FATTY ACIDS FOR SOAP MAKING

INTRODUCTION

Description

Students titrate a sample of fatty acids with standardized sodium hydroxide solution. From the amount of base needed for neutralization and the mass of sample used for titration, the apparent average molar mass of the sample is determined. Students use their results to determine whether or not their sample matched the fatty acid ratio best suited for soap making.

Goals for This Experiment

The goals for this experiment are to have students:

1. titrate an unknown sample,
2. determine the moles of sodium hydroxide used to titrate a fatty acid sample, and
3. determine whether or not the unknown sample contains the desired 80:20 ratio of tallow fatty acids to coco fatty acids.

Recommended Placement in the Curriculum

We used the Fatty Acids for Soap Making scenario lab as the first titration, after the students had experience with measuring volumes with burets.

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SCENARIO

Imagine that several carloads of soap-making fatty acid stock mixture were being sold at an auction for a reduced price. In order to use this stock in your current soap plant, you need to know if it has the ideal 80:20 ratio of tallow fatty acids to coco fatty acids. To find out, you decide to titrate a sample of the stock mixture with sodium hydroxide solution. Such a titration should work because the fatty acids react quickly and completely with an aqueous solution of sodium hydroxide (as long as the fatty acid remains in solution). At the end point of the titration, the number of moles of sodium hydroxide is equal to the number of moles of fatty acid.

BACKGROUND

Bar soaps are usually composed of mixtures that are about 80% tallowate (a mixture of sodium salts of fatty acids derived from tallow, in which the most abundant ingredient is sodium stearate) and 20% cocoate (a mixture of sodium salts of fatty acids derived from coconut, in which the most abundant ingredient is sodium laurate). The tallowate helps provide the soap bar its firmness, and the cocoate provides lather. The sodium salts for soaps are made from the corresponding fatty acid mixtures.

YOUR TASK

Your task in this experiment is to determine the tallow fatty acid-coco fatty acid ratio in the sample.

PROCEDURE

The following procedure describes one analysis. At least four analyses should be done. The four determinations should not differ from the mean value by more than ±1.2 %. If your range is greater than this, consult your instructor for how to proceed.

The sodium hydroxide solution provided in lab will have been standardized prior to use. (Its approximate concentration is 0.2 moles/liter.) Be sure to record the actual concentration from the stock bottle *using the appropriate number of significant figures.*

1. Prepare the 50-mL buret you will use for the titration by rinsing with just a little standardized sodium hydroxide solution. Drain. Repeat.

2. Fill the rinsed buret with standardized sodium hydroxide solution. Carefully read, to the nearest 0.01 mL, the beginning volume on the buret. Record this initial reading.

3. Weigh accurately to the nearest 0.01 g and record the actual mass of about 2 grams of soap stock into a 250-mL Erlenmeyer flask.

4. Add 50 mL of ethanol to the soap stock and heat to boiling on a hot plate set on medium low. Holding the neck of the flask with your tongs or with a paper collar, cautiously swirl the

mixture occasionally. By keeping the heat low, the chance of charring and fire will be reduced. Once the soap stock is in solution and the mixture is near boiling it is ready to be titrated.

CAUTION: BE CAREFUL WITH THE HOTPLATE, GLASSWARE, AND VAPORS SO YOU DO NOT BURN YOURSELF. ETHANOL IS FLAMMABLE. DO NOT LEAVE THE FLASK UNATTENDED. DO NOT ALLOW THE SOLUTION TO BOIL DRY. THE SOAP STOCK WILL CHAR IN A BLACK TAR ON THE BOTTOM OF THE FLASK.

5. Add 5–10 drops of phenolphthalein indicator solution to the hot ethanol-soap stock solution.

6. Titrate the dissolved fatty acid-ethanol mixture with the standardized sodium hydroxide solution. Using a paper collar to protect your hands, cautiously swirl the flask as the titrant is added. Stop the titration at the first **faint** pink color which appears and persists for 30 seconds. Record the reading of the volume of the buret at the end of the titration. It will be necessary to slow the addition of titrant to dropwise additions as the end point is reached. (Some chemists do a quick titration to find the approximate volume to add to a sample and then follow that with careful titrations with dropwise additions at the end of those titrations. The quick titration is not used in the determination of the mean value, but if you do one, it must be recorded in your notebook.)

   Note: If at any point during the titration the solution turns cloudy or crystals appear in the titration flask, the titration should be interrupted and the flask returned to the hot plate and its contents heated and swirled until the material is back in solution. The titration may then be resumed.

7. Repeat the titration procedure for each of the other samples.

✏ Be sure to record:
- the sample number of soap stock analyzed,
- the mass of soap stock titrated in each titration,
- the standardized concentration value of sodium hydroxide solution, and
- the beginning and end readings of the buret in each titration.

Disposal
The contents of the titration flask will be collected in a large waste container. It turns into a gel at about 30°C and can then be disposed of in the trash.

You may want to put a little of the titrated material aside to gel and observe its properties in water. Does it act like consumer soap?

