Gas Chromatography - Analyzing a Mixture of Acetates

CHEM 315

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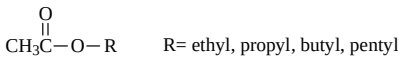
Due dates

- Today
  - Identification of an Unknown - IR Spectroscopy
  - Spectroscopy problems 1 and 2 (Part 2)
  - At the end of lab - your notebook carbon copies
- Next week
  - GC lab report (see instructions on website)
  - Spectroscopy problems 3 and 4

Purpose

- Today's lab, you will be analyzing a mixture of four alkyl acetates



R= ethyl, propyl, butyl, pentyl

- By the end of today, you will learn:
  1. how to ID a compound by its retention time via chromatography
  2. use relative peak areas to calculate mole percentages of a compound in a mixture

Why is GC used?

- Separate compounds in a mixture
  - Determines purity too
- Can analyze the components of a mixture
- Isolate pure compounds
  - Compounds are separated by the moving gas phase and a stationary liquid phase
  - The components of a mixture are separated based on boiling points

How a GC works

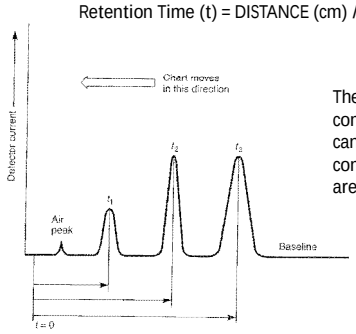
1. Sample is injected into chromatograph
2. Sample is vaporized in heating chamber
3. The sample meets the carrier gas that brings it to the column
   - This column has the stationary liquid phase (particles coated with liquid adsorbent)
4. the sample is subjected to gas-liquid partitioning processes
   - Separates the sample to its components as it passes through the column
5. Each component is detected by a detector that generates a signal that is recorded on a strip chart recorder
   - *VOILA!* You get your printed chromatograph!

Retention times

- The sample vapor is partitioned between the gas and liquid phases
- The time that different components in the sample spend in vapor phase is a function of their vapor pressure
  - The more volatile components (high vapor pressure/low b.p.) arrive at the end of the column first and pass into the detector

Retention times, con't

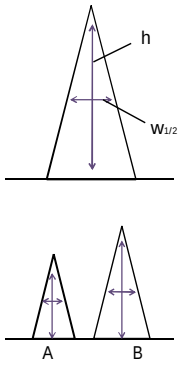
Retention Time (t) = DISTANCE (cm) / VELOCITY (cm/min)



The molar percentage composition of a mixture can be approximated by comparing the relative areas of the peaks

Retention times, con't

- Peak area = h x w
  - h = peak height
  - w = width of peak at ½ peak height
- Total Peak Area (TA) = A + B
- Mole Fraction (MF) = (A/TA) or (B/TA)
- Mole Fraction = MF x 100



Procedure

1. In your groups, inject a sample of the standard sample mixture into the entrance port of the GC
   - IMMEDIATELY mark the chromatogram paper to indicate the time of injection
   - Make sure you have recorded the chromatograph conditions in your notebook
2. Each student inject your unknown in the same manner
   - Remove your portion of the chart paper and clean syringe for the next student

Syringe care

- These syringes are expensive and must be handled with care!
- Syringe should be cleaned between injections
  - Draw up acetone then quickly squirt it into a small beaker containing a Kimwipe
  - Move plunger up and down a few times to dry the acetone
- Mark sure there are no air bubbles!

Injection technique

- Because you need to do things sequentially, make sure you know where the injection port is and where the start button on the recorder is
- Push needle into injection port and immediately press the plunger, then immediately press the start button on the recorder (or have one of your groups members do this) and mark the paper to indicate moment of injection
  - You might feel some resistance from the rubber septum in the injection port; this is alright
  - Gently apply some pressure until the needle is in the instrument all the way up to base of the needle

Calculations

- Since there is no internal standard component from which to calculate the actual moles of the components, you will need to calculate the mole percent of each component
- The mole ratios of each component will be done relative to ethyl acetate

Calculations, con't

- The mole ratio of each component relative to ethyl acetate in the unknown mixture is calculated from:

(1) $$\frac{area_i}{area_2} = \frac{mol_i}{mol_2} \bullet \frac{TR_i}{TR_2}$$

In here, 'i' stands for the number of carbons in the carbon backbone (2, 3, 4 or 6) and '2' stands for ethyl acetate (which has 2 carbons in the backbone)

- Since we will need the TR2/TRi ratios for the above equation, they will be calculated from the areas under the peaks in the standard equimolar mixture:

(2) $$\frac{area_2}{area_i} = \frac{TR_2}{TR_i}$$

Calculations, con't

1. First, from equation (2), each individual TR2/TRi ratio is calculated from the peak areas in the GC trace of the standard mixture
2. Using each TR ratio, the mole ratio of each component in the unknown mixture, relative to ethyl acetate, is calculated from equation (1)
3. From the mole ratios of the pairs, the mole percent of each component is calculated

Calculations, con't

- Then, for the multi-component mixture:

$$\%mol_i = \frac{mol_i / mol_2}{\sum_i \left(mol_i / mol_2 \right)} \bullet 100$$

OR

$$\%mol_i = \frac{\left(Area_i / Area_2 \bullet TR_2 / TR_i \right)}{\sum_i \left(Area_i / Area_2 \bullet TR_2 / TR_i \right)} \bullet 100$$

Calculations, con't

Example:

		EtOAc	PrOAc	BuOAc	HexOAc
Standard mixture	area	1.44	1.09	1.16	.975
	1b. TR_2/TR_i	1	1.33	1.24	1.48
Unknown mixture	area	2.14	2.18	2.12	1.54
	2b. mol_i/mol_2	1	1.35	1.23	1.07
	3.$mole_i$ percent	22%	29%	26%	23%

Final notes

- You will be working in groups of 3 today (by bench assignment)
  - **Each group** will inject one standard sample
  - **Each student** will inject an unknown sample
- Mark starting point of injection consistently to calculate RT's
- All calculations of the peak area are to be done exactly as shown in the Pavia text
  - Write directly on the chromatogram

Things to note in your lab notebook

- All chromatographic conditions
- Temperature of injection port
- Temperature of detector
- Flow rate of carrier gas
- Identity of liquid phase column
- Recorder chart speed