Filtering under a nitrogen atmosphere

When a reaction has been conducted under an inert atmosphere such as nitrogen, it is quite likely that the product must also be handled under the same conditions. If that is not the case and the product is totally air-stable, then a normal filtration can be conducted. However, many stable solids are still air-sensitive when they are in solution. In this case, a special filtration process must be considered.

The type of filtration must be planned before the reaction is started, as the outcome could be affected by your choice of reaction vessel. If you will need to filter under nitrogen, it will be best to start with either a Schlenk tube or a 2-neck rb flask as your reaction vessel.

Once you are getting close to needing to filter, set up glassware as in Figure 1. Note that this would be a second Schlenk tube, if you used one as your reaction vessel. Once you have the joints greased, apply a vacuum and then let it down to a nitrogen atmosphere. Do this three times to thoroughly flush your glassware.

Arrange your reaction flask so that nitrogen is going directly into one of the necks. The other neck should be the same joint-size as the Viking cap. Leaving the nitrogen line attached to the Schlenk tube, remove the Viking cap and invert the whole set of glassware into the reaction flask. Use Keck clips and/or elastic bands to hold all the joints together.

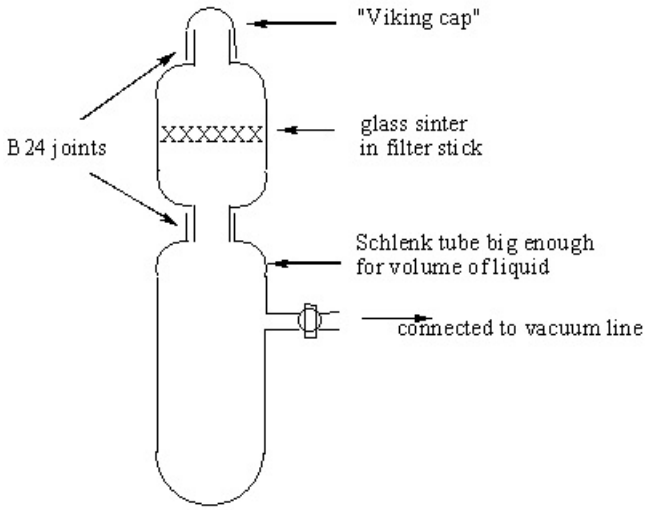

Figure 1: Preparing the receiving flask and filter stick under nitrogen.

Remembering to keep the two nitrogen lines uppermost, slowly tilt the joined apparatus through 180°. Allow the liquid to run freely through the sinter.

If the liquid flow stops before it has completely drained through the filter, close the lower tap to the nitrogen line, and then turn the tap on the vacuum line slowly through 270°. Then slowly turn the lower tap (the one on the Schlenk tube) through 180°. This should apply a gentle pressure

differential to re-start the flow. This is much more effective than applying a full vacuum as the sinter will often freeze and clog if the solvent starts to evaporate.

Once the filtration is complete, return the lower vessel to a nitrogen atmosphere. Remove the Keck clip and/or elastic bands on the lowest joint and separate.

If you want to retain the solid, cap the open end of the filter stick with the Viking cap and invert the assembly. Apply a vacuum to dry the solid. With luck, the dried solid will fall to the bottom of the reaction vessel.