Lab Information Sheet

CH 203 2019

Experiment 2

Acetylsalicylic Acid + TLC
(according Pavia et.al., Introduction to Organic Laboratory Techniques. See:
drugs, Techniques: Thin-Layer Chromatography, Heating; Cooling; Crystallization

LAB NOTES:

1. Write up the two experiments separately

2. The second TLC plate should be spotted with pre-made solutions of Thomapyrin
and Eudorlin, a solution you make of your analgesic from week 1, a solution of your
acetylsalicylic acid from week 2, an unknown tablet (prepare solution yourself) which
you will be given, and the reference mixture (6 spots). Use the 50% methylene
chloride- 50% ethanol mixture to dissolve the solids and use the ethyl acetate/acetic
acid mixture as the developing solvent. If you do not have your week 1 analgesic, get
some from another student. To check that the solutions which you prepare are
concentrated enough look for the spot on the spotting line with the UV lamp before
developing the plate. Please use your reference mixture to identify the content of
active ingredients in Thomapyrin and Eudorlin.

3. Be sure to do a separation and purification scheme

Lab quiz: A five minute quiz will be given at the start of lab
Be able to write the reaction equation for the synthesis of acetylsalicylic acid
using chemical structures.

Pre-lab questions
1. Calculate the theoretical yield of aspirin in mg if 150 mg salicylic acid and an
excess of acetic anhydride are reacted. If a student used 150 mg of salicylic acid and
isolated 90 mg of product, what is her percentage yield?
2. What would happen if the spotting line and positions were marked on the plate
with a ballpoint pen?

Post-Lab questions
1. Why is acetic acid added to the ethyl acetate developing solvent? What effect
does it have on the carboxylic acid groups of the acetylsalicylic acid and the
ibuprofen? How would the Rf value for these compounds be affected if the acid were
not added? Would they tend to move faster or stick to the silica gel plate better?

2. What do you think would happen if you used ethanol as a developing solvent for
the TLC of analgesics instead of ethyl acetate?