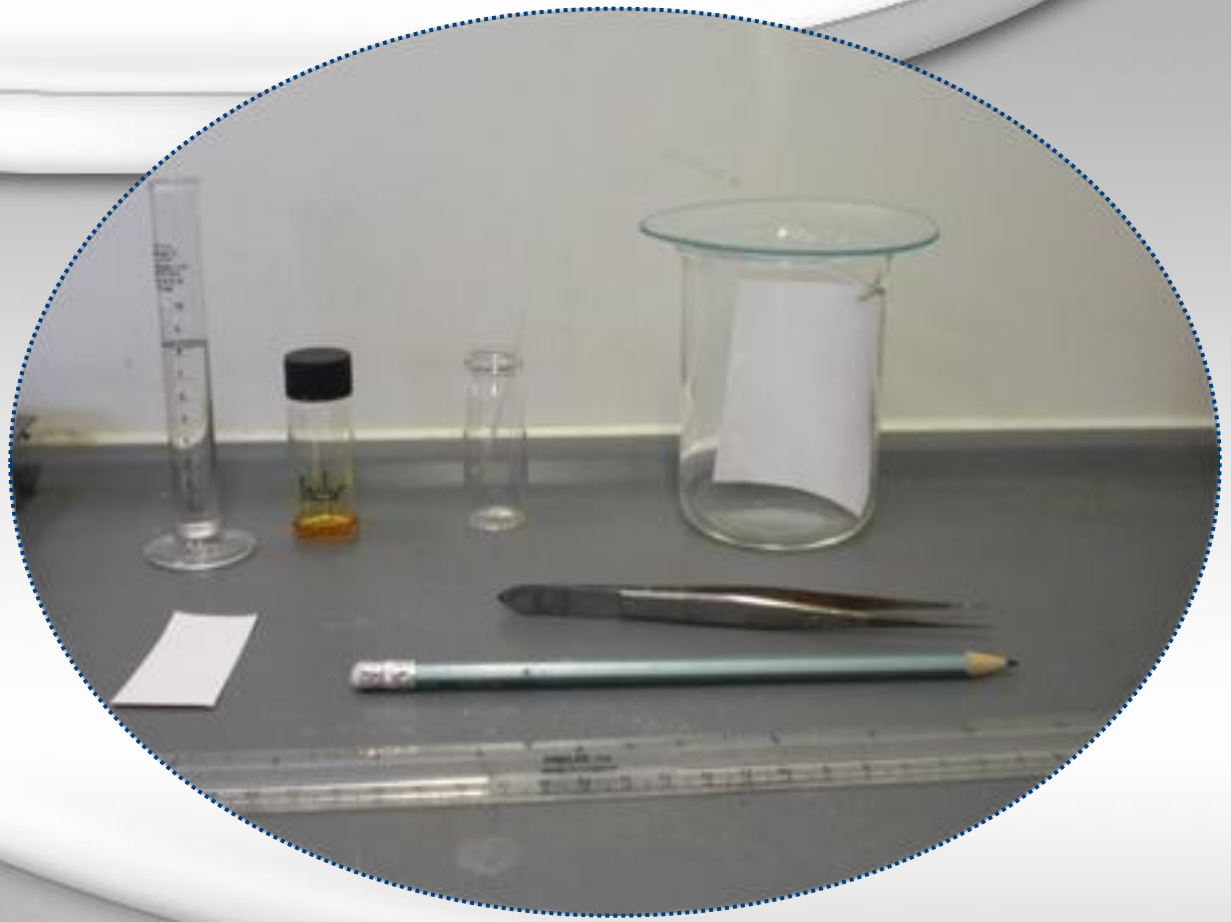
Thin layer chromatography (TLC)



