LAB 23: FATS, OILS, & SOAP:
PROPERTIES & PREPARATION

PURPOSE: To observe physical and chemical properties of fatty acids and triacylglycerols.
To synthesize soap by saponification of vegetable oil.
To compare the physical and chemical properties of soap and detergent.

SAFETY CONCERNS:

Always wear safety goggles.
Lye (Sodium Hydroxide) is caustic to skin and will dissolve eyes especially the hot concentrated solution used in this experiment. Wash with soap and copious amounts of water if contacted.
Bromine is toxic and irritating to eyes and mucus membranes.

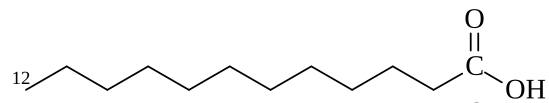
FATTY ACIDS, FATS, AND OILS:

Fats and **oils** are mixtures of complex esters. Fat esters are made from long chain carboxylic acids (called **fatty acids**) and an alcohol containing three OH groups (called **glycerol**).

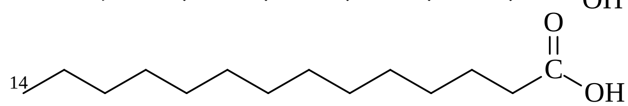
A common fat is called a **triacylglycerol** or **triglyceride**. The hydrocarbon chain of the fatty acids determines the physical and chemical properties of the compound. Triacylglycerols made from long-chain (C-16 to C-18) **saturated** fatty acids (like palmitic and stearic acids) are solid or semisolid at room temperature. Solid animal fats contain an abundance of long saturated fatty acids.

