Organic Chemistry 211 Laboratory
Identification of Analgesic Drugs
(TLC: Thin Layer Chromatography)

Objective: A few common analgesic drugs will be analyzed and an unknown analgesic drug will be identified via the Thin Layer Chromatography technique.

Applications:
1. To identify substances. This identification could also be used to monitor the progress of certain chemical reactions.
2. To separate the components of a mixture.

Theory:
a) Based on: Solubility (like dissolves like) and Adsorption (adhering to a surface)
b) Phases:
   i. <u>Stationary phase</u>: the chromatography plate. In Organic Chemistry, usually the plate is a glass or a plastic plate sprayed with a layer of alumina or silica. ***This is the <u>more</u> polar phase. (note: use "more" or "less" when talking about polarity)***
   ii. <u>Mobile phase</u>: The solvent, running up the plate. Usually, in Organic Chemistry, organic solvents (or mixtures of organic solvents) are chosen as the mobile phase. ***This is the <u>less</u> polar phase.***

The differentiation of the polarities of the two phases gives the stain (substances at the bottom of the plate) the preference for the rate they travel with the solvent. Therefore depending on the similarity in its polarity with one phase or the other, a substance stain will travel at a certain rate. For example, a non-polar organic substance will travel at a faster rate, since it is more similar to the solvent, and not to the stationary phase. On the other hand, a more polar organic solvent will travel at a slower rate and hence will end up not so high on the plate.

c) R_f value: (distance traveled by the spot divided by the distance traveled by the solvent)

$$\text{Retention Factor: } R_f = \frac{D_{spot}}{D_{solvent}}$$

   i. No unit for R_f.
   ii. The units of the fraction HAVE to be the same, in order to cancel each other.
   iii. The concept of R_f gains relevance because a simple distance of the spot, varies with varying plate length. However, the ratio will ALWAYS stay the same for a given substance run with a given mobile and stationary phase.

Disposal Notes:
The leftover solvent should be placed in the nonhalogenated organic waste container.
The used chromatography plate should be placed in the broken glass waste container.
The used microcapillary tubes should be placed in the broken glass waste container.

Procedure: Obtain one TLC plate. Be careful not to touch the silica side of the plate with bare hands (The skin residues will mark and contaminate the plate and affect the accuracy of the TLC results). It is a safe practice to handle the TLC plates with tweezers.

Obtain the unknown drug from your instructor. Partially dissolve it in 2mL of methanol. Return the rest of the unknown to your instructor immediately.

Obtain a TLC chamber and place enough solvent to approximately 0.5 cm height. The solvent of choice is a 100% ethyl acetate.

Obtain an open microcapillary tube, and stain the middle spot of the TLC plate with the solution of the unknown. Stain the TLC plate with the 4 known ingredients and your unknown in the following order:

(A) Aspirin, (B) Ibuprofen, UNKNOWN, (C) Acetaminophen, and (D) caffeine

Wave it dry for about 30 seconds. Run the TLC. Then place the plate under UV light, and draw the outline of the spots visible under UV light, with a pale pencil mark. Then place the plate in an iodine chamber, cap and shake the chamber for about 20 seconds. Let the iodine and silica powder settle, and then take the plate out. Carefully draw the outline of the spots that are now visible. At this point, the plate is developed.

Measure all the distances traveled by the spots. Copy the picture of the developed plate, to the scale, in your laboratory notebook, label the spots (both shape and color) and also record all the measurements immediately. After obtaining the instructor's initial, place the used plate in the broken glass waste container.

For your report, calculate the R_f values. The identity of the unknown should be determined based on the R_f value and also the appearance of the spot(s).

Listed in the table below are the active ingredients of a few common analgesic drugs (possible unknowns):

Common Analgesic Drugs:

Drug (Brand Name)	Ingredients
Anacin	aspirin, caffeine
Motrin	Ibuprofen
Tylenol	acetaminophen
Vanquish	aspirin, acetaminophen, caffeine