



Oil Refining Virtual Field Trip Teachers Guide

Recommended Usage:

- *For greatest understanding it is suggested to follow the oil refining virtual field trip in sequence.*
- *However, each segment can be explored as an individual topic.*
- *The virtual field trip can be used either in the traditional classroom or online.*
- *Teacher facilitation is recommended for secondary level while facilitation at the post secondary level could be optional.*

Goals or objectives of this activity:

- define crude oil, refinery, distillate, flash tower, pig, and barrel;
- understand the process of separating crude oil into usable products and list several of these products;
- locate major refineries in Alaska as well as nearest refineries to where s/he lives;
- identify reasons why crude oil refineries are important/necessary in today's society;
- assess the current and projected future amounts of petroleum in the world and their impacts on world energy consumption;
- identify and compare alternative forms of energy resource refining.

Additional Questions to be answered or explored:

1. Internet scavenger hunt
2. What and How is Oil Refining
 - a) A lab could be developed to fit your classroom making a table top model of an oil refinery and its components and or actually making diesel fuel.
 - i. At the end of the document a lab provided by Dr. Kenneth Walz, Chemistry Instructor at the Madison Area Technical College for which you can use all or part.

ii. The chemistry of refining crude oil:

http://www.powernaturally.org/Programs/pdfs_docs/12_Oil_Refining_Chemistry.pdf

3. Where is Oil Refined: Once locations of refineries are found exercises could be developed to create maps of where the oil is coming from, where it is going and in what form. Discussions could be developed about the efficiency of this process. Does production meet the demand/need?
4. Why Do We Need Oil Refining: An exercise on identifying what products in the classroom and at home are made from petroleum products would be helpful to emphasize how much our community uses such products. Lower level classes could be given a list of products to find and compete to see who can find the most. Upper level could be required to first develop a list, find the products. Discussions about the need for the product would emphasize dependence on such products
 - a) Activity to explore why we need oil (Grades 3-7)
http://www.beloit.edu/sepm/Geology_and_the_enviro/Petroleum_ne_ed.html
 - b) Activity to explore why we need oil (Grades 7-12)
<http://www.eia.doe.gov/kids/classactivities/EnergyAnalysisEIA.pdf>
5. What are the Alternatives: Students can research alternative fuel sources looking at availability, benefits, detriments, level of renewability. This could be done individually or in a group. If done as a group project, each group could choose an alternative method of providing energy to examine and make the argument to the class of its feasibility. Students will most likely lean on only one type though a combination of sources might be more appropriate.
 - a. While activities 3&4 are about building models of oil drilling and oil rigs the rest of these activities pertain to alternative energy systems: <http://fossil.energy.gov/education/OceanEnergyMMS.pdf>
 - b. This article examines drilling in the Arctic refuge:
http://www.rmi.org/images/PDFs/Energy/E01-04_FoolsGoldAnnot.pdf
 - i. Discussions could be moderated about whether or not to drill in the Arctic refuge
 1. What will be the impacts on the environment?
 - a. If drilling occurs there
 - b. If it doesn't occur there

Minimum System Requirements

- PC/Macintosh with a FREE download of Google Earth. To obtain Google Earth™, go to <http://earth.google.com/>
- High-speed Internet connection is highly recommended.
- The videos located inside of the virtual tour are streamed from various sources such as Google Video, Vimeo, Viddler, or YouTube. If you cannot see the videos, check with your school IT department to ensure they are not blocking the Google Video web site.
- [See the system requirements on this page](#) (We encourage the “Recommended Configuration”).