Common Fatty Acids

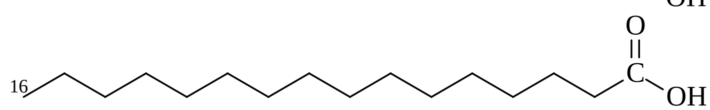
Saturated



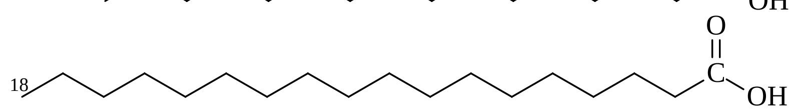
Lauric



Myristic

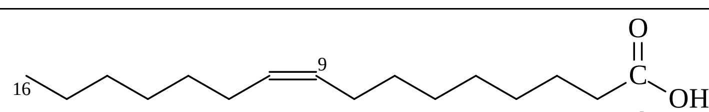


Palmitic

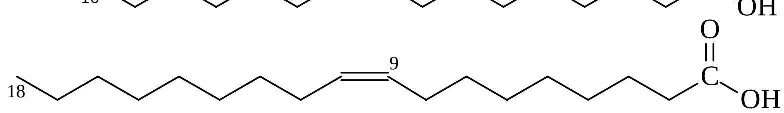


Stearic

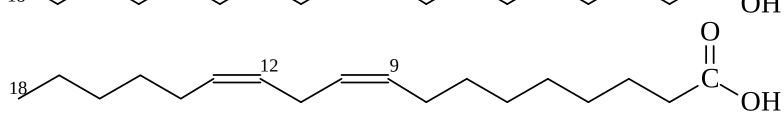
Unsaturated



Palmitoleic



Oleic

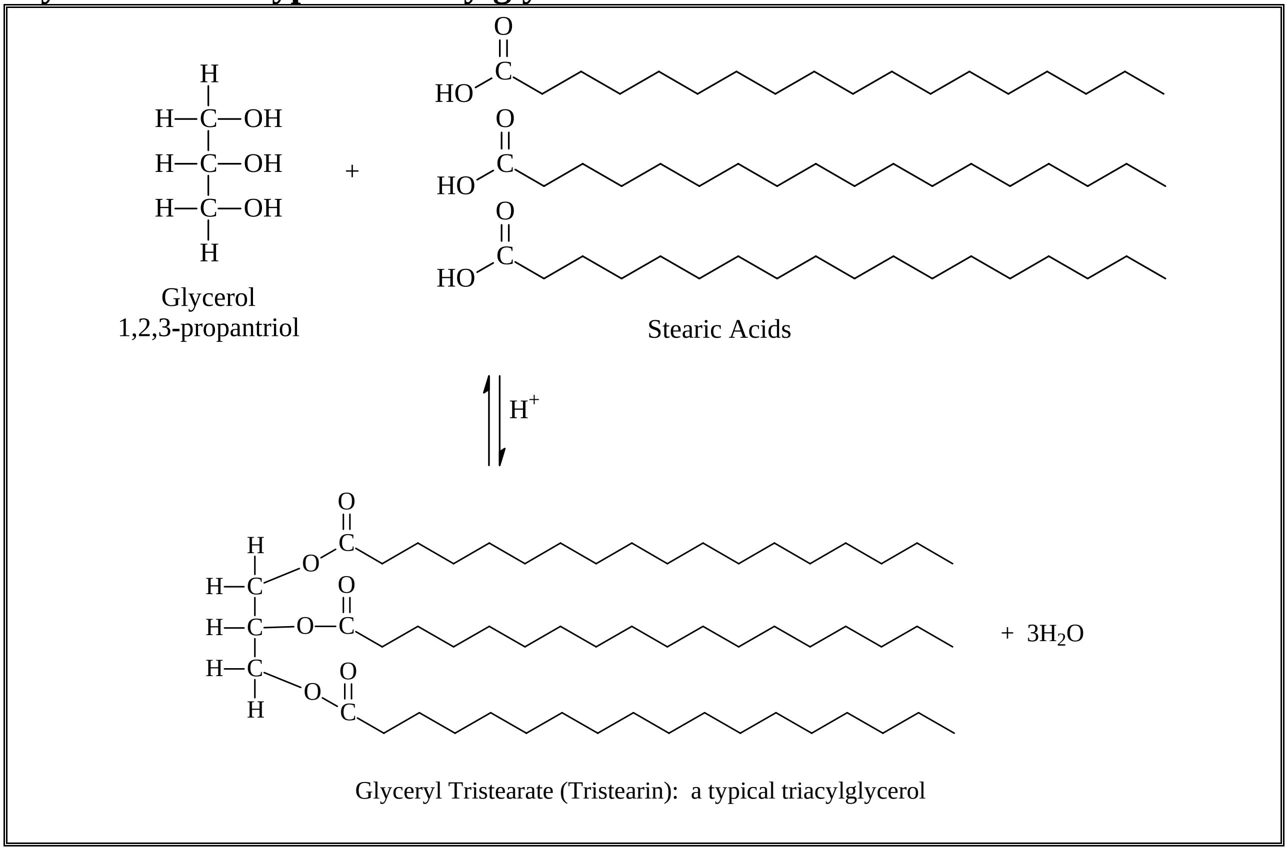


Linoleic

Triacylglycerols made from long-chain **unsaturated** fatty acids (like oleic or linoleic acids) are liquid oils at room temperature. Liquid vegetable oils contain an abundance of long unsaturated fatty acids.

A few oils owe their characteristic liquid nature to the presence of shorter chain fatty acids (C-6 to C-14). Coconut oil contains large amounts of lauric (C-12) and myristic (C-14) acids, as well as smaller amounts of C-6, C-8, and C-10 acids.

Synthesis of a Typical Triacylglycerol:

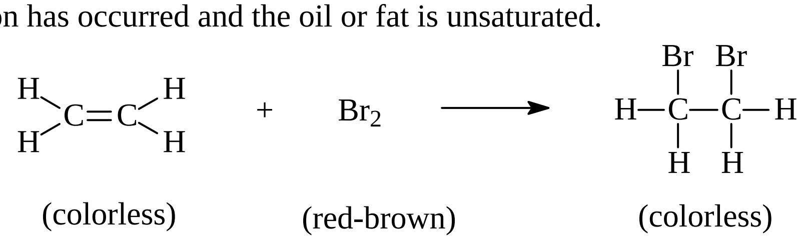


SOLUBILITY:

Lipids are a family of compounds that are grouped by similarities in solubility rather than structure. As a group, lipids are more soluble in nonpolar solvents such as ether, chloroform, or benzene. Most are not soluble in water.

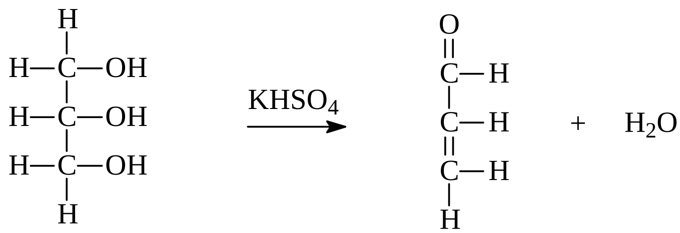
TEST FOR UNSATURATION:

The presence of unsaturation in a fatty acid or a triacylglycerol can be detected by reaction with bromine. If the orange color of a bromine solution added to a lipid fades quickly, an addition reaction has occurred and the oil or fat is unsaturated.



ACROLEIN:

When triacylglycerols are heated with potassium hydrogensulfate, $KHSO_4$, (also know as potassium bisulfate or potassium acid sulfate) the glycerol is dehydrated and oxidized to acrolein. As KHSO4 is a weak acid it can catalyze both the hydrolysis of the triglyceride esters as well as the dehydration/elimination reaction of the glycerol carbohydrate. Acrolein may be detected by its sharp irritating odor. It can often be present in charcoal broiling of fatty food. It is volatile and will evaporate and be lost with continued heating. The distinctive odor of acrolein serves as the basis of the test for a saponifiable lipid.

