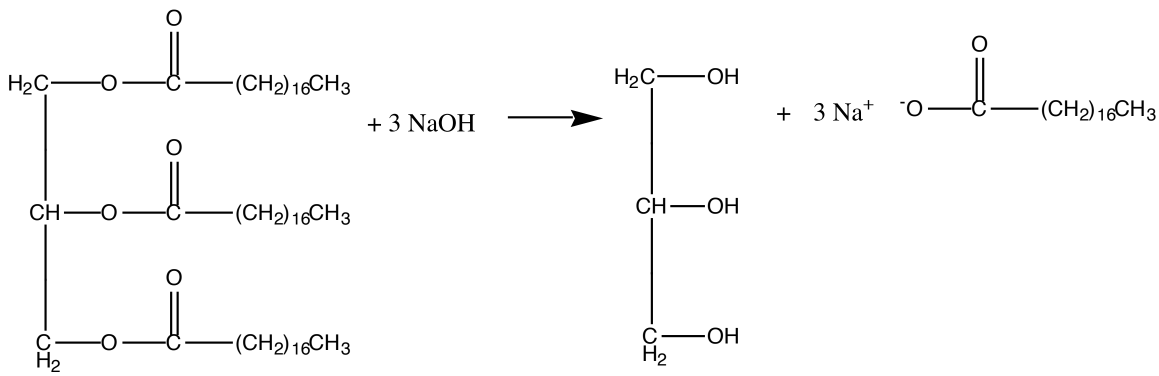
Experiment 13 – Preparation of Soap

Soaps are carboxylate salts with very long hydrocarbon chains. Soap can be made from the base hydrolysis of a fat or an oil. This hydrolysis is called **saponification**, and the reaction has been known for centuries. Traditionally, soaps were made from animal fat and lye (NaOH). (Lye was traditionally made by pouring water through wood ashes.) An example of a saponification reaction is shown below.



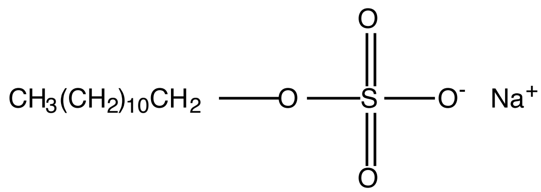
Glyceryl tristearin, a fat — Glycerol — Sodium Stearate, a soap

As you may remember, fats and oils are triesters of glycerol and three fatty acids. Esters can be hydrolyzed to their alcohol and carboxylic acid components in the presence of acid or base. Fats, oils, and fatty acids are insoluble in water because their hydrophobic tails are so long. If a base is used for hydrolysis, the fatty acids produced are deprotonated and are present as the corresponding carboxylate salts. Because these product carboxylate salts are charged, they are much more soluble in water than the corresponding uncharged fatty acids. Since the carboxylate salts also each have a long nonpolar tail, they are also compatible with nonpolar greases and oils.

Soap can **emulsify** fats and oils by forming micelles around oil droplets. The soap molecules surround an oil droplet so that their nonpolar tails are embedded in the oil and their charged "head" groups are on the exterior of the droplets, facing the water. If the oil droplets are small enough and if there are enough soap molecules to surround them, the oil droplets become dispersed in the water and can then easily be washed away. Therefore, using lots of soap, hot water, and agitation can help clean greasy dishes. Hot water can melt solid fats, and agitation can help break up the fats and oils into smaller droplets. Using lots of soap makes it more likely that there will be enough soap molecules to surround and emulsify all of the fat droplets.

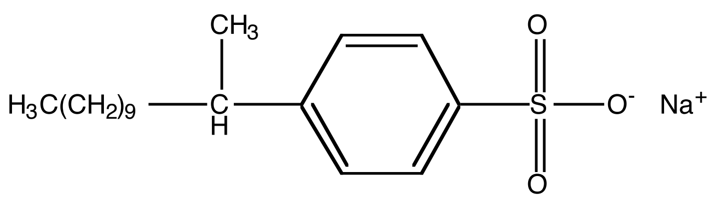
Soaps are less effective in **hard water**, which is water that contains a significant concentration of Mg^{2+} and Ca^{2+} ions. These ions form precipitates with soap molecules, and this precipitate is often seen as a gray line on a bathtub or sink and is often called "soap scum". Since soap forms a precipitate with these ions, it means that many of the soap molecules are no longer present in the solution. Therefore, soap will form fewer suds in hard water. "**Soft water**" is water that contains very few or no ions that precipitate with soap. Soap will therefore be much more effective in soft water than in hard water.

