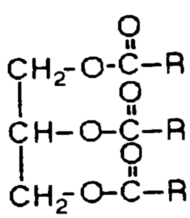
TEACHER REFERENCE PAGES-ESTERIFICATION LAB

INTRODUCTION

Esters are important industrial and biological compounds. Fats (**1**) are esters of three long-chain carboxylic acids with glycerol, a triol. The pleasant odors from fruits are complex mixtures consisting primarily of volatile esters. The odor of isopentyl acetate (**2**) is that of bananas or pears, butyl butanoate (**3**) resembles pineapples, and propyl 2-methylpropanoate (**4**) is like that of rum. Polyesters constitute an important part of the synthetic polymer field. Polyesters such as Dacron (**5**), Terylene, and Mylar are marketed and widely used to make clothing fiber, tire cord, or plastic film.

In this experiment, students will synthesize isopentyl acetate (banana oil) from acetic acid and isopentyl alcohol. After purification, banana oil will be identified not only by its odor but also characterized by using a modern technique, gas chromatography.



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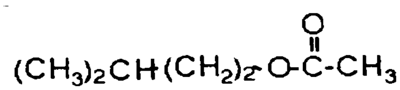

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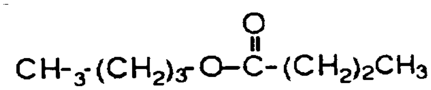

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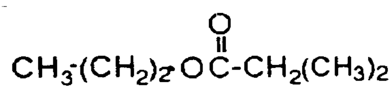

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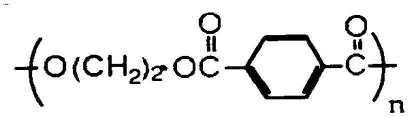

5

GAS CHROMATOGRAPHY

One major technique that is used to study organic molecules is gas chromatography. **Gas chromatography (GC)** is the separation of compounds in the gas phase, depending on their relative ability to adsorb onto the column packing and their volatility into the gas phase at the temperature used. The gas chromatograph is a simple, sensitive instrument which can be used to separate and identify about 60% of all known organic compounds.

The compounds to be separated are injected into a gas stream which passes through a column at a preset speed. Under a constant set of conditions in terms of temperature, gas flow rate, and column packing and size, repeated injections of a compound elute from (come out of) the column at a nearly constant time from injection. Different compounds elute at different times. One factor which affects elution time is the molecular weight of the compound; heavier compounds move more slowly through the column. Elution time is also affected by polarity and other factors. The column is first injected with known compounds called standards, and their retention times are determined. Then, unknown mixtures of compounds can be injected, and if the known compounds are in the mixture, their peaks can be recognized by their characteristic retention times.

A gas chromatograph detects the presence of a compound in its eluate (exiting stream) by means of some property of the compound. One common method used by GC detectors is to compare the conductivity of a heated filament which is placed within a stream of pure reference gas (helium in our lab) to a heated filament placed in a stream of gas containing our sample molecules. When molecules from our sample pass the detector filament, the changes in conductivity caused by temperature changes are converted into electrical signals which appear as peaks on a computer data screen. Peaks seen in the eluate are plotted on a chart, and the integrated peak area is proportional to the concentration of the compound. Many GCs report the integral area of each of the peaks, following the plotted graph of the peaks. An approximate proportionality between peak height and concentration can also be seen (see sample printouts).

EQUIPMENT

10	Stirring Hotplates
10	Microkits
	Micropipets
2	Gas Chromatographs
2	Analytical Balances

SUPPLIES

10	Ring Stands
10	3finger Clamps
4	DI Water Washbottles
4	Acetone Washbottles
20	Tygon tubing
20	Sample Vials
10	Metal Pans
10	Metal Thermometers
box	Gloves
1pr	Hot Mitts
	Sand for baths
	Fiberglass
	Cotton
1	Al cooling pan for sand

20 Pasteur pipets
2 Syringes for GC

CHEMICALS

5% $NaHCO_3$
95% H_2SO_4
Anhydrous $MgSO_4$
Glacial Acetic Acid
Isopentyl Alcohol
$CaCl_2$
Silica Gel
Acetone
GC Standards

KEY WORDS

Students should understand the following terms before running this lab:

Adsorption, Absorbtion, Distillate, Eluate, Elution time, Polarity, Reflux band, Volatility.