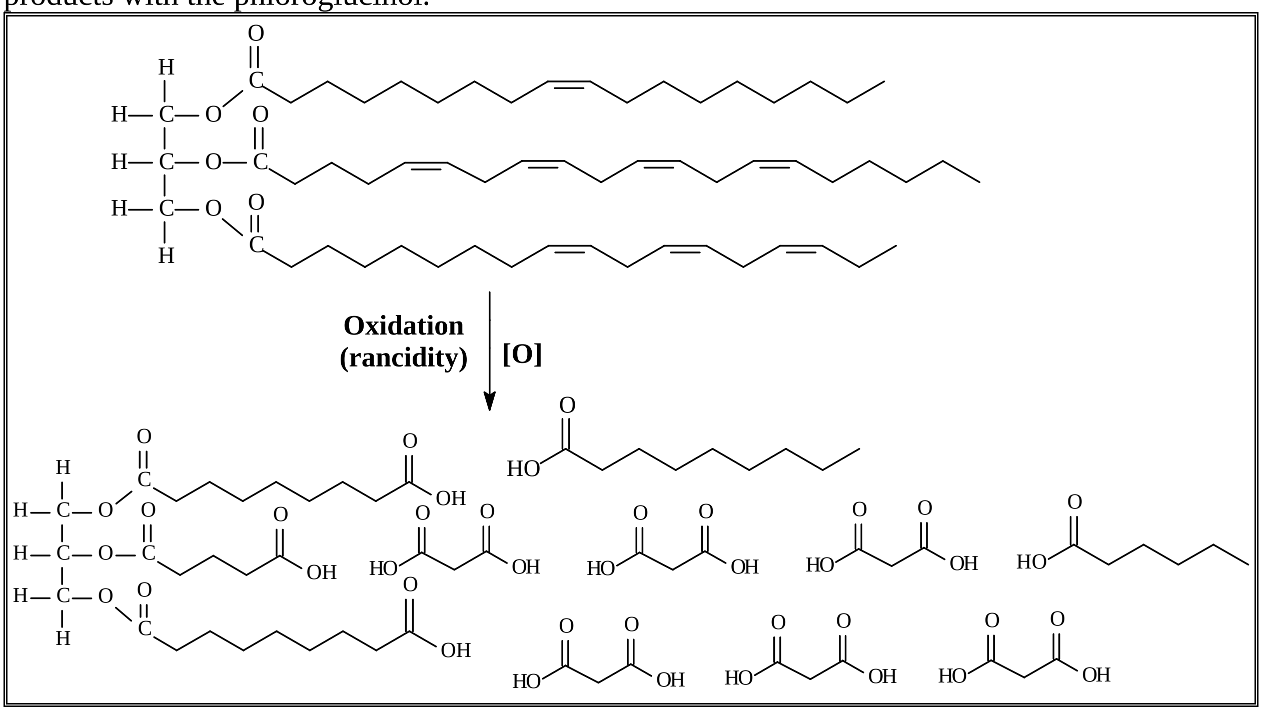


Glycerol
1,2,3-propantriol

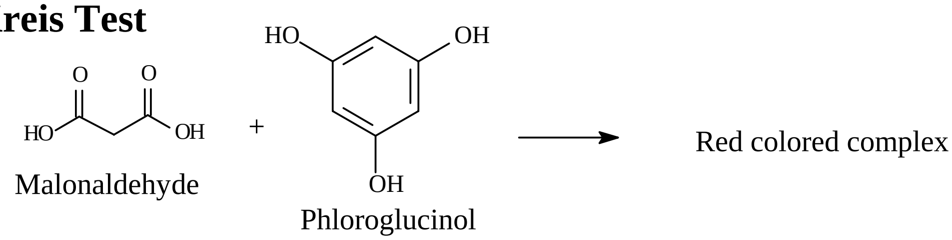
Acrolein

OXIDATIVE RACIDITY:

Oils are more easily digested than fats. They become rancid more easily than fats because of the ease in which their double bonds can be hydrolyzed or oxidized. The most commonly used test to determine oxidative rancidity is the Kreis test. A small quantity of concentrated hydrochloric acid is added to the lipid to be tested, followed by the addition of a 0.1% solution of phloroglucinol (1,3,5-trihydroxybenzene) in ether. The red or pink color indicating rancidity is believed to result from a reaction of epihydrin (an isomer of malonaldehyde) or other oxidation products with the phloroglucinol.