MATC, Chem 2 Organic Reactions and Biofuels

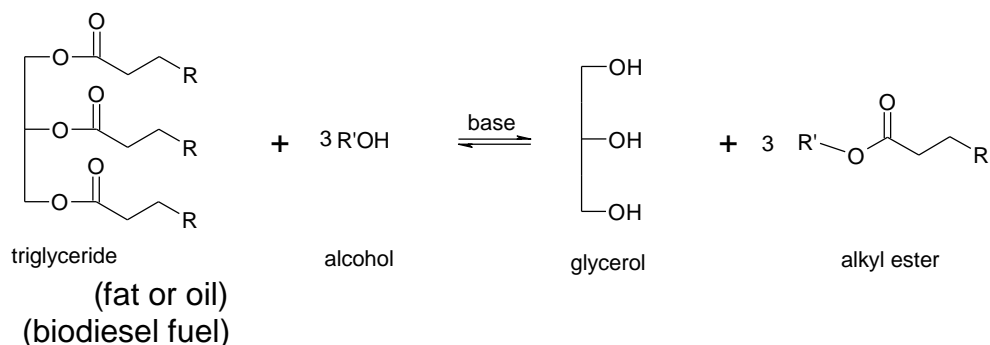
January,

2008

As oil reserves dwindle, gas prices rise, and CO₂ accumulates in the atmosphere, we are turning to biofuels to provide our energy needs domestically while controlling greenhouse gas emissions. Fuels derived from plants such as corn or soybeans are considered to be “greenhouse neutral” because the CO₂ released by the plant is equivalent to the CO₂ absorbed by the plant as it grew. In 2006, Governor James Doyle announced Wisconsin’s “Declaration of Energy Independence” which set a target to obtain 25% of the state’s transportation fuels from renewable biofuel resources (ethanol and biodiesel) by the year 2025.

Ethanol is the fastest-growing biofuel in the U.S and serves as an alternative fuel for gasoline (spark ignition) engines. In many urban areas, gasoline commonly contains 5 to 10% ethanol as an oxygen additive to help control vehicular air pollution (smog). E85, a mixture of 85% ethanol and 15% gasoline, is also being used in flex-fuel vehicles that can run on E85 or gasoline. In 2005, 4 billion gallons of ethanol were produced in the United States, about 170 million gallons of which were made in Wisconsin. Ethanol is produced by fermentation of sugar-containing biomass, mostly sugar cane, sugar beets, or sweet corn. The fermented product is then distilled to yield pure ethanol. Wisconsin industry has a rich background in the fermentation (i.e. brewing) sciences, and the state also is a top producer of corn. For these reasons it is widely predicted that Wisconsin’s ethanol fuel production will increase significantly over the next two decades.

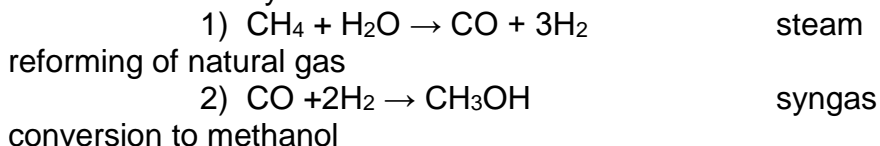
Biodiesel fuel is produced from renewable sources of oil including pure vegetable oils, waste fryer grease, and rendering tallow and serves as an alternative fuel for diesel (compression ignition) engines. The fat or oil is reacted with an alcohol (typically methanol) using a basic catalyst (such as KOH) to form alkyl esters (biodiesel), and glycerol (a byproduct).



Biodiesel can be blended with conventional petrodiesel in any ratio, although the most common are B2, B5, B20, and B100, which are 2%, 5%, 20%, and 100% biodiesel mixed with petrodiesel. These blends can be used to fuel ordinary diesel engines without modification. About 75 million gallons of biodiesel were produced in the United States in 2005. Wisconsin produced less than 1 million gallons of biodiesel in 2005, but the state's strong soybean crop has encouraged development of several new biodiesel plants that will increase WI production to nearly 100 million gallons in 2007.

Two other biofuels that have received somewhat less attention are methanol and butanol. Methanol has been used for many years as a fuel in auto racing. Historically, methanol was produced by fermentation of cellulosic biomass sources such as wood, and it was given the common name, "wood alcohol". Today, most methanol is produced from natural gas (methane) by a less expensive steam reforming process. Methane is first reacted with water at high temperatures and pressures to produce a mixture of carbon monoxide and hydrogen called synthesis gas, or syngas.

Syngas then reacts over a catalyst to form methanol.



Methanol can also be made from methane derived from the decomposition of municipal solid wastes or sewage. These renewable sources of methanol are not used currently due to the low cost of natural gas. Butanol is one of the newest alternative fuels. In 2006, British Petroleum and Dow Chemical announced a joint research effort to study the feasibility of butanol production from agricultural waste products.

