

Standard Operating Procedures for Dry Stills

Things to understand- House nitrogen is constantly passed through the dry still system. The positive pressure of nitrogen is what will carry the solvent from the primary container through the alumina-filled columns and into the collection bulb. The flow of the nitrogen is observed through the mineral oil bubbler. The nitrogen flow should be maintained at 1 bubble per second when solvent is not being taken from the dry still.

Retrieving Solvent

1. Equip your receiving flask with a dry-still adapter
2. Attach the dry-still adapter to the bottom joint of the collection bulb of the dry-still.
3. Open the stopcock on the dry still adapter and open the stop cock on the side arm of the collection bulb.
4. Let the system purge with nitrogen to ensure a dry, oxygen-free solvent transfer.
5. Increase the nitrogen flow rate to 5 bubbles per second by twisting the black knob connected to the desired solvent cylinder (top set of black knobs).
 - a. Make sure the oil does not spew out of the bubbler
6. Fill the collection bulb with desired amount of solvent by turning the swagelok at the top of the solvent cylinder from perpendicular to the cylinder to parallel. Close the swagelok once sufficient solvent has reached the collection bulb.
7. Open the stop-cock at the bottom of the collection bulb to let the solvent drain into the receiving flask, adjusting the nitrogen flow with the stopcock on the side arm of the dry still to allow air to escape the receiving flask while filling with solvent. Watch the mineral oil bubbler to ensure a positive pressure is maintained.
8. Once finished, close the stopcock directly below the collection bulb. Close stopcock on the side arm of the receiving flask. Close the stop cock on the dry-still adapter. Return the nitrogen flow rate to 1 bubble per second.
9. Remove your flask and log the amount of solvent taken on the dry still record sheet.

Disassembly of the Dry Still

1. Close the valve on the primary container, this will prevent solvent from flowing from the primary container into the alumina column.
2. Close the bottom valve on the alumina column.
3. Have a beaker ready and disconnect the line that runs from the primary container to the alumina column. Disconnect it from the primary solvent container side. The solvent in this line will be drained with the help of gravity.
4. If the dry still has two consecutive alumina columns: Now is the time to disconnect the line that connects them.
 - a. Make sure the top valve of the first column is closed and the bottom valve of the second column is closed.
 - b. Disconnect the line and drain the solvent into a beaker

5. The line that was connecting the primary solvent container to the first alumina column should still be connected to the alumina column and open on the other end. Place the open end of the line in a large beaker (expect about 1.5-2L from one column). Open the top valve followed by the bottom valve on the column and solvent will begin to drain. Wait for the entire column to empty out.
6. Now the majority of the solvent should be emptied from the column. Unscrew the hardware and take the column off the wall.
7. If working with two consecutive columns: Drain the second by transferring the line to the bottom of the second column. Make sure the line is in a large container and let gravity drain the solvent from the column.
8. To remove all of the solvent attach a hose adapter (obtained from Building Manager/Architect) to one end of the still. Attach the hose adapter to a nitrogen valve in a fume hood, open the still and blow nitrogen through overnight.

Reactivating the Alumina

Alumina should be reactivated 2-3 years following replacement, or if water is detected in the solvent after passing through the alumina.

1. Once the column is completely free of solvent, place the column in a heat mantle sleeve (obtained from Building Manager/Architect), while still passing nitrogen through the column, and connect it to a variac.
2. Leave the hose adapter connected but remove the valve at the end of the column. This valve has a Teflon septum in it and it will melt as hot nitrogen passes through it. Maintain a constant flow of nitrogen throughout the entire process.
3. Place a thermocouple underneath the column. Begin to slowly heat. Bring the temperature to 275°C. Do not exceed 275°C for reactivation. Maintain this temperature for 5 hours.
4. Turn off the variac. Open the heat mantle sleeve to let the column cool while still under nitrogen flow.
5. Once cooled, close the column maintaining a nitrogen atmosphere.
 - a. To do this, attach the valve at the end of the column. Close the valve at the end of the column and then once the column is filled with nitrogen close the valve at the other end of the column.
6. This column now contains reactivated alumina and is closed to prevent water and air from getting in. The column is now ready to use.

