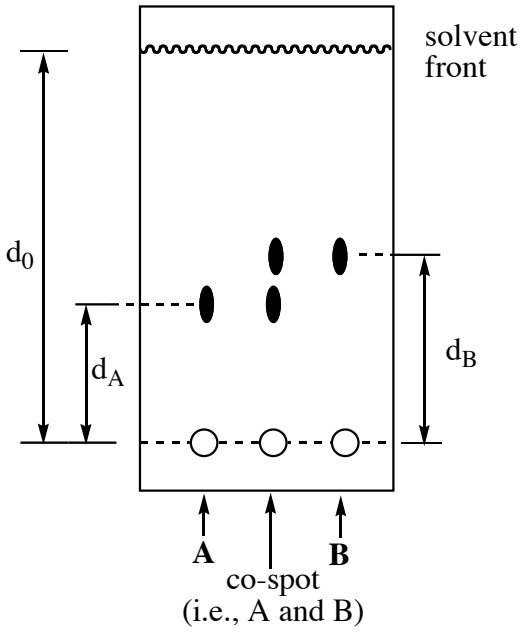
Thin-Layer Chromatography (TLC) (see: Zubrick, pp 225-237).

I. TLC - A highly sensitive analytical technique used to:
(1) Determine the purity of a sample (# of components in a mixture)
(2) Determine the identity of two compounds
(3) Monitor the progress of a reaction

- Requires *only microgram or* γ (10^{-6} grams!) quantities of a sample.
- Every reaction to be performed in the lab should be monitored by TLC.

II. TLC plates: glass plates coated with silica gel that is impregnated with a fluorescent dye (use a 254 nm UV lamp for detection of the spots).



d_0 travel distance of the solvent
d_A travel distance of compound A
d_B travel distance of compound B

R_f (ratio to front) value for A = d_A / d_0

R_f (ratio to front) value for B = d_B / d_0

- By definition, Rf values are between 0 and 1.0.
- Best resolution is achieved in the Rf 0.3 - 0.7 range.

- Make sure to dissolve only a mall amount of a smaple (2-3 mg) in a small volume of solvent (usually 0.5 - 1.0 mL of dichloromethane).
- You need to dissolve a sample in a solvent even if the sample is a liquid.

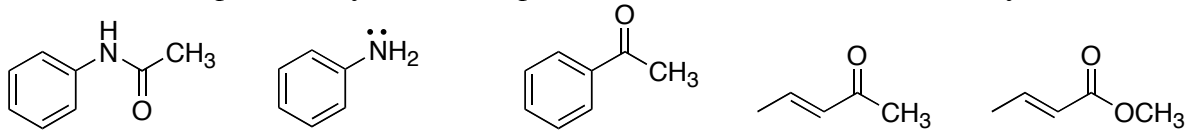
III. Detection or visualization of TLC spots.

(1) UV lamp – The UV lamp in the lab emits UV light having the 254 nm wavelength.

The silica gel TLC plates we use have an inorganic fluorescent agent (<0.5%) impregnated into the adsorbent layer. When illuminated with an ultraviolet (UV) lamp, the absorbent then glows the pale green or blue colored fluorescent light. However, dark spots show up at the places where UV-absorbing organic compounds are located on the TLC plate because they quench the fluorescence.

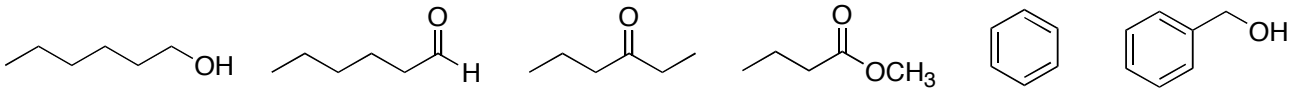
To be detectable under the UV lamp, compounds must have strong UV absorption in the 220 – 280 nm range.

For example, hetero atom-substituted benzene compounds, and aryl ketones/aldehydes.
The 254 nm UV lamp can easily detect the spots for α,β-unsaturated ketones/aldehydes/acids/esters.



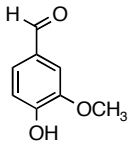
detectable unde the 254 nm UV lamp

However, non-conjugated aldehydes/ketones/acid/esters/amides, alkyl substituted benzene compounds are normally not detectable under the 254 nm UV lamp.



not detectable unde the 254 nm UV lamp

(2) Vanillin stain: a generally applicable staining agent for TLC analysis; gives a range of color upon heating; effective in detecting alcohol, aldehyde, and ketone compounds.

vanillin 

Vanillin stain: Vanillin is dissolved in ethanol together with conc. H_2SO_4.

After dipping the dried developed TLC plate into the vanillin stain (solution), the plate needs to be heated (on a hot plate) in order to visualize the spots.

(3) Iodine jar: C=C bond-containing compounds can be detected by exposing the dried, developed TLC to the iodidne vapor.

IV. Silica gel TLC

Silica gel (silicic acid polymer): highly polar stationary phase
Solvents used to develop TLC plates: less polar moving phase

"**More polar**" compounds tend to better interact with
silica gel ⇒giving **lower** Rf values in a given solvent system

In general,
Fast-moving; higher Rf ← Slow-moving; lower Rf

alkanes alkyl halides	> alkenes aromtaic hydrocarbons aromatic halides	> esters ethers ketones aldehydes	> amides	> alcohols	> phenols	>> carboxylic acids

Note: The use of a "***more polar***" solvent system ALWAYS results in a ***higher*** Rf for any compound.