In this lab you will react methanol with restaurant fryer waste oil (soybean) to synthesize biodiesel. You will also test and compare the properties of conventional petroleum and alternative fuels. In this way, you will examine how the microscopic structure of molecules influences the macroscopic physical properties of various liquid fuels.

A fuel's flashpoint is the minimum temperature at which there is a sufficient concentration of evaporated fuel vapor for combustion to propagate after an ignition source (spark or flame) has been introduced. The concentration of fuel vapor is directly related to the fuel's vapor pressure, which in turn is dependent on temperature (see figure 1 below). Different fuels require different vapor concentrations to initiate combustion, thus flash point varies significantly between fuels (see Figure 2). In fact, the flash point differences between gasoline and diesel fuels are integral factors in the function of spark ignition versus compression ignition engines. In this lab, you will measure the flashpoint of biodiesel and compare this with other liquid fuels.

Figure 1. Vapor Pressure as a function of temperature for ethanol and biodiesel fuel.

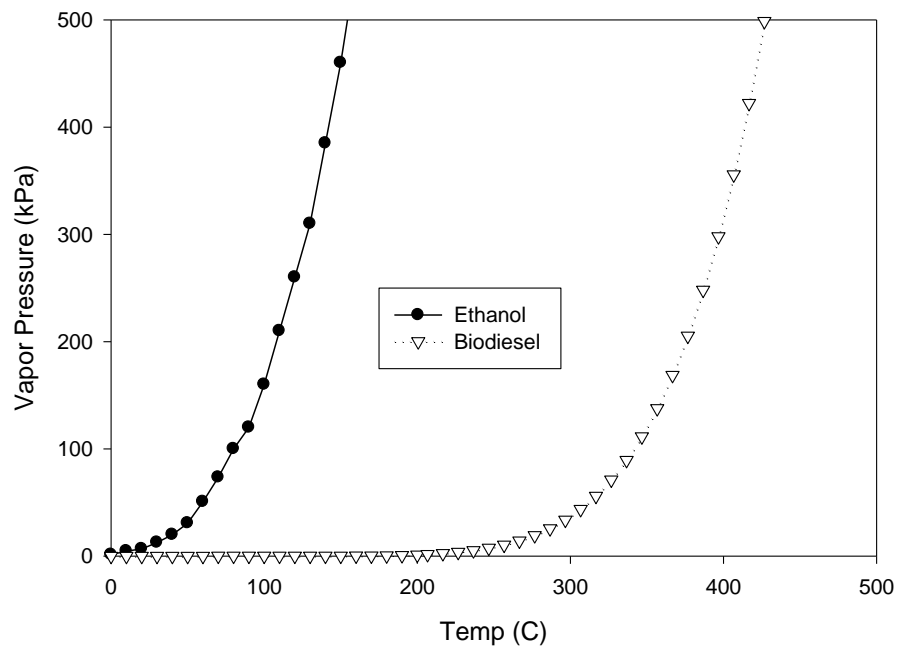


Figure 2. Flashpoints of several common fuels. Fuels marked with * indicate this value is the minimum allowed by ASTM.

Fuel	Flashpoint (°C)	Flashpoint (°F)
Gasoline	-43*	-45*
Acetone	-18	0
Methanol	10	50

Ethanol	12	54
Isopropanol	12	54
Octane	13	55
Butanol	35	95
#1 (winter) diesel	38*	100*
#2 (summer) diesel	52*	126*
#4 (marine) diesel	55*	131*
#6 bunker fuel	66*	151*
Methyl stearate	110	230
Methyl laurate	113	235
Biodiesel	130*	266*
Cetane	135	275
Methyl oleate	180	356
Methyl linoleate	186	367
Canola oil	192	378

Prelab—Please complete the following in your lab notebook before coming to lab:

(hint you may wish to consult chemfinder.com or the Merck Index for molecular structures)

- 1) Go to the Internet website “how stuff works” and view the page describing diesel engines (<http://auto.howstuffworks.com/diesel.htm>). In a few sentences, briefly describe the major differences between a gasoline engine and a diesel fuel engine.
- 2) In a few sentences, briefly describe the chemical differences between gasoline and diesel fuel.
- 3) Using structural formulas, write the balanced chemical reaction that occurs between potassium hydroxide and methanol to form water and potassium methoxide.
- 4) Using structural formulas, write the balanced chemical reaction that occurs between trilinolein and methanol to form glycerol and methyl linoleate.
- 5) Draw structural formulas for each of the fuel molecules included in data table II of the lab.
- 6) Perform an internet search to determine the ASTM (American Society for Testing and Materials) specification that biodiesel fuel (B100) must meet for viscosity and flash point (Hint you may wish to consult the National Biodiesel Board, www.nbb.org, check this URL).

