Standard Operating Procedure: Wet Stills

Stills are used to remove excess water from solvents. A still is created by first selecting which solvent you wish to purify. Once the solvent is known you must select an appropriate drying agent which can be determined by checking the literature (i.e. *Preparation and Purification of Laboratory Chemicals*). The solvent and drying agent are then placed within the still and sealed. If sodium or potassium metal are used, mark the amount used on the label for the still (this information is necessary for quenching). A condenser and nitrogen flow are also attached. The still should be kept under a continuous nitrogen stream to avoid contamination of the solvent by air. Stills should always be set up in a fume hood.

When the solvent in the still is desired, the heat is turned on as well as the water flow through the condenser. The solvent is refluxed for approximately 30 minutes, then the collection valve is opened so that condensed solvent can begin to gather in the upper vessel. Once the desired amount of solvent has collected in the upper vessel, the solvent can be emptied into an appropriate container for use.

Following collection of the dry solvent, the operator should turn off the heat and allow the solvent to cool back to room temperature with the water still running. Once room temperature has been achieved once more, the water can be safely shut off. The nitrogen flow must remain on at all times.

If a solvent still is no longer needed, the drying agent must be quenched using a method specific for that drying agent. See the SOP for quenching solvent stills.

Personal Protective Equipment:

Safety goggles, safety gloves, lab coat

Waste Disposal:

Collected solvents should be disposed of within the appropriate solvent waste container.

Standard Operating Procedure: Quenching Solvent Stills

The quenching of used still-pots, especially ketyl pots, is potentially dangerous but can be done safely if appropriate precautions are taken. These include employing the proper personal protective equipment, working in a well ventilated fume hood behind a safety shield, and quenching the reactive compounds slowly.

Stills that used calcium hydride as the drying agent are the easiest to quench. After the majority of the solvent has been decanted away from the drying agent, the remainder, along with the calcium hydride, should be poured slowly over crushed ice. The ice must be replaced as it melts so that the unreacted calcium hydride is always being added to a solution that consists mostly of ice. Lumps stuck in the still-pot must be carefully removed with a spatula. When only but a thin film of hydride remains in the still pot it can be washed out with cold water. Submit the water/calcium hydride waste as calcium hydride quenched with water.

Ketyl pots require special care. First, one must be certain as to whether the still had contained sodium, potassium, of NaK. The process is similar for all three:

1. The entire quenching process should be carried out under a steady stream of nitrogen with a large opening to vent both the nitrogen stream and the hydrogen gas which is generated.
2. Pour off excess solvent, and refill the flask with dry xylene or toluene.
3. Add a reflux condenser and an addition funnel filled with sufficient dry tert-butyl alcohol to react with 150% of the expected amount of metal.
4. The alcohol is added dropwise, stopping if the solution begins to boil too vigorously.
5. After the addition is complete, the solution is heated to reflux overnight.
6. The process is repeated with isopropanol and then methanol.
7. If no bubbling is observed upon addition of methanol a small (1 mL) quantity of water is added confirm that all of the metal has been quenched. If the still only used sodium, the tert-butyl alcohol step may be omitted and the process may begin with isopropanol. The final mixture may be safely disposed of in a hazardous waste container marked as quenched sodium/potassium/NaK.

Personal Protective Equipment:

Safety glasses, labcoat, gloves.