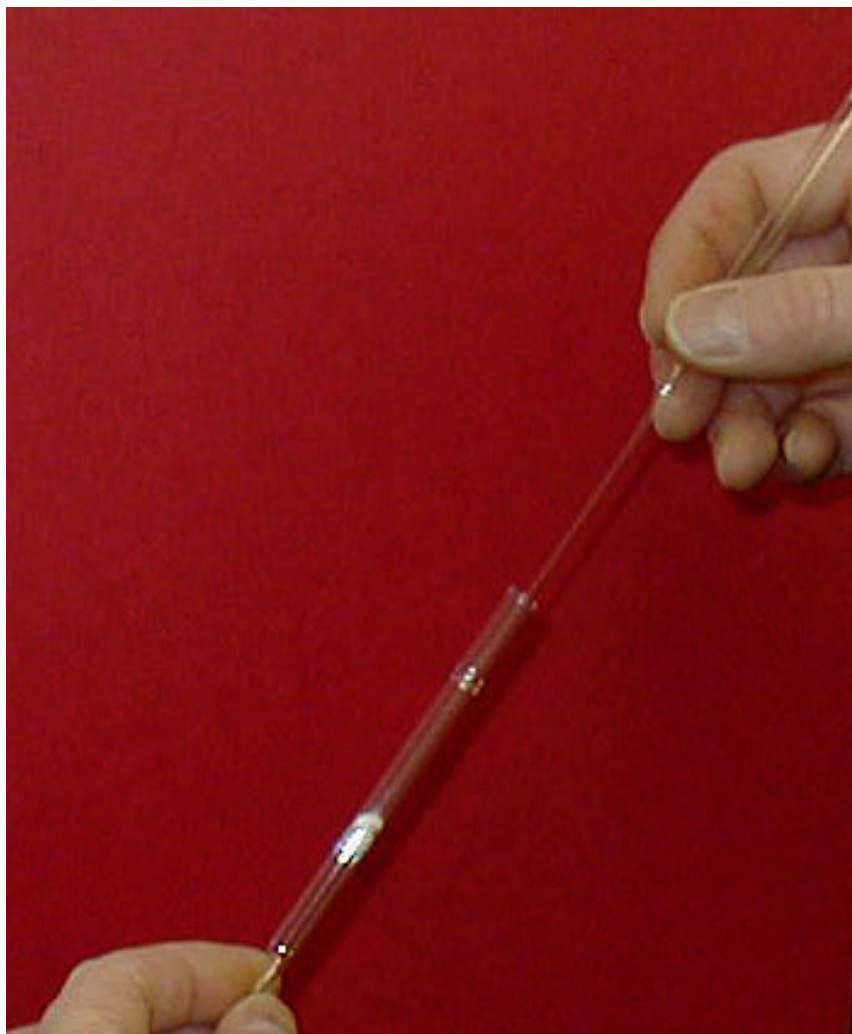


Chromatographing the Ester

Loosely roll a very small piece of cotton into a cylindrical shape, insert it into a pipet and, using a second pipet, loosely push the wad of cotton down into the small end of the pipet. The keyword is loose. Packing the cotton too tightly will cause a very slow flow through the column. The purpose of the cotton is simply to keep the silica gel in the column.