- 7) Define vapor pressure. Briefly describe how vapor pressure varies with temperature. Explain the differences in vapor pressure that you observe for the fuels shown in figure 1.
- 8) Define flashpoint. Based on Figures 1 and 2, briefly explain how the flashpoint of a fuel is related to its vapor pressure.
- 9) *Draw illustrations for the stepwise experimental procedures for parts I, II, and IIIA below in your lab notebook as demonstrated by your instructor in class.*

Equipment and Supplies:

Ring stand	Utility clamp	Large beaker
Water bath	Heating/stir plate	125-mL Erlenmeyer flask
Thermometer	Magnetic stir bars, (1" and 1.5")	100-mL Erlenmeyer flask
Test tubes	Stir bar retriever	10-mL graduated cylinder
Thermometer clamp	pH paper	100-mL graduated cylinder
Rubber stoppers (0 & 1)	Disposable culture tubes	15 mL disposable plastic cups
Stopwatch	glass microscope slides (several)	1-2 mL disposable beral pipettes

Chemical reagents:

Glycerol	Restaurant fryer waste oil (soybean)
Methanol	Commercial Biodiesel (ASTM Spec)
DI water dyed with food coloring	KOH/ methanol solution (35 g KOH / L methanol)

Procedure Part I—Synthesis of Biodiesel

Watch as the instructor demonstrates the biodiesel synthesis at room temperature. You will be able to compare your results to this demonstration at the end of the class.

1. Add a stir bar to a 125 mL Erlenmeyer flask. Measure out ~55 mL of the restaurant waste soybean oil into a graduated cylinder (at the front). Transfer it to the Erlenmeyer flask.
2. Place Erlenmeyer flask containing the fryer oil into the water bath and clamp it to the ring stand.

3. Use a beaker to obtain hot tap water (40 to 50°C) for the water bath. Fill the bath so that the water level is roughly equal to that of the fryer oil in the flask. Clamp the thermometer to the ring stand so that it reads the temperature of the water bath. Turn the hot plate on to a low setting (setting 1-3). Record the water temperature, and turn on the stir bar to create a vortex and begin preheating the oil.

4. Watch the reaction temperature carefully. When the temperature reaches 50°C, turn off the heat until the bath cools to 45 °C, then turn the hot plate back on and resume heating. *Attempt to keep the temperature between 45 and 55 °C. Note that the boiling point of methanol is 64°C!*

IMPORTANT! THE KOH/METHANOL SOLUTION IS CAUSTIC! WEAR GLOVES AND SAFETY GOGGLES FOR THIS PROCEDURE. WASH EXPOSED SKIN IMMEDIATELY WITH WATER. CLEAN UP ANY SPILLS AND TELL THE INSTRUCTOR.

5. Measure 14 mL of KOH/methanol solution in a graduated cylinder and carefully transfer to the flask of pre-heated fryer oil. Record the time at which the addition is complete.

6. Watch the mixture for the first few minutes and record any notable observations in your lab notebook. Some things you might want to look for include: How well do the components mix? Do they mix more or less easily after a few minutes, or is there no change? What color is the mixture? What happens if you stop the stir bar for a minute?

7. Proceed to parts II-IV while the reaction runs. Allow the biodiesel reaction to continue for 30-45 minutes while monitoring the reaction temperature. Then return to step 8.

8. Stop the stir bar and allow the reaction mixture to settle for ~10 minutes until two distinct phases have completely separated. Record the final temperature and time the stir bar was stopped.

9. Obtain two plastic weighing boats and label them as “biodiesel” and “glycerol”. Weigh the empty boats and record their mass.