Detergents are similar to soaps in that they have a charged head group and a long nonpolar tail group, but they are not prepared from natural fats or oils. Detergents are useful because they do not form precipitates with magnesium or calcium ions, which means that they work in both soft and hard water. Shown below is a typical detergent molecule, sodium lauryl sulfate (which you may recognize from ingredient lists of shampoos or other cleaning products):



Sodium Lauryl Sulfate (a nonbiodegradable detergent)

After detergents started being widely used, it was discovered that they were not broken down in sewage treatment plants. Many streams and lakes became contaminated with detergents and large amounts of foam appeared in natural waters. Biodegradable detergents were then developed. Shown below is an example of a biodegradable detergent, sodium laurylbenzenesulfonate.



Sodium Laurylbenzenesulfonate (a biodegradable detergent)

Many commercial detergents also contain phosphate compounds. This can be a problem, because phosphate is a nutrient for plants. Too much phosphate in a pond, lake, or stream accelerates the growth of algae, which consumes too much of the dissolved oxygen in the water. This disturbs the ecosystem in the pond, and some organisms will die. Therefore, you will see some detergents these days that are labeled “phosphate free”. These are better for the environment than phosphate-containing detergents.

In this experiment, you will make soap from a fat or an oil by heating it with sodium hydroxide. You will precipitate the soap by adding it to a concentrated salt solution, and then you will collect the solid soap using vacuum filtration.

You will then test the soap you made for its pH and foaming ability. You will test to see how well it emulsifies oil and you will also test its behavior in hard water. You will carry out the same tests on a commercially prepared soap solution and on a commercially prepared detergent solution, and you will compare your soap to the commercial soap and detergent.

Safety Precautions:

- Wear your safety goggles. This is especially important in this experiment, since NaOH can cause permanent eye damage.

- Wear gloves if they are available.
- Watch your reaction mixture at all times as it is heating. Do not let it boil over and do not let the volume of the heated solution decrease too much.

Waste Disposal:

- Waste ethanol (if any) should be disposed of in the **organic** waste containers (which have a pink label) in one of the fume hoods.
- Waste sodium hydroxide solution (if any) should be placed in one of the **inorganic** waste containers (which have a blue label) in the fume hood.
- All other waste may be poured down the sink with plenty of running water.

Procedure

Part 1 – Saponification – Preparation of Soap

1. Weigh a 150-mL beaker and record the mass. Add about 5 g of a fat or oil, reweigh, and record the mass. Calculate the mass of fat or oil used by subtraction. Record the type of fat or oil you are using.
2. Add 15 mL of ethanol and 15 mL of 20 % NaOH to the beaker. (Be very careful when pouring the NaOH solution, and don't let it splatter.) Add a small magnetic stir bar to the beaker and heat and stir the mixture on a magnetic stirrer-hotplate. Heat the mixture (with constant stirring) for 30 minutes or so, until the solution no longer has two separate layers. The solution should be transparent at this point. **Important:** as it is heating, some of the solution will evaporate. You must make sure that the volume does not decrease too much, so you will need to add more liquid as the reaction progresses. Add 5 mL portions of a mixture containing equal volumes of ethanol and water to the beaker in order to maintain the same volume in the beaker. Also, do not let the mixture overheat or foam over, and do not allow it to boil to dryness! If this happens, you will need to start over.
   Caution: the mixture of oil and ethanol will be very hot, and may splatter or catch fire. Have a watch glass nearby to smother any flames. Wear goggles at all times, because NaOH can cause permanent eye damage!
3. When the saponification is complete, carefully remove the beaker from the heat. Measure out 50 mL of a saturated NaCl solution in a graduated cylinder and pour it into a 400-mL beaker. (If a saturated NaCl solution is not available, you can make your own by mixing 30 g of solid NaCl with 100 mL of deionized water. Once the salt has been dissolved, the solution can be shared by two groups of students.)
4. Pour the soap solution into the salt solution in the 400-mL beaker and stir with a stirring rod. This process is called "salting out". It increases the density of the solution and causes the soap to precipitate and float on the surface of the solution.
5. Place the beaker in an ice-water bath until it reaches the approximate temperature of the bath. In a separate container, chill about 20 mL of deionized water – you will need it to rinse the solid you collect in the funnel.
6. Collect the soap curds by vacuum filtration with an aspirator. To do this, collect a ring stand, a clamp, a side-arm flask, a piece of heavy-walled rubber tubing, a

Büchner funnel, and a piece of filter paper. Clamp the flask to the ring stand, and then attach one end of the tubing to the side arm and the other end to an aspirator on one of the sinks. Put the Büchner funnel on the flask (make sure it forms a seal – you may need a rubber adapter) and the filter paper on the funnel. (The filter paper should lie flat and should just fit the bottom of the funnel, covering all of the holes – if it doesn't, the filtration won't work, so get a different piece of filter paper.) Moisten the filter paper with a little deionized water from a wash bottle.

