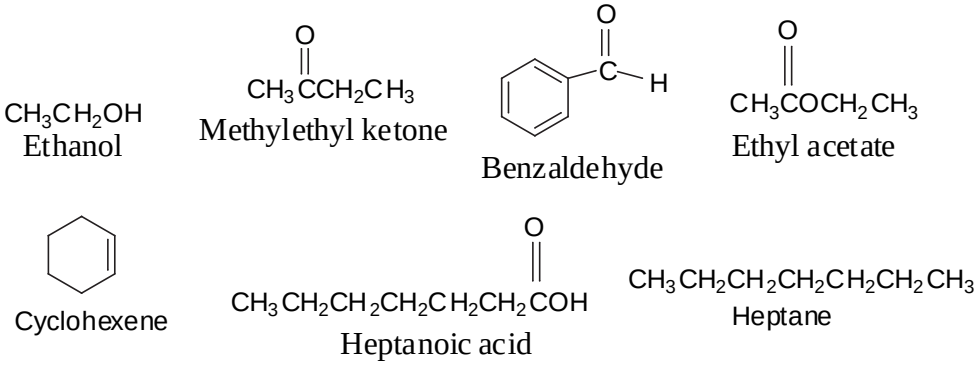
#17 Using the Infra-red Spectrometer to Analyze Liquids

Purpose:
You will identify an unknown organic liquid from its infrared spectrum.

Introduction:
Molecules absorb infrared radiation due to vibrations involving chemical bonds. Different types of bonds and thus, different classes of organic compounds absorb at different wavelengths. The amount of radiation absorbed versus wavelength is the IR absorption spectrum.
A flow chart will lead you to the identity of your compound in this experiment. The main feature you will be looking for is a double bond between C and O (C=O) called the "carbonyl", which absorbs strongly from 1820 to 1650 cm^{-1}. The units of cm^{-1} are wave numbers, the reciprocal of wavelength, $1/\lambda$, a unit often used by spectroscopists.

Your unknown can be one of seven possible compounds belonging to seven different groups of organic compounds. In most cases, the name of each compound indicates the group to which it belongs. This is typical in naming organic compounds.

1. Ethanol is an *alcohol*. Notice the ending 'ol'.
2. Methyl ethyl ketone has the word *ketone* in its name and is similar in structure to acetone, an ingredient in many nail polish removers. Note the ending 'one'.
3. Heptanoic acid is a *carboxylic acid*, similar in structure to the acetic acid in vinegar.
4. Benzaldehyde, as its name suggests, is an *aldehyde*. Each corner of the hexagon has one C and one H atom.
5. Heptane is an *alkane*, a hydrocarbon with only C and H in its formula.
6. Cyclohexene is also a hydrocarbon, but it contains a double bond, C=C and is known as an *alkene*. Each corner of the hexagon contains 1 C and 2 H's, except for the double bonded C's which are bonded to just one H atom.
7. Ethyl acetate, present in solvents such as nail polish removers, is an *ester*.



Apparatus:
Instructions for using the infrared spectrometer and the accompanying software can be found near the instrument.

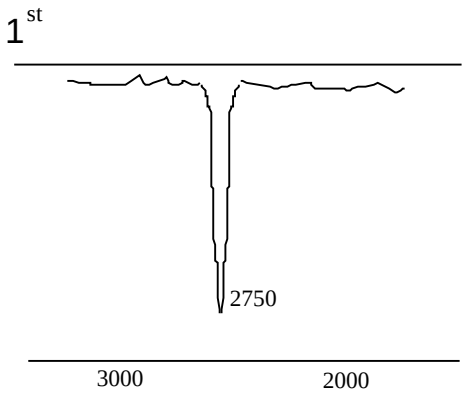
Procedure:
While another group is using the IR spectrometer, use one of the model sets to build the structure of the compounds used in this experiment. You can also practice drawing full structures for organic compounds in the data sheet.

To take a spectrum:
1. Get a small vial of unknown liquid and record the code.

2. Liquid samples are placed between a pair of transparent crystal windows or salt , NaCl, plates. Place 2-4 drops of sample on the surface of one plate near the center. Quickly place the second plate on top of the first one. (If necessary use the top plate to spread the sample on the bottom plate so there are no air spaces or bubbles.)
CAUTION: Remove all water and water containers from the vicinity of the IR.
Handle salt plates carefully using rubber gloves. They are expensive and they will be ruined upon contact with water.