10. Gently decant (pour off) as much as you can of the top biodiesel layer into the first weighing boat. Use a beral pipette to transfer the remainder of the biodiesel, using care to avoid disturbing the bottom layer. Use the balance to determine the mass of your biodiesel product (remember to subtract the mass of the empty boat).

11. Pour the bottom glycerol layer into the second weighing boat and determine its mass as well.

Procedure Part II. Fuel Chemical Structures and Solubility Tests

Note, to save time you should begin Part I of this lab first, and then work on the solubility tests while the biodiesel reaction is being completed.

1. Obtain a bottle of distilled water that has been dyed with food coloring.
2. For each of the compounds listed in the solubility data table, add the compound dropwise to 5.0 mL of colored water in a small test tube. Stopper and shake the tube between each drop. Stop adding drops when two distinct layers form within the tube OR when a total of 20 drops has been added – whichever comes first. Record the number of drops added in the data table.
3. For each compound draw the structural formula, and then rate its solubility in water as very soluble (i.e. miscible), slightly soluble, or not soluble (i.e. insoluble).
4. When finished dispose of the solutions in the labeled waste container.

Procedure Part III – Biodiesel Quality Tests

Procedure IIIA – Viscosity Tests

1. Obtain a sample of the waste fryer (soybean) oil feedstock. You will also need several glass slides, four Beral pipettes, and a stopwatch.
2. Place a paper towel on the bench. Lay a glass slide flat on the paper towel. Using a Beral pipette, carefully place two drops of the waste fryer (soybean) oil standard near one end of the glass slide. Quickly tilt the slide, so that the oil runs down the slide to the paper towel. Begin the timer when the slide is tilted, and stop the timer when the oil reaches the end of the slide. If time allows, repeat as necessary and calculate an average time.

Some things to keep in mind while doing this test:

- Avoid the frosted part of the slide (this gives inconsistent results)
- Use the same amount of oil on each slide (two drops is sufficient)
- Make sure you tilt the slide the same way each time (hold it vertically)

3. Repeat this test with pure glycerol, commercial (ASTM Spec) biodiesel, and then with the biodiesel that you made in the lab.

Procedure Part III B – Density and pH tests

1. Obtain a disposable 15 mL graduated plastic beaker and determine its mass when empty. Fill the beaker to the 15 mL line with a sample of commercial (ASTM spec) biodiesel, and determine the mass of the beaker and fuel together. Using this information, determine the density of the commercial biodiesel and record in the data table provided.
2. Obtain a pH strip, immerse it in the commercial biodiesel fuel sample, and record the pH.
3. Repeat these tests using the biodiesel fuel that you prepared in the lab, and record your data.

Procedure Part III C - Glycerides Test (free and total glycerol)

1. For the glycerides test add 7 ml of methanol to a disposable culture tube. Using a disposable beral pipette, add 0.75 ml of commercial biodiesel. Stopper the tube to seal the open end.
2. Shake vigorously for a minute, and allow the mixture to settle in a test tube rack.

3. Repeat this process using the biodiesel fuel that you prepared in the lab.
4. After 5 minutes observe bottom of culture tubes for unreacted triglycerides and/or monoglyceride and diglyceride intermediates which appear as an insoluble liquid layer at the bottom of the tube.

Procedure Part III D– Flashpoint Tests

Your instructor will assign you and your lab partner(s) a fuel sample (either ASTM spec commercial biodiesel or student biodiesel) to measure the flashpoint. Data will be combined to obtain class averages.

- 1) Inspect the flash point tester to ensure sample test cup is clean, the fluke digital thermometer is in place, and propane supply is turned on.
- 2) Making sure test cup and specimen are at least 18°C (32°F) below the expected flash point. Fill the test cup between 50% and 85% full with the fuel sample, place lid on cup and set cup inside the stove, making sure grooves are properly aligned.
- 3) Adjust the valve screw on the burner block starting with it all the way closed, then open in only 1/10 of a turn. Using the lighter provided light the test flame and adjust the screw if necessary to attain a flame size of 5/32 inches in diameter (use the bead on the burner block for comparison).
- 5) Preheat to the temperature indicated in the data table for part IIIB. Using the variable heat control dial, adjust the heat setting to the level indicated in table IIIB (either 50 or 100) for the fuel that you are using.
- 6) Stir sample using stirring mechanism on the top of the flash point instrument. WEAR A GLOVE DURING THIS PROCESS. The top sections of the instrument will get very hot to the touch.
- 7) Stop stirring (do this each time you apply the test flame). Apply the test flame by lowering the flame for half a second, leaving it for 1 second, and then quickly raising the flame back to the test position.
- 8) Alternate stirring and flash point testing. Test the flashpoint at 5 °C temperature and record in table IIIB.
- 9) Record the flash point for your fuel. The sample is deemed to have flashed when *“a large flame appears and instantaneously propagates itself over the entire surface of the test specimen”*. You should see a flash, which sometimes is accompanied by a whoosh or popping noise as well.

