

Textbook procedure for running a column:

1. Find a solvent system that separates the mixture and move the desired components on analytical TLC to an  $R_F$  of 0.35. If several compounds with similar  $R_F$  are to be separated, run the midpoint between the components to 0.35. If the compounds are widely separated, adjust the  $R_F$  of the less mobile component to 0.35.
2. Chose a column with appropriate diameter:

Column diam. (mm)	Vol. of eluent (mL)	Sample loading (mg) $\Delta R_F \geq 0.2$	Sample loading (mg) $\Delta R_F \geq 0.1$	Typical fraction size
10	100	100	40	5
20	200	400	160	10
30	400	900	360	20
40	600	1600	600	30
50	1000	2500	1000	50

3. Add glass wool and 3mm of sand if there is no frit on the column. 140 mm of silica is added on top, and the column is gently tapped vertically on the bench top to back the gel. Then add another 3mm of sand. Ensuring that the flow control is open on the bottom, carefully pour solvent to fill the column. Rapidly force solvent through dry silica gel. Make sure you keep the pressure on until all the air is flushed out; otherwise the silica will fragment and hinder separation. Flush solvent out to the top of the sand, and the sample is applied by pipette as a 20-25% solution in the eluent to the sand. Push all the sample into the silica gel, and the walls of the column are washed down with a few milliliters of fresh eluent, and the washing are pushed into the gel. Push the selective eluent at a rate of 51 mm/minute, and collect fractions until all solvent is used. If the foregoing instructions are followed exactly, there is little opportunity for the separation to fail.

If you wanted to reuse a column:

Column recycling is effected by first flushing (rate=51mm/minute) the column with approximately 130mm of the more polar component in the eluent (generally ethyl acetate or acetone) and then with 130 mm of the desired eluent. If the eluent is relatively non-polar (ie. Less than 10% EtOAc/light petroleum), it may be more advantageous to use a flushing solvent (ie. 20-50% EtOAc/light petroleum).

Source: Vogel's Textbook of Practical Organic Chemistry (5<sup>th</sup> ed.).