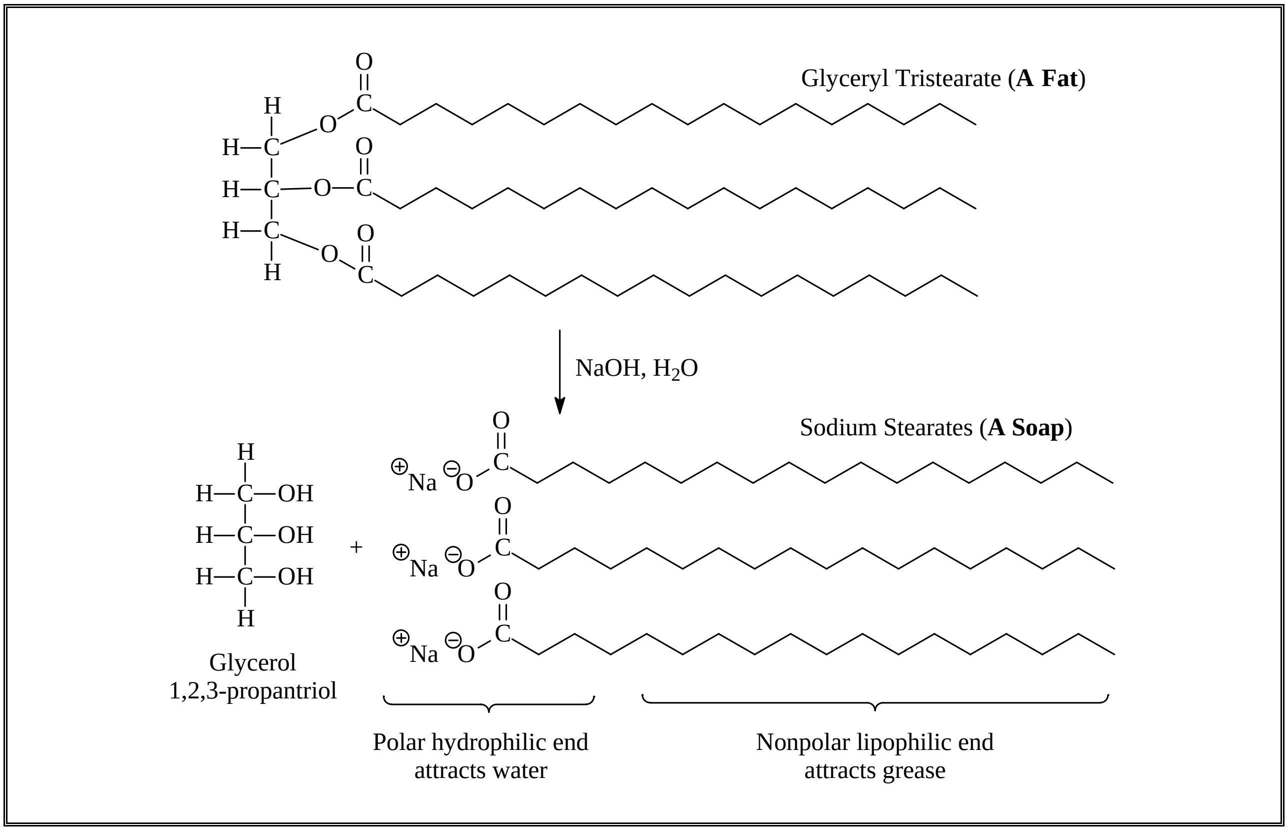
Kreis Test



SOAP:

Hydrolysis of an ester is the reverse of the synthesis of an ester. Hydrolysis of a triacylglycerol into glycerol and fatty acids can be catalyzed by either strong acid or strong base.

The general process of hydrolyzing esters with caustic alkali's (strong bases such as sodium hydroxide, NaOH or potassium hydroxides, KOH) is called **saponification**. The products of basic hydrolysis of fats or oils are glycerol and the salts of fatty acids called **soaps**.



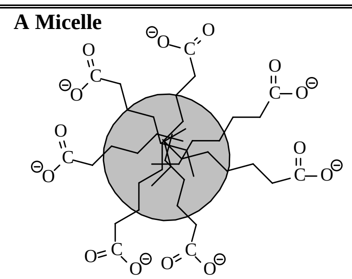
HISTORY OF SOAP:

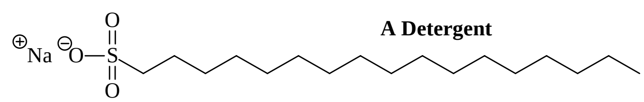
According to Roman legend, soap was discovered after a heavy rain fell on the slopes of Mount Sapo (the name means "Mount Soap" in Latin). The hill was the site of an important sacrificial altar, and the rainwater mixed with the mingled ashes and animal fat around the altar's base. As a result of this fortuitous coincidence, the three key components of soap were brought together: water, fat, and lye (potash leached from the ashes). As the mixture trickled down to the banks of the Tiber River, washerwomen at work there noticed that the mysterious substance made their job easier and the wash cleaner. They were so impressed with the cleansing power of this mysterious substance that trickled down from the mountain, they named the mountain in its honor. They knew to name it after "soap" as there was another substance that they were familiar with that also had exceptional cleansing powers.

