Experiment 4

Thin Layer Chromatography

Chromatography: the science of separation

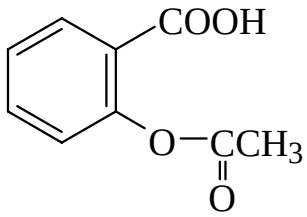
1. Thin layer chromatography — analytical to semi-preparative qualitative
2. Column chromatography — preparative
3. High performance liquid chromatography — analytical, quantitative
4. Gas chromatography — analytical, quantitative
5. Size exclusion chromatography, electrophoresis, ...

Thin layer chromatography

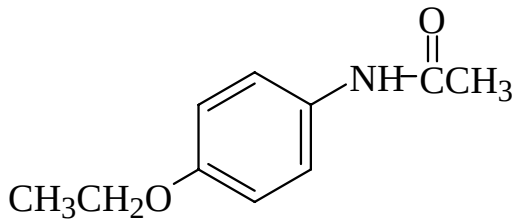
Uses: fast, relatively inexpensive

Used to follow a reaction, to identify optimal reaction conditions

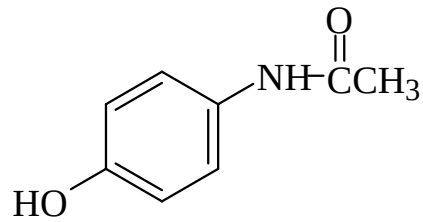
Used for identity, qualitative test for purity;