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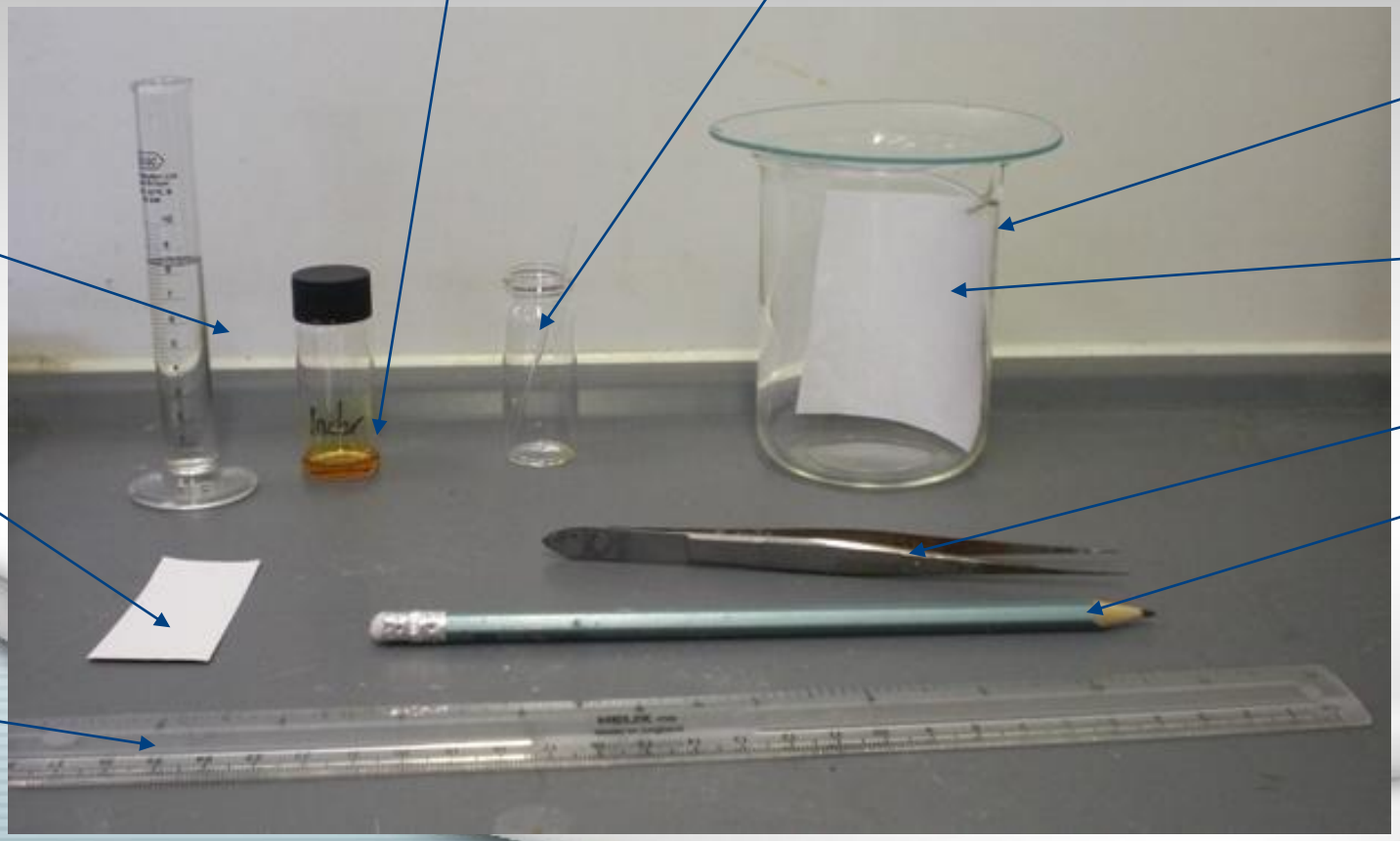
Thin layer chromatography is used extensively for the analysis of mixtures, following the progress of reactions and as a check for purity.