Clothes have been cleaned for ages by beating them on rocks near a stream. Near these streams grew certain plants, such as soapworts, whose leaves produced *saponins*, chemical compounds that give a soapy lather. By mixing these saponins with their wash water, they were getting much cleaner clothes than they got from the water alone. So it was the soapwort plant that gave its name to Mount Sapo. This plant is also known as bouncing bet, a pink-blossomed perennial that grows wild throughout most to the United States.
Over the centuries the basics of soapmaking have remained essentially unchanged from the Roman prescription. Although we don't sacrifice animals at the altar to obtain our fat and then wait for the next heavy rain, we use the same ingredients. To this day in parts of rural America soap is being made much as it was in ancient Rome: out of potash, rainwater, and animal tallow. The potash is found in wood ashes and, when this ash is extracted with water and the resulting solution evaporated in iron pots, a solid is produced. This solid is called potash. It is known chemically as potassium carbonate and when dissolved in water, it gives us a very basic (caustic) solution. Most people who make soap at home do not go to all the trouble of extracting the potash from wood ashes. They use instead the more caustic substance, sodium hydroxide, which is also known commercially as lye. Animal fat is often substituted by a variety of vegetable oils. Avocado oil, olive oil, coconut oil, palm oil and cinnamon oil are just a few of the many oils that are used in today's soap formulations. No matter which fat or caustic solution is used, the same basic chemical reaction takes place. The net result of the reaction, called **saponification**, is the formation of salts from the fatty acids that once were part of the larger fat molecules.

MICELLES:

Soap owes its cleaning ability to the formation of **micelles** which can encapsulate grease or oil and make it water soluble. Micelles are destroyed by "hard" water ions such as Ca^{2+} and Mg^{2+}. These ions precipitate the fatty acid anions causing a "scum" to form. Synthetic detergents (called "syndets") have been developed which are less affected by hard water ions since the magnesium and calcium salts are quite a bit more soluble.





PROCEDURES:

ACTIONS:

I. SOLUBILITY OF LIPIDS:

1. Place 12 dry test tubes in a rack, number them A1, A2, A3, B1, B2, B3 through D1, D2, D3, and add the following:
   Tube A's: 1 mL deionized water in each.[1]
   Tube B's: 1 mL Ethanol in each. [1]
   Tube C's: 1 mL Heptane in each. [1]
   Tube D's: 1 mL Dichloromethane in each. [1]

2. To tubes"A1, B1, C1, & D1" add **palmitic acid**. [2]
   To tubes "A2, B2, C2, & D2" add 4 drops **vegetable oil**.
   To tubes "A3, B3, C3, & D3" add a ½ pea sized bit of solid **lard**.[2]
   Stopper each tube and shake vigorously for several minutes to give enough time to dissolve.

3. Report the solubility (**S** for soluble; **PS** for partially soluble; **I** for Insoluble) on the report sheet.

4. Discard the A tubes (lipids in water), B tubes (lipids in ethanol), & C tubes (lipids in heptane) in the "Waste Organic solvent" container. **Save the D tubes** (lipids in dichloromethane) for use in the **Part II; Test for Unsaturation**.

II. TEST FOR UNSATURATION:

5. To the each of the D tubes from the solubility tests in Part I add 5% bromine in methylene chloride (Br_2 in CH_2Cl_2) drop by drop; counting the number of drops until a permanent red-orange color in obtained or until 30 drops have been added.

6. Discard the D tubes in the "Waste Halogenated Solvent" container.

III. KREIS TEST FOR OXIDATIVE RANCIDITY:

1. Obtain 2 large test tubes or small Erlenmeyer flasks and label them #1(old) and #2(new).

2. Into tube #1 place about 1 g of **"old"** solid[3] fat or 1 mL (20 drops) of **"old"** liquid vegetable oil. [4]

3. Into tube #2 place about either 1 g of **"new"** solid shortening or 1 mL (20 drops) of **"new"** commercial vegetable oil.

4. Record the brand names & date of opening of each sample if available.

NOTES:

[1]Volumes do not need to be exact. Measure 1 mL once and then estimate the volume in the other tubes to match.

[2]Mass does not need to be exact but uniform in all solvents. For solid lipids use the amount of solid held on the tip of a spatula, about ½ the size of a pea.

