

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

Disposal Notes:

The leftover solvent should be placed in the halogenated organic waste container.
The used microcapillaries should be placed in the broken glass waste container.

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

Procedure: Cut 2 TLC plates with (2x7)cm and (1x7)cm dimensions. Be careful not to touch the silica side of the plates with bare hands (The skin residues will mark and contaminate the plates and affect the accuracy of the TLC results). It is a safe practice to handle the TLC plates with tweezers. The larger and the small plates will be used for the TLC analyses of the known drugs and the unknown drug, respectively.

Obtain a TLC chamber and place enough solvent to 0.5 cm height. The solvent of choice is a 1:1:2 mixture (by volume) of petroleum ether, methylene chloride, and ethyl acetate, respectively. First run a TLC of the 4 known drugs. 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 10-20 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 and also record all the measurements immediately.

Calculate the R_f values for all the spots on the plate.

After showing the actual TLC plate to your instructor and obtaining his/her initials, place the plate in the broken glass waste container.

Obtain the unknown drug from your instructor. Partially dissolve it in 2mL of ethyl acetate. Obtain an open microcapillary tube, and stain the second (small) plate with the solution of the unknown. Wave it dry for about 30 seconds. Renew the chamber by pouring out the old solvent from the first run, and replacing it with a fresh portion of the solvent. Run the TLC. Develop, measure, record the distance(s) and calculate the R_f values as before. The identity of the unknown should be possible to be determined at this point, 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:

Common Analgesic Drugs:

| Drug (Brand Name) | Ingredients |
|--------------------------|----------------------------------|
| Anacin | aspirin, caffeine |
| Motrin | ibuprofen |
| Tylenol | acetaminophen |
| Vanquish | aspirin, acetaminophen, caffeine |