Use of the Titration Data
The number of moles of sodium hydroxide needed to react with the same number of moles of fatty acid can be determined by using the following relationship:

Moles of NaOH added = (Volume of NaOH titrant added) x (Concentration of NaOH)

From the mass of the fatty acid sample and the number of moles represented, the molar mass of the soap stock can be determined and be compared to that of an idealized average molar mass of 80% stearic acid $\{C_{18}H_{36}O_2\}$ and 20% lauric acid $\{C_{12}H_{24}O_2\}$ by mass.

Results and Questions
Q 1: What is the average molar mass of the supposed soap stock in the barrel you analyzed?
Q 2: Would you recommend that a company buy the supposed soap stock at auction? Why? or Why not?
Q 3: If some of the material came out of solution during the titration, how would the results be affected?

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INSTRUCTOR NOTES

Time Required
The approximate time to complete this activity in the lab, including pre-lab discussion, is $2^1/_2$–3 hours, depending on the students' abilities to titrate.

Group Size
Students can work individually to practice the skill of titration.

Materials Needed
per student:
- 50-mL buret
- two 250-mL Erlenmeyer flasks
- hotplate
- tongs

per titration:
- 2 g soap stock: See notes below for information on sample unknowns.
- 50 mL 95% ethanol
- 5–10 mL phenolphthalein indicator solution: To 100 mL of 95% ethanol add 1 g of phenolphthalein.
- standardized 0.250 M sodium hydroxide solution: In a 1-L volumetric flask, add 10.0 g of sodium hydroxide to 900 mL of distilled water. Stir until dissolved. Bring the solution up to volume with distilled water. See notes below for the standardization procedure.

Notes
✔ To standardize the 0.250 M sodium hydroxide, follow these steps:
1. Dry potassium hydrogen phthalate (KHP) in an oven for at least an hour at 100°C.
2. Weigh out an approximately 1.2 g sample of KHP into a 125- or 250-mL Erlenmeyer flask. Record the exact amount taken.
3. Dissolve the KHP sample in 25–50 mL of distilled water. You may have to heat the solution slightly to dissolve all of the KHP.
4. Rinse and fill a buret with the solution to be standardized. Add 4–5 drops of phenolphthalein indicator to the KHP solution. Titrate the sample.
5. Calculate the sodium hydroxide concentration (the molar mass of KHP is 204.23 g/mole).

✔ Two different unknown samples are used in this experiment. One sample is 100 % stearic acid. The other unknown is 20% lauric acid and 80% stearic acid by weight (so a 100g sample would contain 20 g of lauric acid and 80 g of stearic acid). For this unknown to be homogeneous, the mixture needs to be pulverized or rolled together. You can place the mixture in a zipper-type plastic bag and roll over the outside with a large bottle or rolling pin.

✔ Students should be given approximately 14 g of their unknown so 6 titrations can be done if necessary.

Safety, Disposal, and Special Handling
Review the Material Safety Data Sheets (MSDS) of any chemical used in the experiment for information regarding safety and handling. A waste container should be made available for titration residue (which will turn into a gel). Dispose of waste according to your local ordinances.

Points to Cover in Pre-Lab
Tell students that soap making is an art as well as a science. This scenario is not meant to explore the art of soap making. (Solvent choices, salting out the soap, and effects of additives like glycerin and fragrances are beyond the scope of this introductory experiment.) Soap making ingredients like fatty acids, fats, and oils are described by chemical properties and tests. Appropriate chemical properties or tests are used to evaluate soap making materials. One such important property is the amount of acid present.

If students have not performed titrations previously, show them how to read the meniscus and how to determine the endpoint (the first persistent pale pink color). You may need to remind students that at the endpoint, the moles of acid are equal to the moles of base.

Likely Play-Out of Lab
After titrating, students determine the formula and molecular weights of stearic and lauric acids, the models for tallow fatty acids and coco fatty acids, respectively. Then, the average molecular weight of the ideal soap fatty acid is calculated. From the amount of base required for neutralization and the mass of the sample used in the titration, the apparent average molecular weight can be determined. Whether the apparent molecular weight of the sample matches the ideal soap fatty acid average molecular weight is determined by the student. (See Question 1.) Students may have questions about the end-point because the mixture is not perfectly clear. However, they improve with practice—reproducibility is typically within acceptable limits. Most results are clearly either the ideal mixture or not.

Possible Answers to Questions

1. **What is the average molar mass of the supposed stock in the barrel you analyzed?**
   The molar mass of stearic acid is 284.48 g/mol and that of lauric acid is 200.32 g/mol. Since one unknown has the ration 80:20 stearic acid:lauric acid, the ideal molar mass is (0.8 x 284.48 g/mol) + (0.2 x 200.32 g/mol) = 267.65 g/mol. The other unknown is 100% stearic acid; its molar mass is 284.48 g/mol. However, students' results will vary according to their titration skills.

2. **Would you recommend that a company buy the supposed soap stock at auction? Why or why not?**
   This answer will depend on the students' calculated molar mass of the soap stock. The company should purchase the soap stock only if it is found to have the ideal 80:20 ratio of tallow fatty acids to coco fatty acids.

3. **If some of the material came out of solution during the titration, how would the results be affected?**
   The molar mass of the soap stock would increase since the calculated moles of acid present in solution would be too low.