[3]You do not need to measure 1 g exactly. A gram of a fat is a hunk about the size of the tip of your little finger.

[4]The fats tested can be commercial shortening, or oil. lard, or other triglyceride containing products.

PROCEDURES:

ACTIONS:

5. To each fat or oil sample add 8 drops of concentrated hydrochloric acid (18M HCl).[5] Stopper and shake to mix.

6. Now to each sample add 1 mL (20 drops) of 0.1% **phloroglucinol**[6] **in ether** solution. Stopper and gently shake to mix. Let stand for about 10 minutes.[7]

7. Compare the color[8]. Record your results and make conclusions.

IV. SOAP:

A. Preparation:

1. Into a porcelain evaporating dish pour
   2 mLs vegetable oil[9]
   3 mLs ethanol[10]
   20 drops 50% Sodium hydroxide (NaOH) [11]
   Stir with a stirring rod to mix well.

2. Heat the mixture gently over medium heat on a hot plate or the moderate flame of a laboratory burner. Stir constantly until it becomes a thick paste.[12]

3. Allow the dish to cool and record your observations on the report sheet.

4. Place the soap you just prepared into a 150 mL beaker with about 50 mLs of deionized water. Heat the mixture, with stirring, until all of the solid soap has dissolved in the water.[13]

5. Test the pH of this soap solution and record the result.[14] Use this soap solution in each of the following procedures.

[5]18M HCl is concentrated hydrochloric acid (con HCl). Use with caution.

[6]Phloroglucinol is another name for 1,3,5-trihydroxybenzene.

[7]Ether evaporates rapidly; slight pressure may build up in the stoppered test tube. If too much ether is evaporating add another 1 mL of ether, stopper and gently shake then let stand.

[8]A red or pink color indicates rancidity.

[9]Vegetable oils, or solid animal fat would work as they are all triacylglycerols.

[10]Ethanol is a good solvent to dissolve both the nonpolar oil and the polar NaOH in order to better mix them together.

[11]Sodium Hydroxide (50% NaOH) is extremely dangerous. Make certain you are wearing eye protection. Do your best to avoid splashes. If skin contact is made wash immediately with soap and lots of water.

[12]Do not overheat and burn your soap.

[13]If there are clumps that will not dissolve, scoop them out and discard them.

[14]This soap solution contains some leftover sodium hydroxide that didn't react so it will show a very basic pH. The crude basic soap is too harsh to use on your skin so it must be purified.

B. Purification:

1. Obtain two 50 mL beakers. Into beaker #1 pour 10 mLs of the soap solution you just made and allow it to cool.

2. Into beaker, #2, pour 10 mLs of a detergent solution.

3. To each beaker add solid sodium chloride (NaCl) a little at a time with stirring, until no more NaCl dissolves and the bottom of the beaker is covered with NaCl. Record your observations on the report sheet[15].

4. Remove the mass of pure soap floating on the surface of beaker #1. This could be done by scraping it off the top with a spatula or piece of filter paper.

5. Put a small piece of the purified soap into a test tube and dissolve it in deionized water. Stopper the tube and shake the soap to see if it lathers.[16]

6. Test the pH of the purified soap solution in the test tube and also test the pH of the detergent solution in the second beaker. Record the results.

C. Reactivity with Hard Water Ions:

1. Obtain two test tubes.
   Into test tube #1 pour 5 mLs of the unpurified soap solution (from step VA5).
   Into test tube #2 pour 5 mLs of detergent solution.

2. Add 1M Calcium Chloride ($CaCl_2$), one drop at a time, to each tube (up to a maximum of 10 drops) until a precipitate forms.

3. Record the results and complete the equation for the reaction on the report sheet.[17]

D. Reactivity with Acid:

4. Obtain two test tubes.
   Into test tube #1 pour 5 mLs of the unpurified soap solution (from step VA5).
   Into test tube #2 pour 5 mLs of detergent solution.

5. Add up to 4 drops of 6M HCl (hydrochloric acid), one drop at a time to each test tube and observe if a precipitate forms.

6. Record the results and complete the equation for the reaction on the report sheet.[18]

[15]Soap can be forced out of solution by dissolving NaCl in it. Water can only dissolve a limited amount of stuff and since NaCl is more polar than the long hydrocarbon chain of the soap, the water lets go of the soap and dissolves the NaCl. The soap that has been let go floats to the surface of the water. The NaOH leftover from the preparation of soap reaction is very polar like sodium chloride so stays dissolved in the water. Therefore, the soap that comes to the surface of the water is now more pure.

[16]You could also test the lathering ability by using the purified soap to wash your hands.

[17]The sodium salts of fatty acids, soaps, are soluble in water. When other metal ions like calcium, magnesium, or iron, form salts with fatty acids they are not as soluble in water and tend to precipitate out.