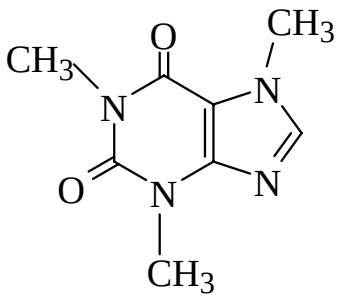
Aspirin



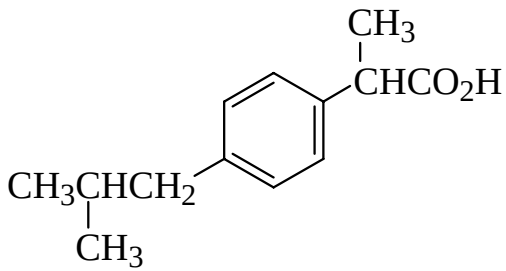
Phenacetin



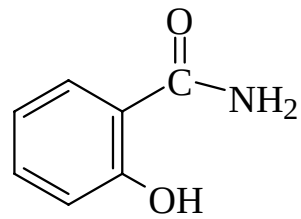
Aetaminophen



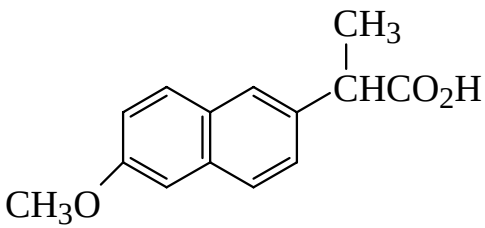
Caffeine



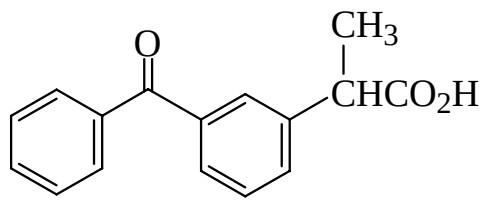
Ibuprofen



Salicylamide

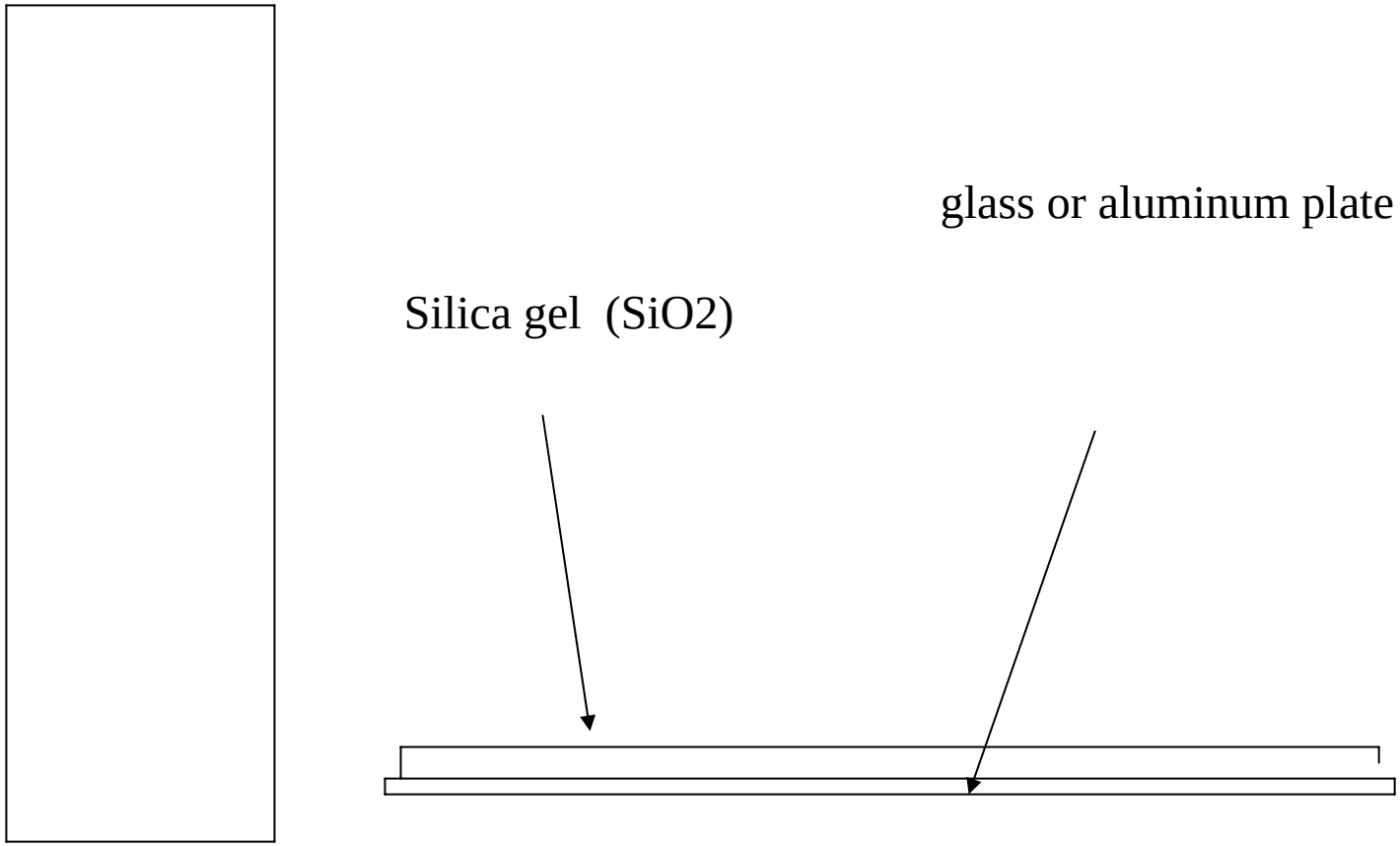