The equipment needed to run a tlc is shown below



Your sample should contain a small amount of mixture (1-3mg) dissolved in two or three drops of a suitable, volatile solvent (eg. ether or ethyl acetate. Your compound must be soluble in it).

sample vial

1-3mg of compound dissolved in a few drops of volatile solvent

The developing tank for a TLC should consist of a beaker/jar with a lid

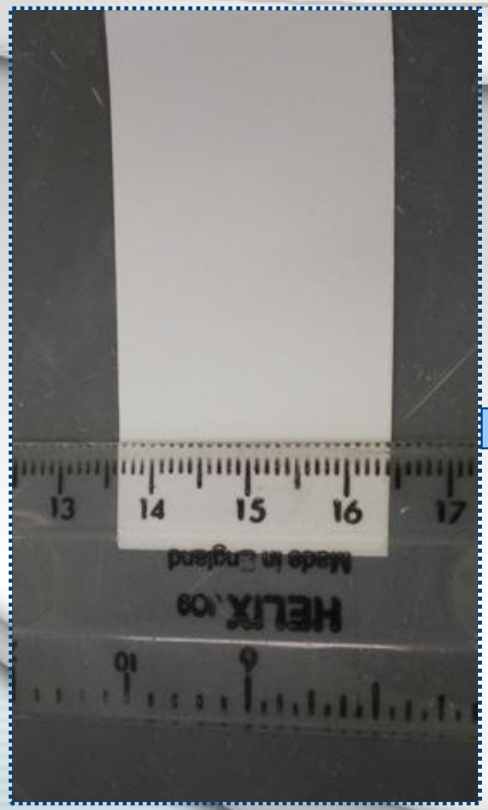
watch-glass cover

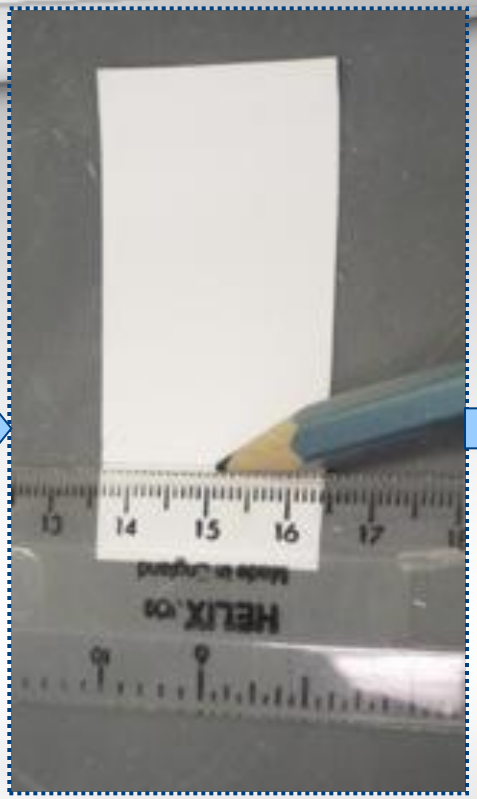
Filter paper should be used saturate the beaker atmosphere with solvent vapour

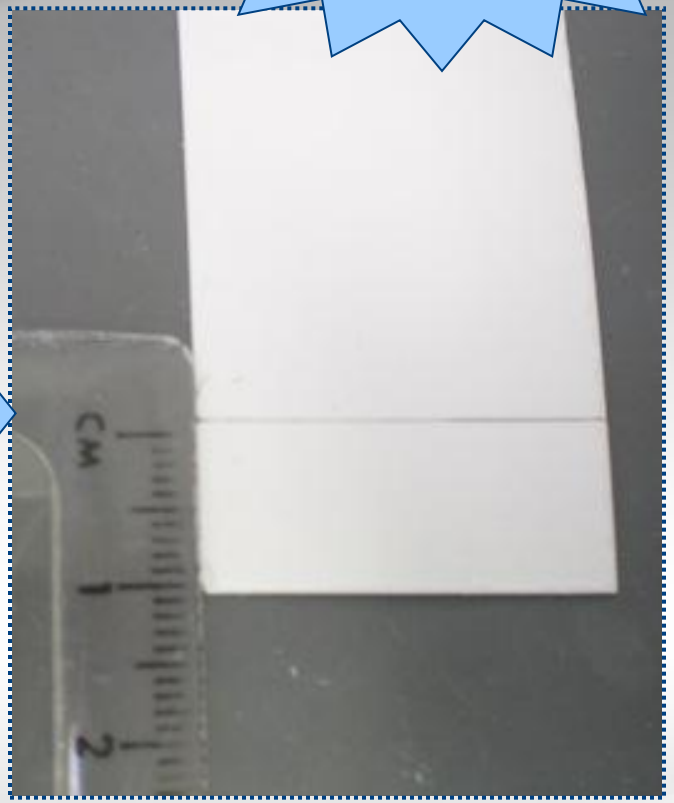
a clean, dry beaker

The TLC plate consists of a thin layer of silica gel on an aluminium foil backing.