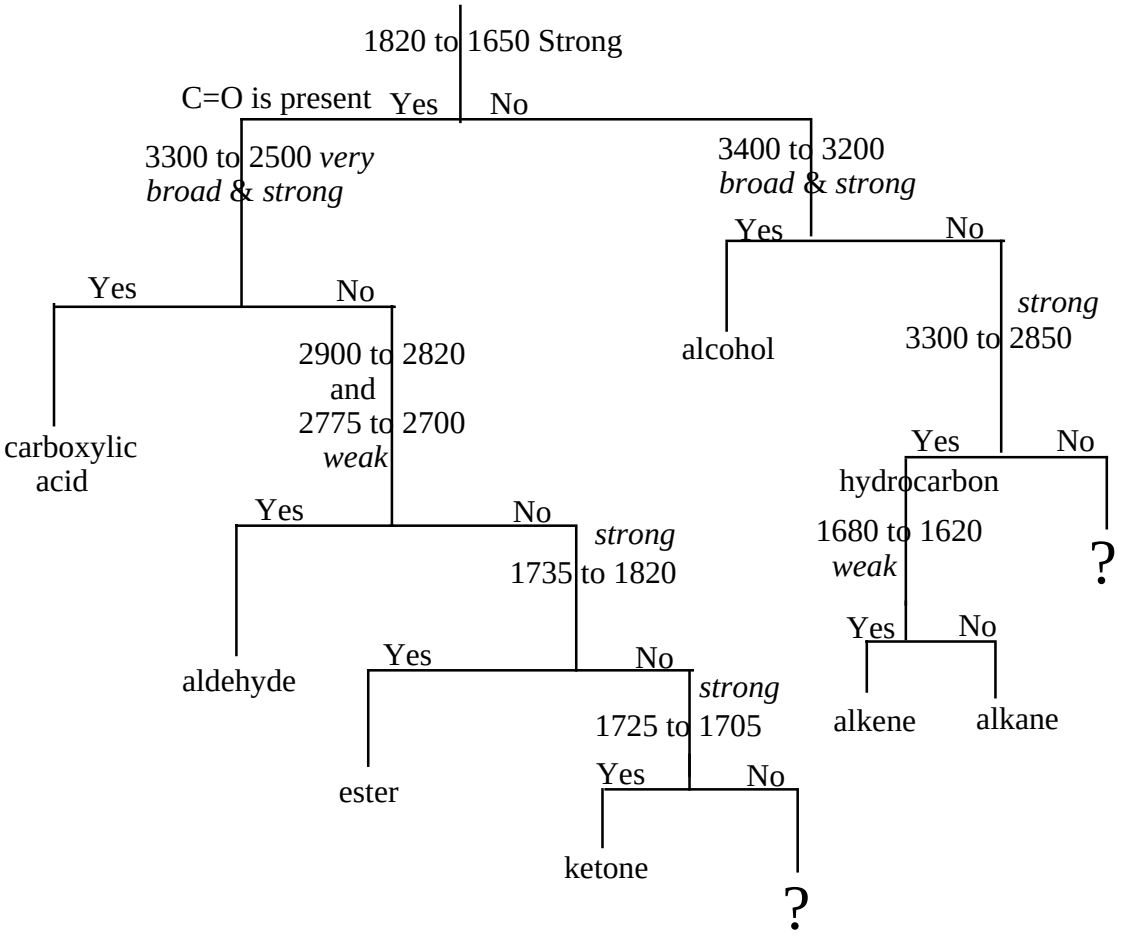
3. Place the NaCl plates in the salt plate holder, and place in the infrared spectrometer.

4. Run a scan of the sample. See instructions on using the IR spectrometer. The peaks are at the *bottom* of the print-out. *Note:* If the spectrum stays very close to the top of the chart and shows little change throughout, too little sample was used. Apply more sample and obtain another spectrum. If the spectrum goes to the bottom of the chart and the peaks are very wide and flat on the bottom, you probably used too much sample. Separate the plates, wipe one of the plates off, put them back together, and run another spectrum.



5. Label each of the major peaks in the regions used in the flow chart (see the next page). The software does not automatically label smaller peaks, but you can mark them by hand.
Look first for the carbonyl, C=O, then continue as the flow chart guides you. Identify the unknown liquid.

Flow Chart for Identifying Liquids:



Data and Results (Infrared Spectroscopy)

Name(s) _____

Taking a Spectrum

Unknown # _____

Identity of Sample:_____ (Attach spectrum or make a sketch of it, labeling peaks included in the flow chart. Explain how you identified the unknown.

Questions:

1. Why must water be kept away from this experiment?

2. Which two compounds have broad and strong peaks around 3000 cm^{-1}? Give their formulas. Which bond present in these two molecules is likely to be involved in the vibrations producing that absorption?

3. Convert 2000 cm^{-1} to a wavelength in nm. How does this compare to wavelengths of visible radiation? (1 cm = 10^7 nm)

_____

Writing Organic Formulas

Instructor's Guide
IR of Liquids

Identity of Sample: *Cyclohexene* (Attach spectrum or make a sketch of it, labeling peaks included in the flow chart. Explain how you identified the unknown.

There are two strong peaks at 2837 and 3022 and one very weak one around 1650.

There is no C=O peak around 1600 to 1800 cm^{-1}.

There is no broad and strong absorption from 3200 to 3400, so it can't be an alcohol.

There are peaks 2837 and 3022 in the hydrocarbon region (3300 to 2850).

There is a very weak band, less than 10% as intense as the other peaks in the spectrum, between 1680 and 1620. This is the double bond region, so the compound must be an alkene. The only alkene among the 7 compounds is cyclohexene.

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1. Why must water be kept away from this experiment?

2. Which two compounds have broad and strong peaks around 3000 cm^{-1}? Give their formulas. Which bond present in these two molecules is likely to be involved in the vibrations producing that absorption?

3. Convert 2000 cm^{-1} to a wavelength in nm. How does this compare to wavelengths of visible radiation? (1 cm = 10^7 nm)

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1. *NaCl salt plates dissolve in water .*

2. *Acids and alcohols. They both have an OH bond in their formulas.*

3. $\frac{1 \text{ cm} \times 10^7 \text{ nm}}{2 \times 10^3} = 0.5 \times 10^4 \text{ nm}$ *This is* 5×10^3*, 5000 nm.*

Instructor's Guide
IR of Liquids

Time: 45 min

Equipment and Materials per group

Items	**Number**	**Comment**
infrared spectrometer	1	
laptop computer	1	
printer and pack of paper	1	
salt plates	2	
7 liquid samples	1-2 mL	ethanol, benzaldehyde, ethyl acetate, methyl ethyl ketone, cyclohexene, heptane, heptanoic acid (7 liquids)
Pasteur pipets	20 per class	
250-mL beaker	1 per class	To catch runoff from cleaning
wash bottle of acetone	1	to clean salt plates
Kimwipes		
Model sets	6	To use in-between turns with the IR spectrometer
Safety glasses	1 per student	
Rubber gloves	1 box per class	

Ideas/ Information