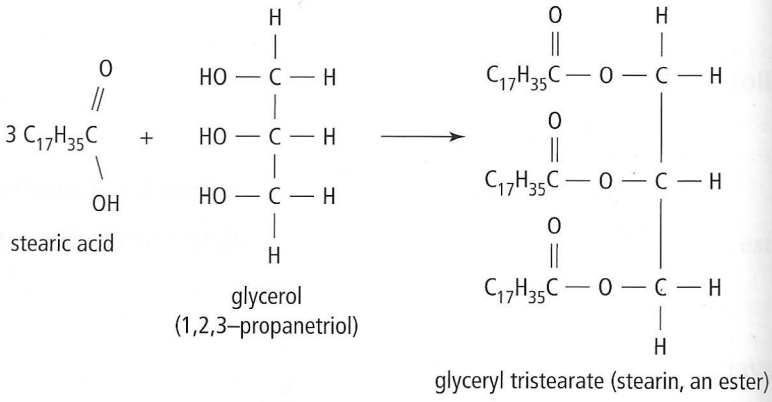
15C Preparation of a Soap

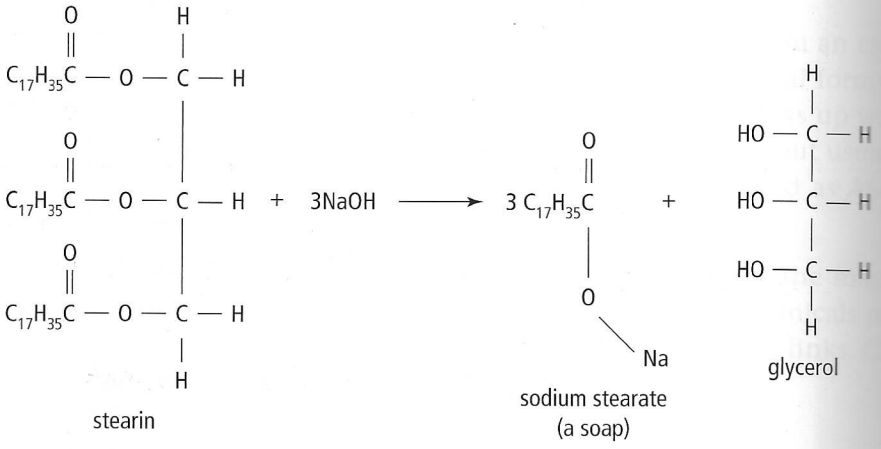
The term "detergent" refers to any substance with strong cleansing power. Detergents are commonly classified as either soaps or synthetic detergents. Synthetic detergents are prepared from synthetically produced chemicals, whereas soaps are prepared from natural fats and oils. (The word "soap" has its roots in a Latin word meaning "animal fat.")

The history of soap making can be traced back 5000 years to the Middle East, where it was discovered that treating fat with alkali resulted in a substance with cleansing and healing powers. In fact, for many centuries soap was used medicinally only, in the treatment of skin wounds. Soap making remained relatively primitive until the 16th century, when techniques that produced a purer soap were developed. The reaction that produces soap is called *saponification*.

In more precise terms, soaps are salts of mixed fatty acids. Today they are prepared by reacting fats (which are esters) with alkali solutions such as sodium hydroxide or potassium hydroxide. For example, stearin, an ester, is a principal component of animal fat. It is the glycerol (1,2,3–propanetriol) ester of stearic acid, glyceryl tristearate, or stearin.



Stearin, when heated with sodium hydroxide, is broken down into glycerol and sodium stearate, a soap:



In a typical commercial process, a sodium hydroxide solution is added slowly and intermittently to a molten mixture of fats and oils. High temperature and good mixing are maintained by passing steam through the mixture. After a time, the fat is broken down to form an emulsified mixture of soap, glycerol, and unreacted NaOH. At this point an NaCl solution is added, which causes the soap to separate as a curd and float to the top of the mixture. In the final stages, perfumes, colorings, antiseptics, and other ingredients are added as necessary.

In Part I of this experiment, you will prepare a soap by reacting a fat with a fairly concentrated NaOH solution. Unlike the commercial process, ethanol will be added to help speed up the reaction. The ethanol serves as a solvent to bring the reacting materials into closer contact so that the procedure can be conducted in one laboratory period. Then in Part II, you will test your soap, a commercial soap, and lard for solubility, sudsing, and acidity.

OBJECTIVES

1. to prepare soap by saponification
2. to compare the results of tests on the soap prepared in the experiment and a soap prepared commercially

SUPPLIES

Equipment

beaker (250 mL)
2 beakers (150 mL)
water-soluble marker
pan (such as a dissecting tray)
3 wooden splints
glass stirring rod
hot plate
heat resistant mat
beaker tongs or crucible tongs
plastic spoon
test-tube rack
3 test tubes (16 mm × 150 mm)
metric ruler
lab apron
safety goggles

Chemical Reagents

lard, fat, or oil
commercially prepared bar of soap
saturated NaCl solution
neutral litmus paper or universal indicator solution
6*M* NaOH
ethanol (denatured)
distilled water

PROCEDURE

Part I: Saponification

1. Put on your lab apron and safety goggles.
2. Obtain a 150 mL beaker and using a water-soluble marker, label it "NaCl(aq)".

It is important to monitor your heating constantly to avoid any boil over.

Ethanol is very flammable so keep it away from any flame.