Remember to use a pencil for drawing your line. If you use ink it will interfere with your TLC.







Use a pencil and ruler to draw a line about 1 cm from the bottom of the TLC plate.

When setting up the development tank it is a good idea to pour the eluent in before you spot your sample on the TLC plate. This is to ensure that the atmosphere in the beaker is saturated with solvent vapour.

Pour sample in

After you have added the eluent to the beaker, swirl it around the edges and replace the watch-glass cover

The eluent to be used depends on the sample. Polar compounds will adsorb strongly to the silica and require a polar eluent to move them up the plate, whilst non-polar compounds will move up the plate with a relatively non-polar eluent

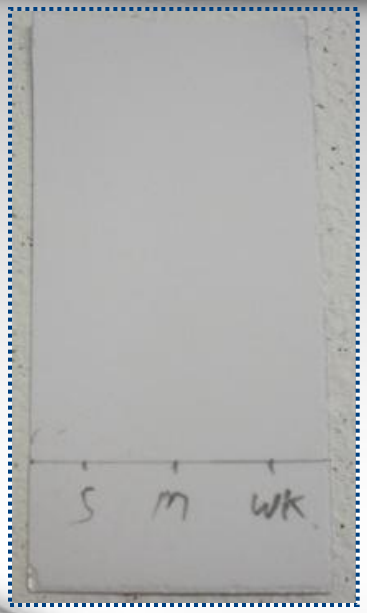
Aim to get the spots moving about half way up the plate for best results.

If the spots are close to the eluent front then decrease the eluent polarity.

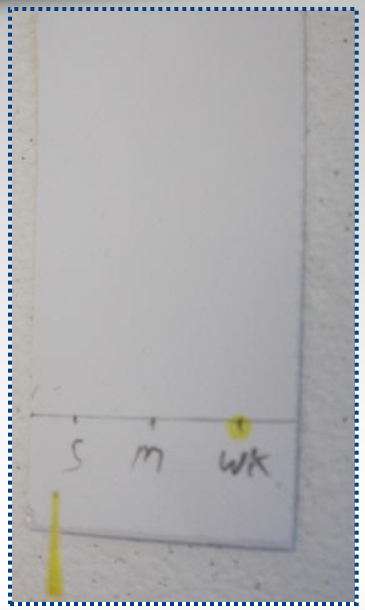
Least polar	Light petroleum (b.pt. 40-60° C)
	Cyclohexane
	Pentane
	Toluene
	Dichloromethane
	Diethyl ether
	Ethyl acetate (Ethyl ethanoate)
	Acetone (propanone)
	Ethanoic acid (Acetic acid)
Most polar	Methanol

If the spots remain on or near the baseline increase the eluent polarity.

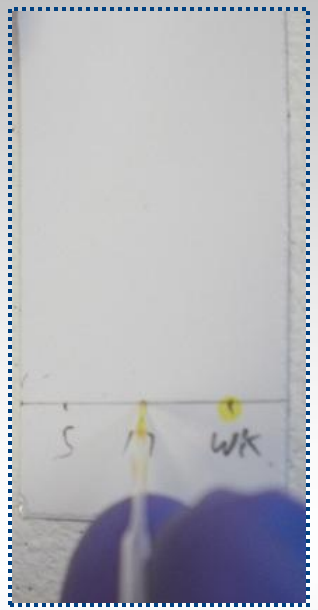
When spotting you sample onto your TLC plate, you should label each spot in pencil. Below the labels ‘s’, ‘m’ and ‘wk’ are used to represent strong, medium and weak samples, but you are free to label them however you wish.