Replacing the Alumina

Alumina should be replaced 2-3 years after reactivation, or if water is detected in the solvent after regeneration of the alumina.

Once the column is completely solvent free remove one large screw at the top of the column. There is nickel tape (obtained from Building Manager/Architect) that is wrapped over the threads of the screw, make sure it is in good condition before reattaching. The large screw should also have glass wool in it.

Once removing the screw simply empty the beads out into a solid waste container. Refill with alumina. While refilling tap the sides of the column to pack the alumina beads so the maximum amount of alumina is added. Once filled reattach the screw.

Activating New Alumina

1. Once the column is completely free of solvent, place the column in a heat mantle sleeve (obtained from Building Manager/Architect), while still passing nitrogen through the column, and connect it to a variac (obtained from Building Manager/Architect).
2. Leave the hose adapter connected but remove the valve at the end of the column. This valve has a Teflon septum in it and it will melt as hot nitrogen passes through it. Maintain a constant flow of nitrogen throughout the entire process.
3. Place a thermocouple underneath the column (obtained from Building Manager/Architect). Begin to slowly heat. Bring the temperature to 300°C. Do not exceed 325°C for activation. Maintain this temperature for 6 hours.
4. Turn off the variac. Open the heat mantle sleeve to let the column cool.
5. Once cooled keep the column closed while under a nitrogen atmosphere.
 - a. To do this, attach the valve at the end of the column. Close the valve at the end of the column and then once the column is filled with nitrogen close the valve at the other end of the column.
6. This column has reactivated alumina and is closed so water from the air does not get in. The column is now ready to use.

Refilling Solvent

- The house nitrogen flow rate does not need to be adjusted for this procedure
 - Turn black valve on bottom of still canister 90 degrees to close.
 - Remove N₂ inlet valve from solvent container.
 - Close the colored valve on the solvent container by turning 90 degrees.
 - Connect the N₂ inlet valve to the port near the colored valve.
 - Place open end of degassing tube (hanging on the wall) into the back of a nearby hood.
 - Connect the valve to the original N₂ inlet port to release pressure built up in the canister.
 - When the pressure is fully released, unscrew the cap to the solvent container, just until the black plastic piece touches the threads on the top of the screw.
 - Turn the handle 45 degrees and remove the cap to the container.
 - Fill container with approximately 4 x 4L bottles of solvent (fill to the line).
 - Replace the cap to the container and screw the lid into place until hand tight, but make sure to complete the seal around the opening.
 - Slowly open the colored valve on the container to reopen to N₂ to degas the solvent.
 - 15 min. of N₂ flow, then 15 min. no N₂ flow, and finally 15 min. N₂ flow. A total of 45 min. to degas the solvent.

- Close the colored valve on the container and remove the N₂ inlet valve from the port.
- Remove the degassing tube, secure and store it back on hook.
- Reconnect the N₂ inlet valve to the proper (original) port. Should hear N₂ flow into the container.
- Have a beaker handy and unscrew the nut at the bottom of the still canister, (the black valve should still be closed from step 1). Hold the beaker under the still to catch any solvent and turn black valve 90 degrees (pointing down) to release any air in the short line from container to still canister. Turn back the black valve 90 degrees to close and screw back in the nut.
- Open the colored valve on the solvent container and point the black arrow on the valve at the bottom of the still canister upward. (Note: the black valve is a 3-way valve, if pointed downward or closed then no solvent will be able to reach the canisters).
- Before walking away, make sure that no solvent is coming out from around the seal of the cap of the solvent containers.

Personal Protection Equipment:

For all dry still use/maintenance: Lab Coat, Lab goggles, gloves.

Waste Disposal:

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