[18]A soap is a weak base so it reacts with HCl by taking an H^+ which turns it into a carboxylic acid. The newly made carboxylic acid is a fatty acid with a long hydrocarbon chain and now that it has no charge it is no longer soluble in water.

LAB 23: FATS, OILS, AND SOAP: NAME______________
PRE LAB EXERCISES: DATE________________

1.___ An unsaturated lipid
- A. is more difficult to digest than a saturated lipid.
- B. has a lower melting point than a saturated lipid.
- C. spoils less easily than a saturated lipid.
- D. is more soluble in polar solvents than a saturated lipid.
- E. more than one of these.

2.___ Why are some solvents more effective at dissolving grease than others?
- A. greases are nonpolar so will dissolve best in nonpolar solvents.
- B. greases are nonpolar so will dissolve best in polar solvents.
- C. greases are polar so will dissolve best in nonpolar solvents.
- D. greases are polar so will dissolve best in polar solvents.

3.___ Acrolein is produced
- A. whenever any lipid is saponified.
- B. from cholesterol.
- C. when certain lipids, upon heating, form glycerol, which is then easily dehydrated.
- D. from nonsaponifiable lipids as well as saponifiable lipids.
- E. more than one of these.

4.___ If a commercial lipid produced a positive Kreis test, you should
- A. return it to the store and demand a refund.
- B. use it regularly for cooking.
- C. use it as a salad dressing.
- D. classify it as a nonsaponifiable lipid.
- E. more than one of these.

5.___ Which of the following makes water considered to be "hard" water?
- A. Ca^{2+}
- B. Na^{1+}
- C. NH_4^{1+}
- D. More than one of these.

Match the following terms with the one best structures they best represent:

6.___ Fatty Acid

7.___ Triacylglycerol

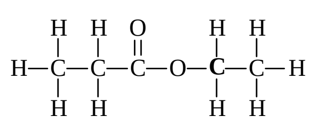
8.___ Detergent

9.___ Soap

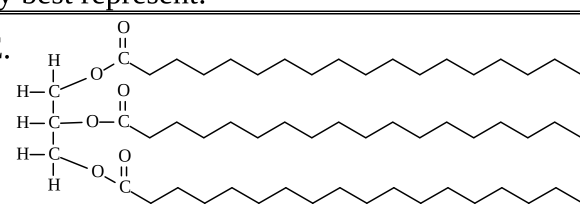
10.___ Micelle

11.___ Ester

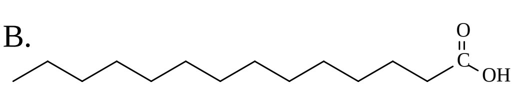
A.



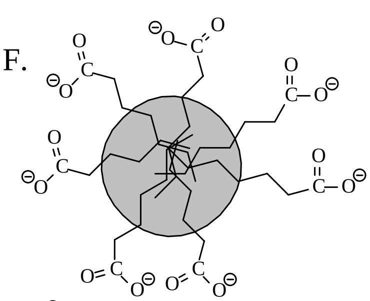
E.



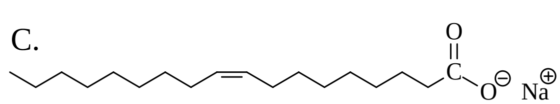
B.



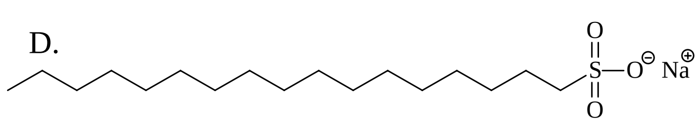
F.



C.



D.



LAB 23: FATS, OILS, AND SOAP: REPORT:

NAME___________________

PARTNER__________DATE___

I. SOLUBILITY:

	1. Water	2. Ethanol	3. Heptane	4. Dichloromethane
Palmitic Acid				
Vegetable Oil				
Lard				
Conclusions:				

___1. List all of the solvents used in this experiment that could act as an effective grease remover?
A. water B. ethanol C. heptane D. Dichloromethane E. none of these

II. UNSATURATION:

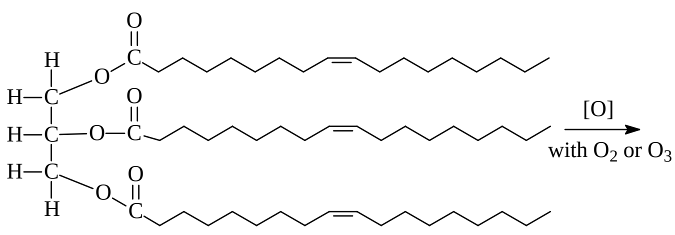
	OBSERVATIONS Drops of Bromines Used	CONCLUSIONS Saturated (S) or Unsaturated (U)?
Palmitic Acid		
Vegetable Oil		
Lard		
Explanation: What happened and why? Are your results expected? Why or why not?		