Label the spots



Spot your samples onto the TLC plate using a micropipette



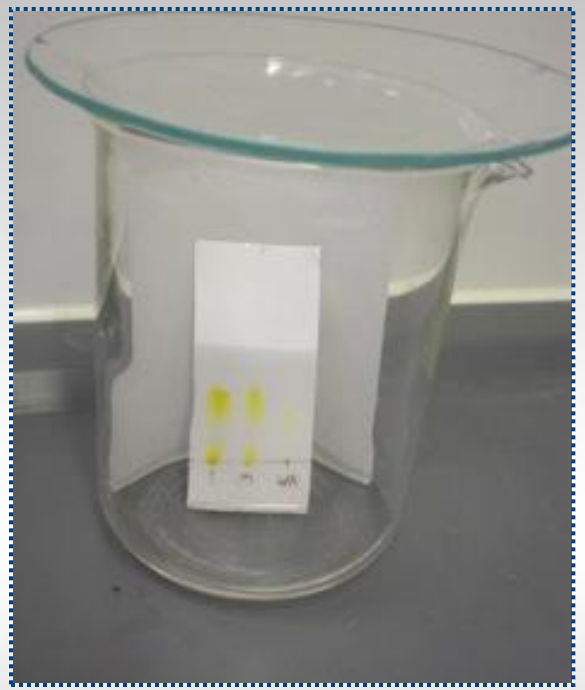
TLC plate with all three sample spots

Tweezers should be used to transfer the TLC plate into the developing tank. Allow the back of the plate to rest on the sides of the beaker at a slight angle.

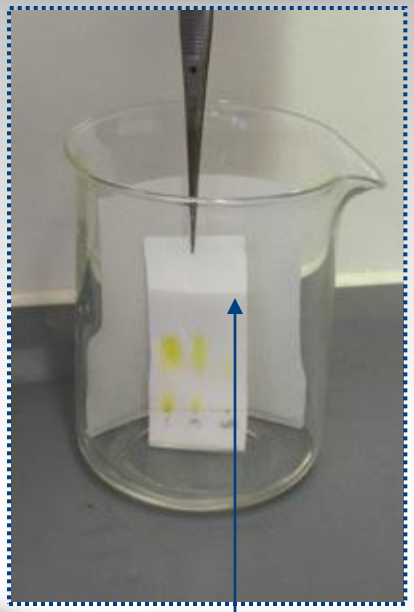
Make sure the level of eluent is below the base line on the TLC plate

Replace the watch-glass cover and leave the plate to develop.

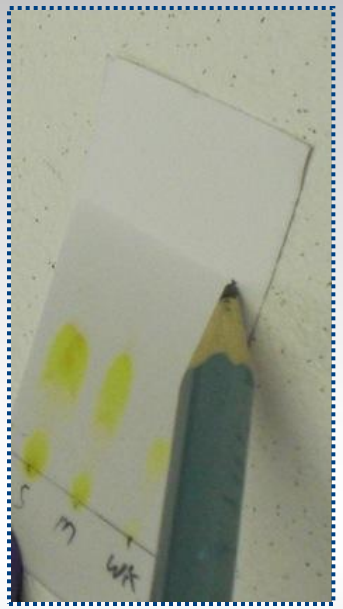
Allow the solvent front to rise up the TLC plate in a horizontal line until it reaches about 1 cm from the top of the plate.

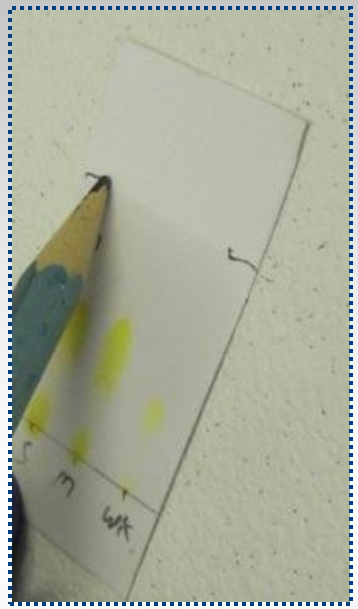


Remove the TLC plate from the developing tank and mark the solvent front on the TLC plate before it starts to evaporate.