PROCEDURE

Start heating the sandbaths as early as possible; it takes time for the temperature to rise and stabilize.

Review the assembly of the microscale kit with students. You may copy the handout for the students if you wish, or just instruct them in the assembly. The parts of this kit are expensive and breakable. Please urge caution. Also review use of the automatic pipettors and preset them to the required amounts. Caution students regarding use of the magnetic stirrers, and the process of cooling the vial in step 8. If time is a problem, students may stop after step 8, or after step 17 and resume at a later time. In step 17, the amount of anhydrous $MgSO_4$ should be sufficient to dry the product. The purification step is not essential to the lab. You may have the students do it if you choose.

The distillate may be tested by means of gas chromatography. First, as a teacher demonstration, inject about one microliter of the isopentyl alcohol standard into the gas chromatograph. Observe the time that it takes for the peak to appear. This should be done on both gas chromatographs. Then, a standard containing a mixture of water, isopentyl alcohol, isopentyl acetate and acetone, will be injected. Observe the time it takes for each of the four peaks to appear.

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Have the students record the retention times and names of the standards onto their data tables. Notice that the retention times will vary slightly with each machine due to the condition of its columm and rate of gas flow. Remind students that they must run their samples through the same machine for which they are recording standard retention times. For convenience, the machines will be labeled A and B.

Next, have students inject about one microliter of their distillate in turn into the gas chromatograph. This will take 8-10 minutes per group. Have students record the machine code number (A or B), and then identify the peaks by name and the percentage yield of each compound in their sample (see page 4). This information will be written on their computer printout first, and then transfered to their data table. The students will need to refer to the data from the standards to identify the components of their sample. Doing examples on paper prior to the lab day will greatly increase the students' understanding of these procedures. Theyshould see three peaks, one for water, one for isopentyl alcohol, and one for isopentyl acetate. Any other peaks are contaminants and can be ignored for this lab.

DATA COLLECTION AND CALCULATIONS

Calculate the number of moles of isopentyl alcohol
(1.0 mL x 0.813 g/mL)/88.2 g/mole = ***0.0092*** mole

Calculate the number of moles of acetic acid
(.550 mL x 1.06 g/mL)/60.1 g/mole = ***0.0097*** mole

What is the limiting reactant ***isopentyl alcohol***

Calculate the theoretical yield
0.0092 moles x 130 g/mole = ***1.196 g***

REFERENCES

1) L. F. Fieser and K. L. Williamson, Organic Experiments, 6/e, D. C. Heath and Co., Lexington, MA, 1987.
2) D. L. Pavia, G. M. Lampman, G. S. Kriz, R. G. Engel, Introduction to Organic Laboratory Techniques, A Microscale Approach, Saunders College Publishing, San Francisco, CA 1990.

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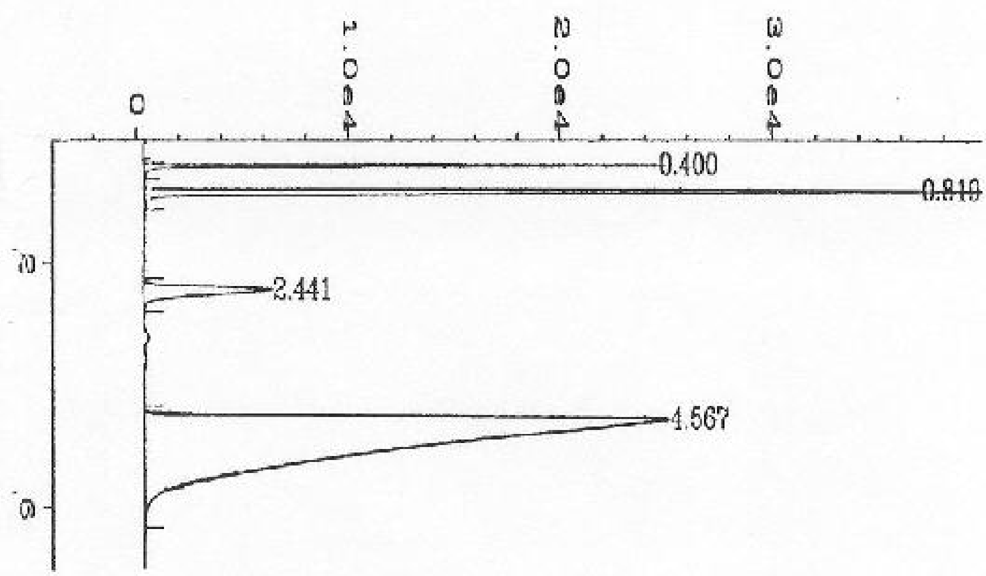
SAMPLE GAS CHROMATOGRAPH PRINTOUT