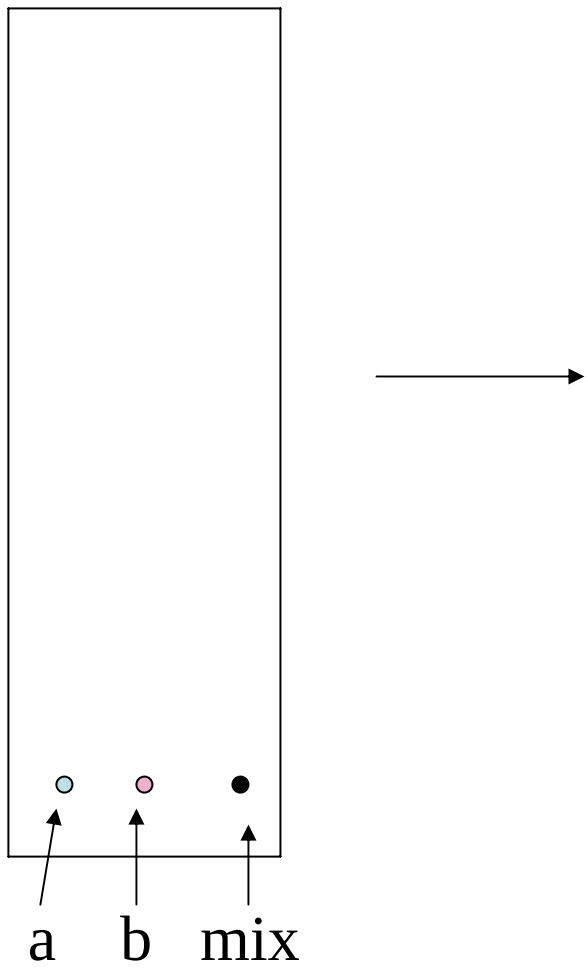
Naproxen

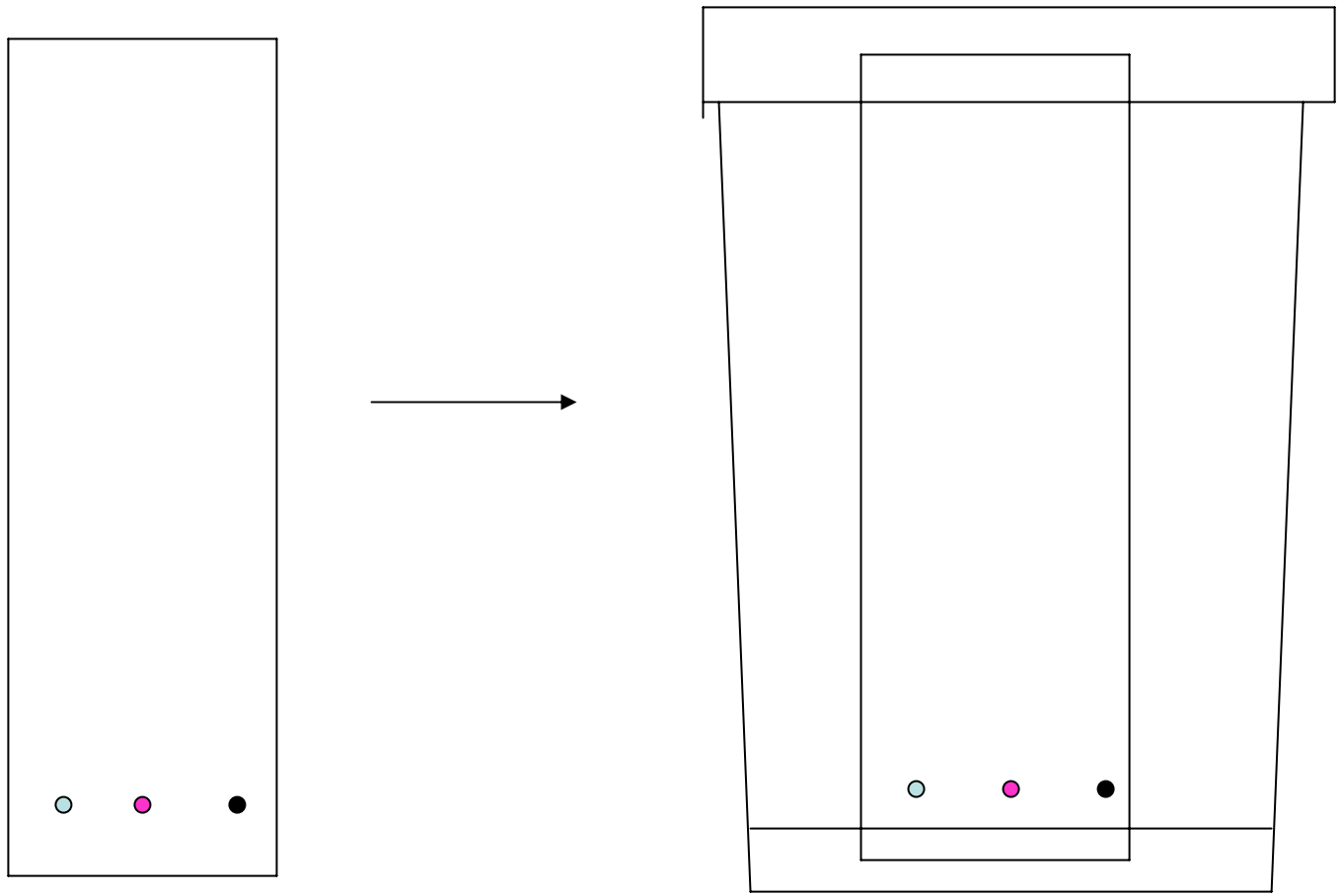


Ketoprofen

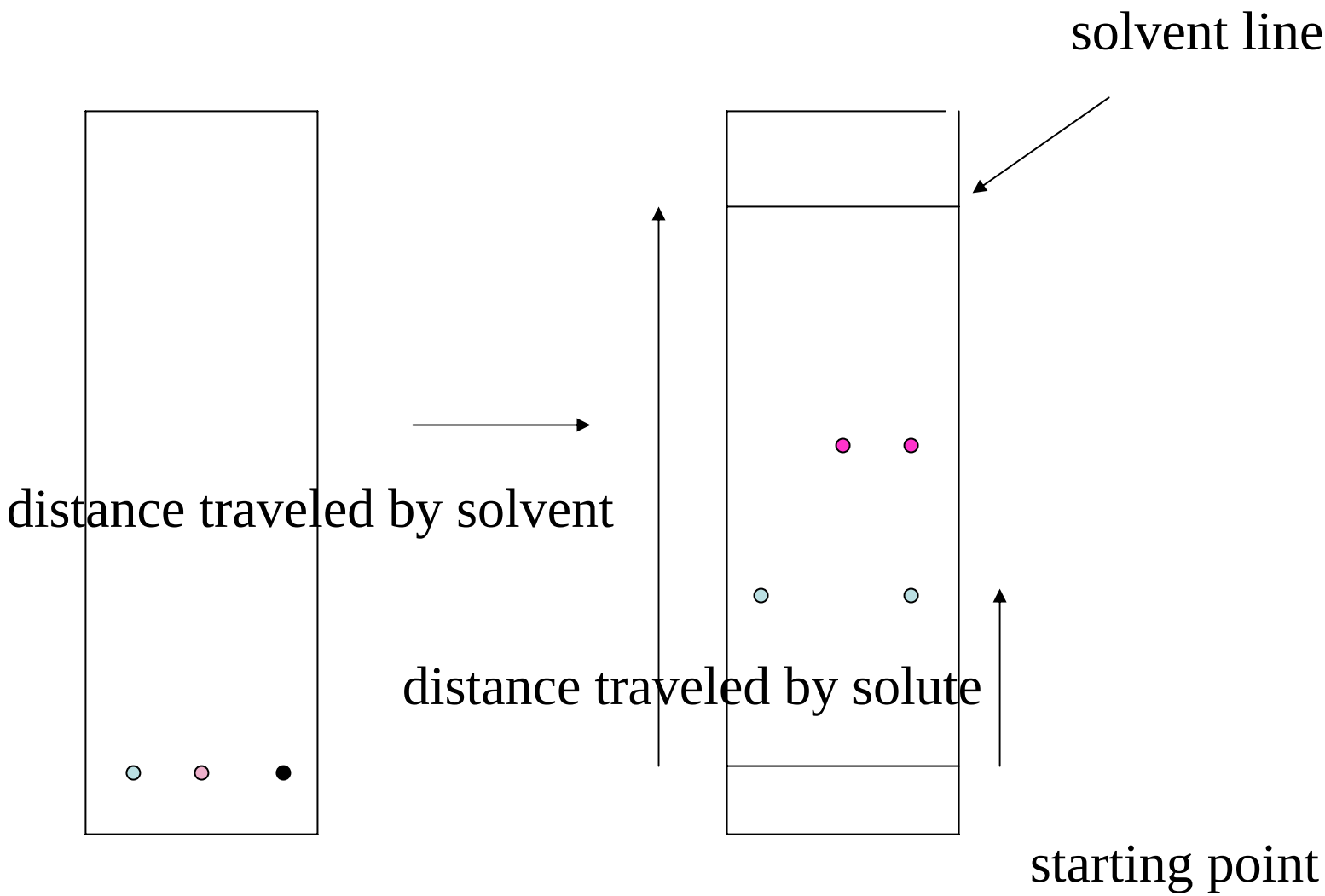
Thin Layer Chromatography




a
b
mix



r_f = distance traveled by solute/distance traveled by solvent



It is the polar sites that are responsible for adsorbing solutes as they pass by. Depending on the polarity of the solute and adsorbent, some solutes are adsorbed more strongly than others. Adsorption is a competitive phenomena. The solvent as well as the solute is in competition for these active sites. If the solvent used is too polar, it will compete for these sites and preferentially bind to them leading to and $R_f > 0.8$.