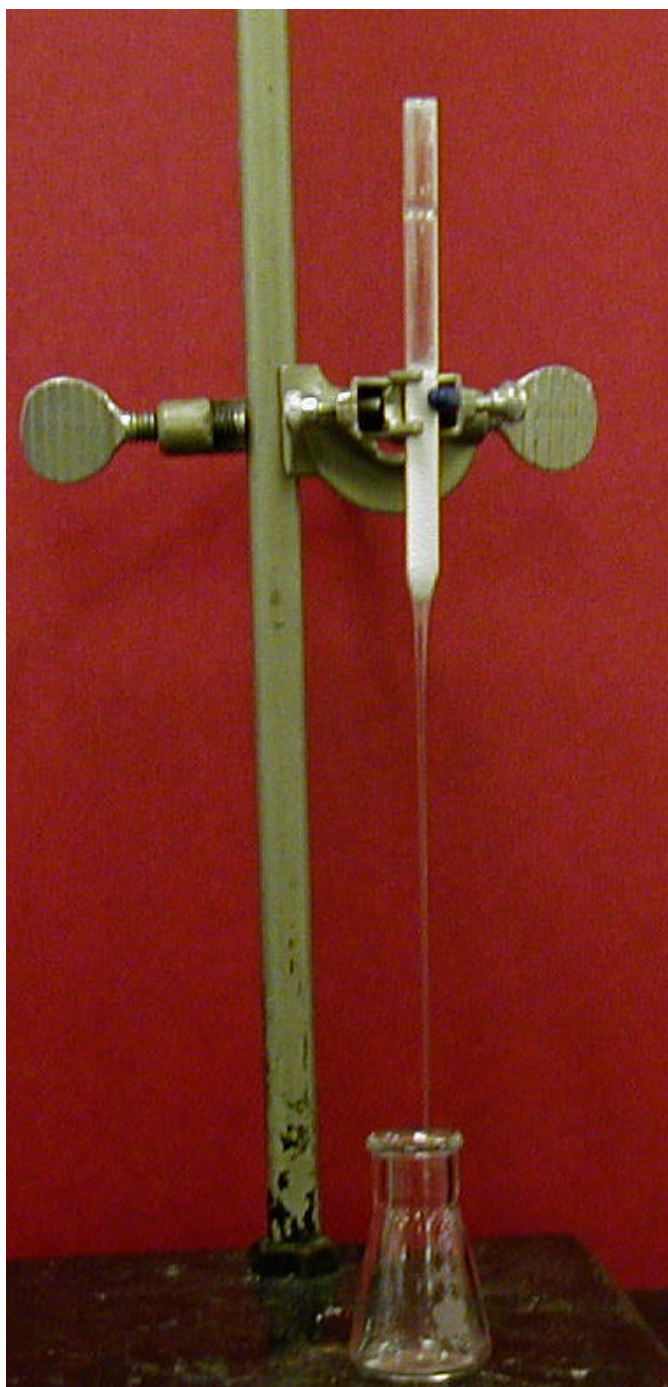




Scoop up some silica gel into the wide end of the pipet, turn the pipet upright, and tap the pipet gently so that the silica gel packs evenly down into the small end. Continue adding silica gel until the pipet is about half full.



Clamp the pipet upright on a ring stand using a small 3-pronged clamp. You are now ready to begin the chromatography.



Add about 1 mL of ligroin to the top of the column and allow it to drain through the column into a small Erlenmeyer flask. It will take a few minutes for the ligroin to pass through the column and begin to drain. If draining is too slow it may be that the cotton was packed too tightly. What

is too slow? The ligroin should drip at about a drop per second or two seconds when the column is filled with ligroin - note that as the ligroin level becomes lower, the drip rate will decrease. If it drips at a rate of a drop per 10 seconds or longer it's probably packed too tightly. If so, prepare another column. When the ligroin drains down to the top of the silica gel it is time to add the ester/ligroin solution. This must be added before the level of ligroin goes much below the top of the silica gel (in other words do not let the column run dry). As soon as the first portion of ester/ligroin is added, change the collection flask to a clean, dry product vial. The ligroin/ester solution should be added in portions until all of it has been added to the column. Allow the last ligroin/ester solution to drain down to the top of the silica gel then add a 0.5 mL portion of fresh ligroin to the column and allow this to drain into the product vial. This extra ligroin ensures that all of the ester is pushed through the column into the product vial. Allow as much of the ligroin as possible to drain into the product vial. At this point you are finished with the chromatography so it is ok to allow the column to run dry.