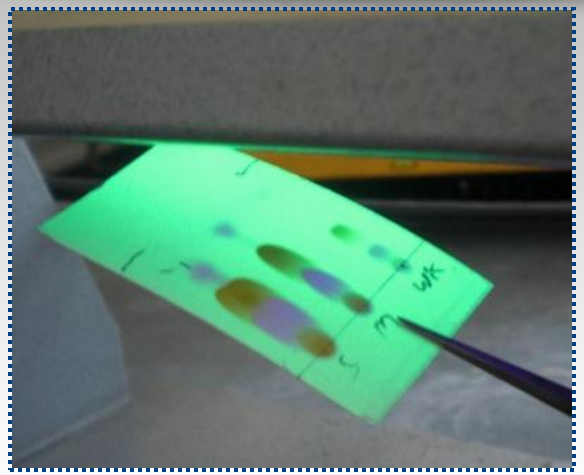


solvent front



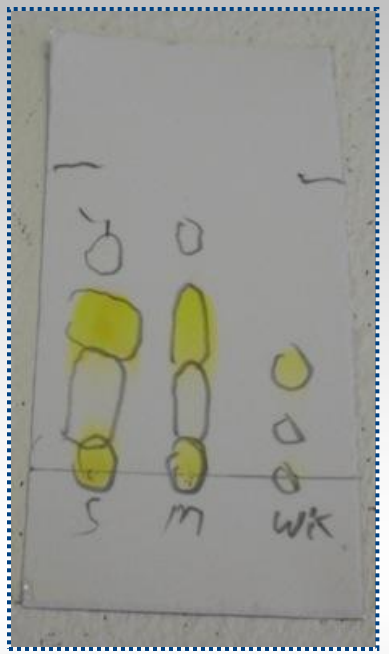


Unless the material being analysed is coloured the plate will need to be treated in order to see the spots and measure the distance travelled. UV light can be used to observe the spots and for some analysis the plate will need to be stained.



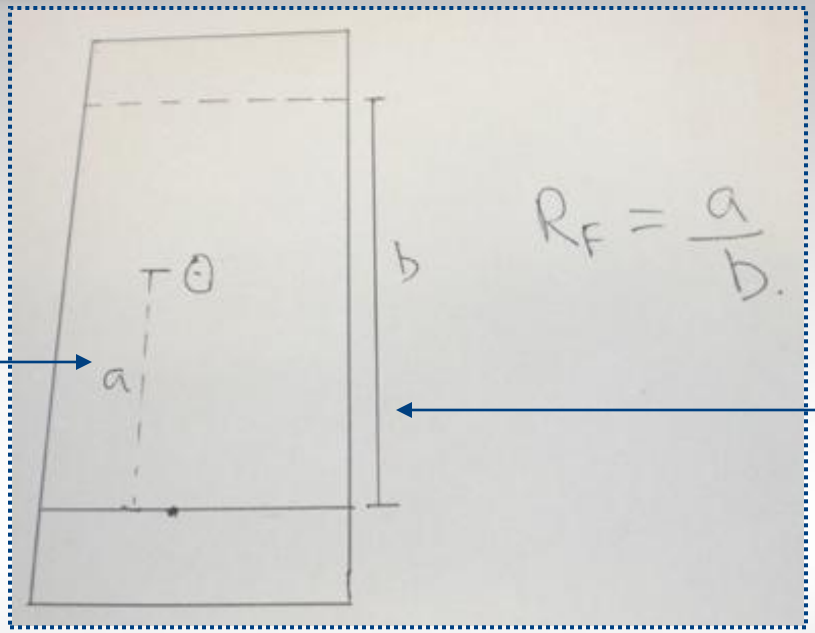
TLC plate under UV light. Observation of the additional spots.

TLC plate in normal light. Observation of the original spots.



TLC plate with the original and additional spots circled.

The retention factor (R_f) is a useful measurement which can be made from a developed TLC plate. It is the relation between the distance the compound has travelled and the distance travelled by the solvent front.



Distance travelled by the compound

Distance travelled by the solvent