

Tips on how to minimise product loss

You might feel like every step of the recrystallisation process is a cruel joke leaving you with less and less valuable product until you have about 1% yield and a sad face. But there are tricks for preventing this from happening!

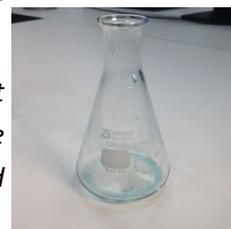
STEP 1: Dissolution of crude solid

⇒ Use the correct sized equipment for the amount of crude solid you start with.

Loss is proportional to the internal surface area of the glassware you use. If you only have a small amount of product and use large glassware then your loss will be proportionally larger than if small glassware had been used. This might mean that a normally insignificant loss becomes significant and impacts your yield!

⇒ Use only the minimum amount of HOT solvent to dissolve your crude product (you're aiming for a saturated solution at a high temperature).

The more solvent you add after this point the greater the proportion of your product that will remain dissolved in the solvent once cooled. This means LOST! Like wise, the further the solvent is from the boiling point the more you will need to add to reach a saturated solution.



Don't let these babies go to waste! Rinse and keep it HOT.

STEP 2: Hot filtration

⇒ Wet the filter paper first with hot solvent before pouring your solution.

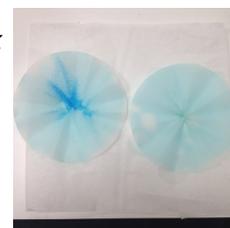
Not too much! Just enough to be absorbed by the filter paper and help keep it HOT and in place.

⇒ Use small amounts of HOT solvent to rinse our the flask, make sure no undissolved product is left.

Make sure the solvent is HOT. You are better off doing several rinses with small amounts of solvent to ensure good transfer then rushing in an using a large amount once and risk using too much.

⇒ Make sure your equipment is HOT!

It might seem obvious but premature crystallisation is preventable by maintaining warm equipment (this include the funnel) the whole time your solution is filtering. You might need to sit it all on a steam bath to keep it warm.



Crystals on filter paper means not HOT enough!

STEP 3: Crystallisation

⇒ Be patient! It takes time for the solution to cool down and maximum crystals to form.

Rushing this step is a sure way to throw the baby out with the bath water. Use the time to clean up and set up your vacuum filtration while your solution chills. The more time you allow for this step the more impressive your crystals will be!

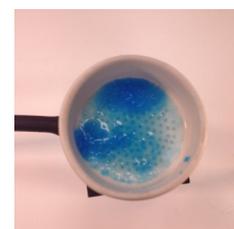
STEP 4: Vacuum Filtration

⇒ Choose the correct sized equipment.

See step one for why! This includes using the correct sized filter paper, so crystals don't get sucked through the funnel or trapped underneath.

⇒ Keep your rinses cool.

Rinsing is a great way to make sure all your product is transferred and residue is removed, but remember your product will dissolve in the solvent you rinse with if it's too warm. So make sure it's really chilled and use small amounts.



Your product will turn to a soggy mess if rinsing solvent isn't COLD enough.