6*M* NaOH is extremely corrosive and can cause severe burns. Wash spills and splashes off your skin and clothing immediately, using plenty of water. Call your instructor.

3. Place about 60 mL of saturated NaCl solution in this beaker and heat it on a hot plate (set at "high") until the solution just begins to boil. Set the solution aside on a heat resistant mat and save it for Step 9. Label a second 150 mL beaker "H_2O", place 100 mL of distilled H_2O in it, and set it aside as well.
4. Place about 15 g of fat (lard) in a 250 mL beaker. Heat the lard over the hot plate (set at "medium") until it melts. Stir the fat carefully with a glass stirring rod while it is melting. Be prepared to remove the beaker from the heat should overheating occur. Record the time of melting, as well as your observations, in your copy of Table 1 in your notebook.
5. Remove the beaker from the heat, then carefully and slowly pour 25 mL of ethanol into the molten lard. Stir the mixture and resume heating on "medium".
6. Slowly pour 25 mL of 6*M* NaOH solution into the mixture in a thin steady stream, stirring constantly and slowly. (Rapid addition or fast stirring may cause the fat to separate from the mixture.)
7. Continue to heat the mixture slowly and stir it regularly for the next 10 min or 15 min, until no evidence of fat globules remains. Occasionally, you will need to add distilled water (from the 150 mL beaker) to maintain a constant volume of mixture. Record the time at which no fat remains, as well as your observations, in Table 1. Turn off the hot plate.
8. Place some cold tap water in a tray for a cold water bath. Set the 250 mL beaker containing the mixture in the cold water bath and then add 40 mL of distilled water to the mixture while stirring it.
9. Allow the mixture to cool for several minutes in the cold water bath and then slowly add the 60 mL of warm NaCl solution from Step 3. Stir the mixture while adding the NaCl solution. In Table 1, record the time at which soap begins to form, as well as your observations.
10. The soap should now be visible as curds on the top of the mixture. Use a plastic spoon to scoop off a sample of soap and place it on a piece of paper towel that has been folded several times.
11. Save your soap sample for Part II. Clean up the rest of your apparatus according to the reagent disposal instructions.

Part II: Laboratory Tests

1. Label 3 test tubes A, B, and C with a water-soluble marker and place them in a test-tube rack.
2. Use separate wooden splints to add a small sample of each of the following substances to the test tubes: a sample of lard to test tube A, a sample of your lab soap to test tube B, and a sample of commercially prepared soap to test tube C.
3. Half-fill each test tube with distilled water and perform the following tests.

   Solubility and Sudsing: Place your thumb over the end of each test tube and shake vigorously for 15 s. Note how well each substance dissolves and, if sudsing occurs, measure the height of the suds that form. Record

your results in your copy of Table 2.

Acidity: Test the resulting water mixtures with neutral litmus paper or universal indicator solution and record your results in Table 2.

4. Clean up according to the reagent disposal instructions.
5. Before leaving the laboratory, wash your hands thoroughly with soap and water.

REAGENT DISPOSAL

For Part I, the solution that remains after the soap has been removed contains glycerol, NaCl, and NaOH. It can be washed down the drain with plenty of warm water.

For Part II, pour both the contents of test tube A and the solid soap into the designated waste container. The contents of the other test tubes can be rinsed down the sink with copious amounts of water.

POST LAB CONSIDERATIONS

Different kinds of fats and oils combined with different basic solutions produce different kinds of soaps. Soaps containing shorter carbon chains (10 to 12 carbons) are generally more soluble than those containing longer carbon chains (16 to 18 carbons). Also, soaps prepared from potassium hydroxide are usually more soluble than those prepared from sodium hydroxide.

In the days before sodium hydroxide was commercially available, soap makers relied exclusively on potash (K_2CO_3) as the source of their basic solution. The potash consisted of wood ash prepared in an iron pot. When potash is dissolved in hot water, a strong basic solution of KOH is produced.

In this experiment you may have noticed that the curds of soap were originally suspended in a thick, viscous liquid. This liquid was glycerol. Commercial hard soap is separated from the glycerol, formed into shapes, then aged for several weeks. In soft soaps (those sold in pump dispensers), the glycerol is not separated from the soap.

EXPERIMENTAL RESULTS

Part I: Saponification

Table 1

Time	Ingredient(s) added	Observations
about 10 rows needed	COMPLETE IN YOUR NOTEBOOK	

Part II: Laboratory Tests

Table 2

Test	Lard	Lab soap	Commercial soap
Apparent solubility in water			
Height of suds (cm)			
Neutral litmus paper test			

ANALYSIS OF RESULTS

1. Describe the soap you prepared.
2. The soap you prepared is likely to be harsh on the skin. Why?
3. In Part II, what properties of your soap were similar to those of a commercially prepared soap?

FOLLOW-UP QUESTIONS

1. **a.** What practical problem might you encounter if you were to filter your final mixture to recover your soap?
   b. Suggest some solutions to this problem.
2. One popular brand of hand soap is made from a mixture of palm oil and olive oil. What do you think is the name of that brand of soap?
3. Soft soaps in pump dispensers are common in homes. State three criteria necessary to making a soft soap. (Refer to the Post Lab Considerations.)
4. During World War II, military manufacturing companies were very interested in the by-product of soap, glycerol (glycerin). Find out what it was used for.

CONCLUSION

State the results of Objective 2.