7. Turn on the water to start the vacuum. Swirl the soap/salt mixture and pour it on the filter paper. When the liquid has been pulled through, rinse the soap with two separate 10-mL portions of chilled deionized water. (When you do this, make sure to rinse the entire surface of the solid, not just one spot!) After the soap has been rinsed, pull air through it to dry it further.
8. Use a rubber policeman to transfer the soap to a clean, dry watch glass or a small beaker. (**Important:** the soap may still contain NaOH, so avoid skin contact with it. Use plastic gloves if possible.) Leave the soap to dry in your locker until the next laboratory period.

Part 2 – Properties of Soaps

Preparation of Soap Solutions

9. In this part of the experiment, you will be comparing the properties of the soap you made with a commercial soap and a detergent. You will start out by making three separate soap solutions, as detailed in the following steps.
10. Mix 1 g of the soap you prepared with 50 mL of warm deionized water. Swirl the solution to mix it well (but try not to shake it, because you don't want to make lots of suds yet). Label this solution.
11. Mix 20 drops of the pink liquid lab soap with 50 mL of warm deionized water. Swirl this solution to mix it well. (If the soap is a liquid, use 20 drops.) Label the flask.
12. Mix 1 g of the commercial detergent (if it is a liquid, use 20 drops) with 50 mL of warm deionized water, and swirl to mix well.

pH Test

13. Label four separate test tubes. In the first tube, place 10 mL of the soap solution you made. In the second tube, place 10 mL of the commercial soap solution. In the third tube, place 10 mL of the detergent solution. In the fourth tube, place 10 mL of deionized water (this will be the control). One by one, stir each solution with a stirring rod and then touch the stirring rod to a piece of pH paper. Record the pH of each solution. Save these solutions for the next part.

Foam Test

14. Stopper each of the tubes from step 13 and shake each one continuously for 10 seconds. Observe and record the amount of suds or foam each soap solution produces. Save these solutions for the next part.

Interaction with Oil

15. Add 5 drops of oil to each test tube from step 14. Stopper and shake each of the tubes continuously for 10 seconds. Observe what happens to the oil layer in each tube. Compare the amount of suds in each tube to the amount of suds they each had in part 14. Do the tubes have more or less suds than they did in step 14? Which substance(s) dispersed (emulsified) the oil? These solutions can be poured down the sink.

Hard Water Test

16. Label three clean test tubes. Put 5 mL of your soap solution in the first, 5 mL of commercial soap solution in the second, and 5 mL of detergent solution in the third. Add 20 drops of 1% $CaCl_2$ solution to each tube. Stopper each test tube and shake continuously for 10 seconds. Compare the amount of suds in each tube to the amount of suds they each had in part 14. Do the tubes have more or less suds than they did in step 14? Record any other observations about the solutions and how they compare. These solutions can also be poured down the sink. Wash and rinse the test tubes.
17. Repeat step 16 with new samples of each soap solution, but instead of adding 1% $CaCl_2$ solution, add 20 drops of 1 % $MgCl_2$ solution. Again, observe how much foam is produced and compare this amount to the amount produced in step 14. Record any other observations. Pour these solutions down the sink and wash and rinse the test tubes.
18. Repeat step 16 with new samples of each soap solution, using 20 drops of 1 % $FeCl_3$ solution. Record your other observations as before. Pour these solutions down the sink and wash and rinse the test tubes.

Questions

1. Write the reaction for the saponification of glyceryl tripalmitate with sodium hydroxide.
2. Why is the product of saponification called a salt?
3. Why was ethanol added to the reaction mixture of fat and base?
4. Do you think the solid soap that you made contains glycerol? Why or why not? Explain.
5. Describe the appearance of your soap.
6. Explain how soaps emulsify oils and fats.
7. Explain why soaps are more soluble than fatty acids in water.
8. Did the solid soap you made contain excess base? Explain how you know.
9. How did your soap compare to the commercially prepared soap in its foaming ability?
10. Which soap was best at emulsifying oil?
11. Explain what happened in the hard water tests. Did any of the soap solutions work better than the others? Explain.
12. How could you determine if a water sample is "hard" or "soft" water? Describe an experimental test.