R_f = distance traveled by the solute/ distance traveled by the solvent

R_f values are always < 1

$R_f \approx$ 0-0.2; solvent not polar enough

$R_f \approx$ 0.8-1.0; solvent too polar

Reverse Phase Chromatography

Using a polar often aqueous solvent and a stationary phase that is non-polar. The non-polar substances are adsorbed more stronly than the polar ones. Generally the stationary phase which can be silica has hydrocarbons attached to the $SiO\text{-}CH_2R$

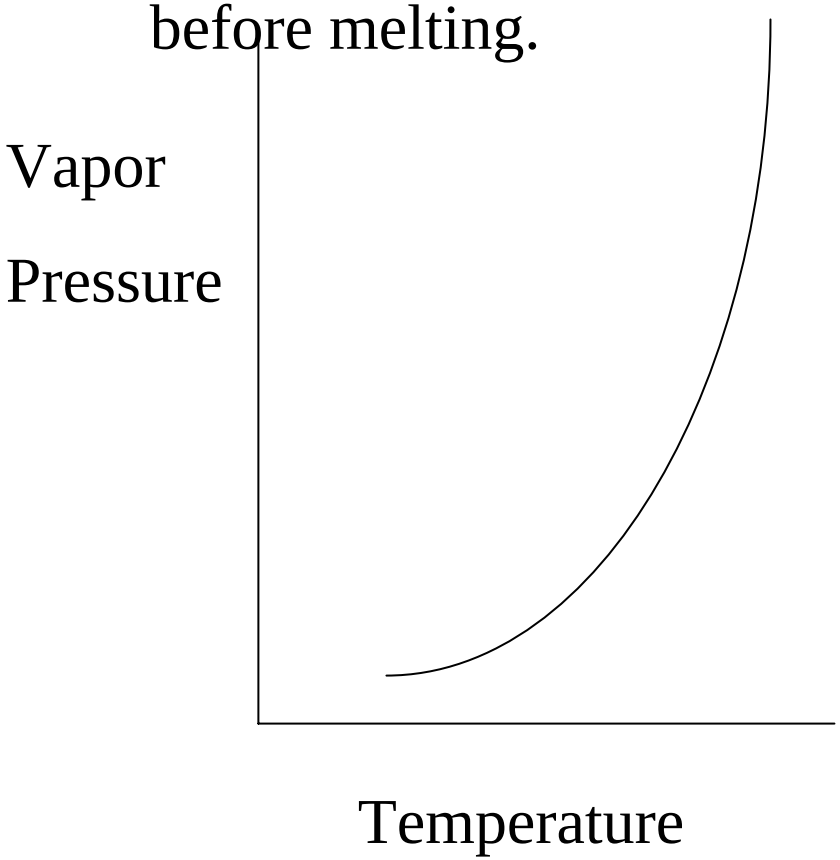
Some solvents arranged in order of decreasing polarity going down and from left to right

water
acids (acetic acid, propionic acid)
amides (N,N-dimethylformamide)
alcohols (methanol, ethanol)
amines (pyridine, trimethyl amine)
nitriles (acetonitrile)
ketones (acetone, 2-butanone, diethylketone)
esters (methyl acetate, ethyl acetate)
halides (chloroform, methylene chloride, carbon tetrachloride)
ethers (tetrahydrofuran, dioxane, diethyl ether)
aromatic hydrocarbons (benzene, toluene, xylenes)
alkanes (hexane, heptane)

Using Mixed Solvents

Occasionally, no one solvent is found to be satisfactory. In these circumstances, using a mied solvent containing a polar and less polar solvent may prove to work best. Miscible solvents can be used in any proportion.

Sublimation: A process by which a volatile solid passes into the gas phase before melting.



A Typical sublimation apparatus