Area Percent Report

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Data File Name    : C:\HPCHEM\1\DATA\PHIL.D
Operator          : phil                       Page Number       : 1
Instrument        : ANALYZER1                  Vial Number       : 1
Sample Name       : ethan                      Injection Number  :
Run Time Bar Code:                             Sequence Line     :
Acquired on       : 30 Oct 92   11:18 AM       Instrument Method: TOPS.MTH
Report Created on: 30 Oct 92   11:25 AM        Analysis Method   : TOPS.MTH
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Sig. 1 in C:\HPCHEM\1\DATA\PHIL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	0.400	Water 37200	24900	BB	0.054	3.3417
2	0.819	156140 Acetone	46960	BB	0.050	14.0262
3	2.441	Isopentyl Alcohol 52374	6016	BB	0.135	4.7049
4	4.567	867482 Isopentyl Acetate	24798	BB	0.457	77.9272

Total area = 1113196

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USING THE MICRO MINI-LAB

Assembly of the Reflux and Distilling Apparatus

1. **HANDLE EQUIPMENT CAREFULLY.** Do not force any fittings or caps. Wash completely with distilled water followed by an acetone rinse at the end of the lab.
2. To construct the apparatus in Figure 1, you will need: a hot plate with magnetic stirrer, sand bath about half filled with sand, drying tube, 3mL conical vial, cooling condenser, magnetic spin vane, metal thermometer which reads to at least 200°C and its clamp, two 3' lengths of Tygon tubing, cooling tube (with cotton and $CaCl_2$) and a clamp for the apparatus.

3. Place sand bath on hot plate and heat to between 160°C and 180°C. Be careful not to overheat the sand bath. Adjust as necessary to maintain the temperature throughout the lab.

4. While the sand bath is heating construct the apparatus as in Figure 1. When making the connections between the different parts slide the cap over the ground glass followed by a large rubber O-ring. Secure the parts by screwing the cap down. Make sure O-rings are in place. Caps should be snug, but **DO NOT FORCE CAPS.** Before you connect the 3mL vial add the chemicals and the magnetic spin vane. Secure the apparatus to the ring stand with one clamp only so that the glassware can be rotated without breaking.

5. Hook up input cooling water at the lower inlet with the outlet at the upper inlet. **Slowly** turn on water and check for leaks.

6. When the reaction is complete, carefully remove the conical vial and transfer the solution as directed in the lab write-up. Disassemble the apparatus, let it cool and clean with distilled water and acetone.
7. Construct the distilling apparatus as in Figure 2. Use the 3mL conical vial, Hickman still, cooling condenser, hot plate and sand bath, thermometer, clamp, Pasteur pipet and magnetic spin vane with spinning band. Make sure the glass wool is wrapped around the bottom of the Hickman still. Connect cooling condenser as in Figure 1, but do not run water through it; the boiling point of the ester is high enough to make that unnecessary. Do not use the drying tube and leave the top open. Use the magnetic vane with spinning band and spin it as slowly as possible. Make sure that the extraction tube on the Hickman still is capped with a vinyl plug so that no oil will escape. At the end of the lab extract oil from the Hickman still with the Pasteur pipet and save in a sample vial (be sure to weigh the vial first.)