicator, fatty acid.

2. Calculate the volume of ~1.5 M NaOH solution needed to prepare 300 mL (2 significant figures) of a ~0.10 M NaOH solution.

3. Calculate the mass of solid sodium hydroxide needed to prepare 300 mL (2 significant figures) of a 0.1 M solution (1 significant figure). If one pellet of sodium hydroxide weighs 0.170 g (3 significant figures), how many pellets should be dissolved?

4. Write a balanced equation for the neutralization of potassium hydrogen phthalate ($C_8H_50_4K$) with sodium hydroxide. Calculate the mass of potassium hydrogen phthalate that neutralizes 30 mL of an ~0.1 M NaOH solution.

5. A sample of a liquid carboxylic (fatty) acid weighing 0.2131 g required 36.50 mL of a 0.0973 M aqueous sodium hydroxide solution to make the pink color of phenolphthalein appear. Calculate the formula weight of the acid, find the formula assuming the acid belongs to the series of carboxylic acids of general formula $CH_3(CH_2)_nCOOH$, and identify the acid by name, if it is one of those in Table 1.

6. Draw a structural representation of potassium hydrogen phthalate that shows all its hydrogen atoms.

7. What are several of the properties that one looks for when selecting a primary standard? Why is a solution of NaOH which was standardized yesterday unsuitable to be used as a standard solution today?