

SiO₂ Column Chromatography for amino acids with hydrophobic protection groups

- ◆ Suspend SiO₂ in petrol ether (PE)
- ◆ Load it onto a gravity column
- ◆ Let the PE flow through the column and tap the column several times to ensure there is no air left in the column (warning: don't let the column get dry)
- ◆ Dry the reaction mixture under reduced pressure to complete dryness
- ◆ Dissolve the reaction mixture in a highly polar solvent. We used ethyl acetate (EtOAc)
- ◆ Add 5 - 8 g SiO₂ beads so that you have a slurry suspension
- ◆ Dry under reduced pressure
- ◆ Put the dried SiO₂ loaded with the reaction mixture onto the column
- ◆ Load approx. 10 g NaSO₄ or MgSO₄ onto the column
- ◆ Flush the column one time with 100 % petrol ether (PE)
- ◆ For the next steps collect the eluent fraction by fraction
- ◆ Start the separation with 2 flushes with 5 % of the highly polar solvent in PE
- ◆ Increase the polarity of the eluent by 5 % steps
- ◆ While separation adjudge the fractions by TLC



Note: The flow rate can be increased by using a hand pump. Don't put too much pressure onto the column to ensure a good separation. It is recommended to collect the flow through dropwise. Depending on the amount of reaction mixture, the size of the column can vary.