2. Write an equation showing the reaction of Bromine with a representative triglyceride that may be present in a vegetable oil.

III. KREIS TEST FOR RANCIDITY:

BRAND	AGE (DATE)	OBSERVATIONS	CONCLUSIONS Rancid or Not?
Old Solid ________			
Old Liquid ________			
Explanation? What happened and why? Are your results expected? Why or why not?			

1. Complete the following showing the products of complete oxidation (rancidity) of the double bonds:



Glyceryl Trioleate

IV. SOAP:

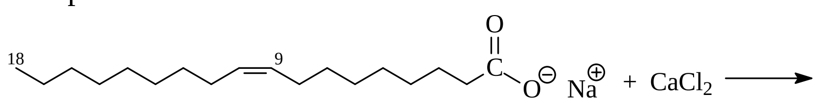
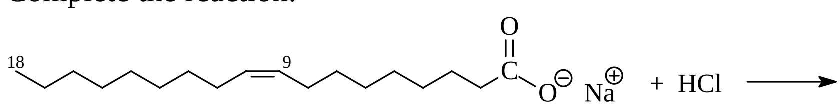
	OBSERVATIONS	pH
A. Preparation	Appearance of freshly made soap:	pH of crude soap dissolved in water: ________
B. Purification	Results of "salting out" the soap:	pH of purified soap dissolved in water: ________
	Describe the "lather" after shaking:	
	Results of "salting out" detergent:	pH of detergent solution: ________

___3. "Salting" works to purify soap because _______.

A. Salt kills bacteria that would make soap impure.
B. Soap is not as soluble in water as salt is.
C. Salt (NaCl) reacts with soap to make a less soluble product that can precipitate.
D. More than one of these.

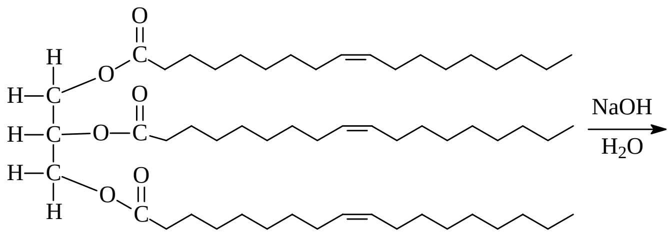
___4. Soaps and detergents dissolve in both nonpolar oil and in polar water because ____

A. they have a nonpolar ionic end that attracts oil and a polar hydrocarbon end that attracts water.
B. they have a nonpolar ionic end that attracts water and a polar hydrocarbon end that attracts oil.
C. they have a polar ionic end that attracts water and a nonpolar hydrocarbon end that attracts oil.
D. Soaps and detergents are neither polar nor nonpolar so the solvent doesn't matter.

<table>
<tr><td rowspan="2">C. Reaction with Hard Water Ions</td><td>Results of adding $CaCl_2$ to soap:</td></tr>
<tr><td>Results of adding $CaCl_2$ to detergent:</td></tr>
<tr><td colspan="2">Complete and balance the reaction:

Sodium Oleate</td></tr>
<tr><td colspan="2">Name of Products formed:</td></tr>
<tr><td rowspan="2">D. Reaction with Acid</td><td>Results of adding to HCl soap:</td></tr>
<tr><td>Results of adding HCl to detergent:</td></tr>
<tr><td colspan="2">Complete the reaction:

Sodium Oleate</td></tr>
<tr><td colspan="2">Name of Products formed:</td></tr>
</table>

RELATED EXERCISES:

1. Complete the following:



Glyceryl Trioleate

2. Name the Products:

___3. The odor of acrolein is frequently strong in restaurants that feature charcoal-broiled food. Which of the following best explains the presence of this odor?
A. the proteins in cooked meat decompose to form acrolein.
B. the tracylglycerols in meat decompose in high heat and the resulting fatty acids then dehydrate to form acrolein.
C. the tracylglycerols in meat decompose in high heat and the resulting glycerol then dehydrates to form acrolein.
D. the charcoal used to cook food decomposes to form acrolein when the heat is high enough.
E. more than one of these.

___4. Which of the following would be expected to produce Acrolein upon heating with $KHSO_4$?
A. olive oil B. stearic acid C. glycerol D. palmitic acid
E. oleic acid F. coconut oil

REFERENCE SEARCH:

5. During World War II homemakers were asked to contribute their bacon grease to the war effort for the production of nitroglycerin. Use a source of your choice to write an equation for the production of nitroglycerin from a fat.

Reference Source =