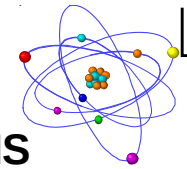
# LABORATORY SAFETY FACT SHEET #33



## QUENCHING SOLVENT DRYING-STILL BOTTOMS

The process of quenching solvent drying still bottoms is potentially dangerous. If not handled properly, fire or explosion can result. Below are commonly-used good practices for this process - the info was adapted from UCI. Fortunately, solvent stills are becoming rarer on campus as they are replaced by the safer drying column method ("Grubbs apparatus") which does not employ solvent heating, or water-reactive drying agents.

When a distillation flask becomes discolored and filled with semi-solid material, it is time to start over with new solvent and drying material (usually sodium or lithium metal or a metal hydride). However, one must first "quench" the old flask. The quenching procedure should be performed as soon as possible to avoid the possible production of peroxides in the solvents. It is not a good idea to let the drying agents sit for weeks in the fumehood for passive quenching, especially if the solvent is a peroxide former.

If you see that the drying agent is coated with tar, proceed very slowly and cautiously in successive addition of alcohols with manual agitation from time to time. The key thing to remember when quenching these sorts of things is to be very PATIENT. Sometimes, the reaction takes a few minutes to get going, so it is easy to add a whole bunch and then have the reaction get out of control. Often, it is safer to let the reaction go overnight with alcohol if killing a large still with tar-coated drying agents.

**Quenching Steps**

The quenching operation must always be performed in a properly operating fume hood. There must not be any other flammables or explosives stored in the fumehood at the time. Have the appropriate fire extinguisher ready and refresh your memory on how to use it. Use safety glasses or goggles, and a face shield if desired. Wear the type of glove, e.g., nitrile, butyl rubber, that is non-permeable to the solvent in question. An apron or lab coat is recommended. Never perform this process with no one else around.

Obtain a container of sufficient size to hold both the still round-bottom flask and enough ice water to effectively cool the flask. Next, decant the bulk of any remaining solvent into an appropriate labeled container. If the still was neglected and there is a ball of metal surrounded by tar, it would be wise to make sure that there is a high boiling point inert and relatively dry solvent (e.g. xylene) to keep the drying agent covered at all times and to act as a heat sink in case of sudden reaction. Place the flask into the ice water bucket; secure it with a clamp and ring stand if necessary to prevent it from falling over. You may want to use dry ice / acetone bath if solvent does not freeze at -78 degrees C to slow reaction. Keep the solution stirred either mechanically or by a spark-proof magnetic stirrer. Aim the mouth of the flask away from any people or equipment. If you are quenching a large volume use a blast shield.

Use a pipette to add a small aliquot of sec-butanol. Perform the entire quenching operation under argon or nitrogen gas. If gaseous bubbles appear, wait until they stop, then add another small aliquot of sec-butanol. Continue this cautious step-wise addition until the generation of gaseous bubbles becomes very slow.

After the sec-butanol, try adding an alcohol with more freely available protons, like n-butanol. Continue the same cautious, step- wise approach until the gas bubble generation slows considerably. Remember to stir or swirl the flask occasionally, always keeping the mouth of the flask pointed away from anyone. Once you've used n-butanol, try the same step-wise cautious addition with these solvents in sequence: isopropanol, ethanol, methanol and water.

**Be Very Careful with the addition of WATER!** Even after methanol has been added, the drying agent can still react violently with water, especially if there hasn't been sufficient mechanical stirring of the solution. Mechanical stirring is vital because water is most dense and immiscible. It will sink to bottom with remaining solid to react vigorously.

Once the reaction with water is complete, use a suitable acid solution to neutralize the basic solution you've created. Good choices include 3 M HCl and citric acid, which may be easier to use. Add the acid in aliquots with the goal of obtaining a pH of 7. Don't be obsessive about obtaining this exactly; in the 5 - 9 range is OK. Pour this solution into a properly labeled waste container. In order to properly label the waste container with the percentages, you must keep track of the approximate amounts of the various solvents you used in this quenching process.

**References:**


a) The Chemist's Companion by Gordon & Ford (lists drying agents with excellent comments about which solvents they work best with or should not be used at all)

b) Prudent Practices in the laboratory, National Research Council (describes procedures for decomposing metal hydrides, alkali metals etc.)


**Spill and Accident Procedures**

If one spills the unquenched flask, MOVE QUICKLY AWAY. The drying agent may spontaneously ignite in the air and the flammable solvent may cause a flash fire. Inform
everyone in the immediate area and have them move to safe location.

If the spill is large, call the EH&S spill response team and inform them of the condition. There are two likely occurrences, the flammable solvent will evaporate and the alkali metal or metal hydride will oxidize with the moisture in the air. Or the alkali metal or metal hydride will react vigorously with a proton source (like water) and will generate hydrogen gas, which may spontaneously ignite with the heat of the reaction. If this occurs, EXIT and CALL the FIRE DEPARTMENT, the entire area may be quickly engulfed in flames.

If the spill is small, and doesn't contain any alkali metal or metal hydride, treat it as a flammable materials spill and 'dike' it with absorbent spill cleanup material, cover the spill with the absorbent then, once the spill is absorbed, sweep it into a bag for waste disposal.

If the spill evaporates completely and leaves the slowly oxidizing alkali metal or metal hydride behind, gather these carefully into a beaker and quench with the same previously described procedure.