10) Turn the temperature dial down to zero and allow the instrument to cool at least 18°C (32°F) below the observed flash point before attempting a second measurement or running a different fuel sample. Only remove and clean test cup once the temperature have decreased to safe handling temperatures of less than 55°C (130°F). If yours is the last sample that will be tested for the day, turn off the propane tank.

11) The instructor may assist you in the use of compressed air to cool the flashpoint apparatus.

Questions- Please answer the following in your lab notebook and submit with your lab report.

Questions Part I: Biodiesel Synthesis

Useful constants:

Molecular weights:

Soybean oil: 872.60 g/mol

Glycerol: 92.10 g/mol

Methanol: 32.04 g/mol

Densities:

Soybean oil: 0.92 g/mL

Glycerol: 1.25 g/mL

Methanol: 0.79 g/mL

1. In your lab notebook, please calculate the ratio of the moles of methanol to fryer oil that was used in your reaction. (To simplify your calculations, assume that the fryer oil was pure soybean oil, also ignore the small negligible amount of KOH in the methanol/KOH solution).
2. Based on the answer to #1, did you use a stoichiometric ratio of methanol and oil? If not, which reactant was limiting and which was present in excess? Explain why this ratio of methanol:oil was chosen to use in the biodiesel reaction.
3. Based on the masses of reactants used, and the mass of biodiesel fuel that you obtained, calculate the percent yield for your biodiesel reaction (*Hint: assume that the molecular mass of biodiesel can be represented as the ester methyl linoleate. See Chang text section 3.10 for help with percent yield*).
4. April Green conducts this experiment and obtains a result of 80% yield. What might account for this result? *Explain your answer and be specific.*
5. Octavius Brown conducts this experiment and obtains a result of 110% yield. What might account for this result? *Explain your answer and be specific.*
6. Based on the results of your synthesis conducted at 50 °C and the instructor's synthesis conducted at room temperature, what effect (if any) does temperature have on this reaction? Explain your answer in terms of reaction rates and molecular collisions.

Questions Part II – Fuel Structure and Solubility Tests

7. Rank the solubility in water for the fuels that you tested from most soluble (#1) to least soluble (#8). How do the structures of the soluble compounds differ from the other fuels?
8. Ethanol is frequently used as a replacement fuel for gasoline. How does the solubility of ethanol compare with that of gasoline? Can you think of any challenges that this might pose for the storage, transport, sale, and use of ethanol fuel?
9. Draw the structure for methyl *tert*-butyl ether (MTBE). Which would you expect to be more soluble in water, MTBE or ethanol? *Explain your answer.*
10. In the 1990s, MTBE was used in the United States as an oxygen additive in gasoline. This compound caused major problems when it was found to migrate into groundwater supplies due to leaky underground storage tanks (See Chemistry In Action, pp 1026-1028 in the Chang 9th ed. textbook). MTBE can make water taste foul, and is a suspected carcinogen. In recent years, ethanol has replaced MTBE as an oxygen additive for most gasoline supplies in the U.S. List a few pros and cons regarding the use of ethanol for this purpose.

Questions Part III – Viscosity, pH, Glycerides, and Flashpoint Tests

11. Rank the liquids in terms of their viscosity from #1 = most viscous (longest run time) to #4 = least viscous (shortest run time).
12. What functional group is present in the glycerol molecule? How does this functional group affect glycerol's viscosity as compared to the other liquids that were tested?
13. How does the viscosity of your biodiesel compare with the commercial biodiesel standard?
14. Forrest Steward conducts this experiment and finds that his biodiesel is more viscous than the commercial biodiesel. What might account for this result? *Explain your answer and be specific.*
15. Skye Shepard conducts this experiment and finds that her biodiesel is less viscous than the commercial biodiesel. What might account for this result? *Explain your answer and be specific.*
16. How did your biodiesel pH measurements compare with the commercial (ASTM spec) biodiesel? What factors might have influenced the pH of your fuel?

17. How did glycerides tests compare between your biodiesel and commercial biodiesel? What might account for any differences that you observed?

18. The Department of Transportation uses flashpoint to determine shipping and safety regulations. The DOT classifies substances as either flammable or combustible using the criteria listed in the table below:

	Flashpoint (°C)	Boiling Point (°C)
Flammable		
Class IA	< 22.8°C	< 37.8°C
Class 1B	< 22.8°C	> 37.8°C
Class IC	22.8°C - 37.8°C	N/A
Combustible		
II	37.8°C - 60°C	N/A
IIIA	60°C - 93°C	N/A
IIIB	> 93°C	N/A

Classify the commercial biodiesel and biodiesel you made according to DOT convention. Also classify gasoline and ethanol based on the flashpoints that were listed in Figure 2. Briefly describe the storage and transport implications for these fuel classifications.

19. How did the flashpoint of the biodiesel fuel made in class compare with that of commercial (ASTM spec) biodiesel? Briefly explain what might account for any difference that you observed. What does the difference in flashpoints indicate about the vapor pressures of commercial (ASTM spec) biodiesel and your biodiesel?

20. As you get on the Madison Metro Bus today the bus driver notices the Chem 212 syllabus hanging out of your backpack, and asks you about replacing petroleum diesel with biodiesel fuel. List at least four potential benefits of biodiesel fuel.

Organic Reactions and Biofuels Data Tables

Data Table I – Biodiesel Synthesis:

Volume of waste fryer oil used = _____

Volume of methanol used = _____

Mass of empty glycerol weigh boat = _____

Mass of weigh boat + glycerol = _____

Mass of glycerol = _____

Mass of empty biodiesel weigh boat = _____

Mass of weigh boat + biodiesel = _____

Mass of biodiesel product = _____

Data Table 2 – Fuel Density Determinations:

Mass of empty 15 mL disposable beaker = _____

Mass of disposable beaker + 15 mL of commercial biodiesel = _____

Mass of 15 mL of commercial biodiesel = _____

Density of commercial biodiesel = _____

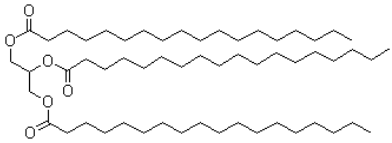
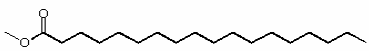
Mass of empty 15 mL disposable beaker = _____

Mass of disposable beaker + 15 mL of your biodiesel product = _____

Mass of 15 mL of your biodiesel product = _____

Density of your biodiesel product =

Part II. Fuel Chemical Structures and Solubility Test Data Table:

	Structural Formula	# of drops dissolved in water?	Solubility in water? (very soluble, slightly soluble, insoluble)
octane			
cetane (hexadecane)			
methanol			
ethanol			
butanol			
Soybean vegetable oil			
Commercial Biodiesel (ASTM spec)			
glycerol (1,2,3-propanetriol)			

Part III – Fuel Quality Test Data tables:

Table IIIA) Fuel Viscosity Drop Run Times (in seconds)

Trial #	Waste Fryer (Soybean) Oil	Glycerol	Commerical Biodiesel	Your Biodiesel
1				
2				
3				
average				

Table IIIB) Flash Point Measurements (in °C)

Fuel Type	Commercial Biodiesel	Student Biodiesel
Preheat Temp	100 °C	20 °C
Heat Setting	100 (full scale)	50 (half scale)
Flash Test Interval	5 °C	5 °C
Team #1		
Team #2		
Team #3		
Team#4		
Class Average		

Table IIIC) Biodiesel Fuel Quality Comparison Chart

	Average Viscosity Run Time (s)	Density (g/mL)	pH	Glycerides Present? (Y/N)

Commercial Biodiesel (ASTM spec)				
Your Biodiesel				