Ball Lab Standard Operating Procedure 1

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Introduction and Purpose: This SOP details the procedure and safety considerations for the withdrawing of solvents (THF, toluene, DCM, pentane and ethyl ether) from the solvent system. This can be used for reactions that require those 5 different types of solvent.

Kind of SOP: Technique

Last Revision: 01/07/2014

Uses: Standard organic chemistry reactions with those solvents under inert gas conditions.

General Procedure:

1. Prior to the actual solvent withdrawal, determine the volume of solvent needed and the physical location of the reacting flask within the hood. Obtain the appropriately-sized syringe that is able to withdraw the necessary amount. If the flask is located deep within the hood, obtain a long syringe needle from the oven. Otherwise, obtain a standard-length syringe needle from the oven. Note: always use the needles from the leftmost tray to ensure complete dryness. Attach the syringe needle to the syringe.
2. Locate the correct solvent to use and insert the needle into the small red septum of that solvent container. Try to use the same hole of insertion to avoid introducing too much leak to the system. Let the inert N_2 gas fill up the syringe chamber. Withdraw the needle and point it away from any lab members. Expel the inert gas by pushing down the syringe and pointing the needle tip away from any personnel.
3. Repeat step 2 for a total of 3 repetitions of withdrawing-expelling of nitrogen.
4. Insert the syringe deep into the solvent container again, into the solvent reservoir this time, while holding down the syringe handle to prevent any nitrogen from entering the syringe chamber. Release the handle to allow the solvent to enter the syringe. Gradually tilt the syringe until it is upside-down while the solvent is filling. Stop the filling of solvent by controlling the syringe handle until the solvent volume exceeds the necessary amount slightly.
5. Keeping the syringe upside-down, position the needle away from the reservoir and into the nitrogen above. Withdraw some nitrogen to create a bubble within the syringe chamber.
6. Keeping the syringe upside-down, remove the syringe needle from the solvent container and transfer it near to the reaction flask.
7. Insert the needle into the reaction flask via the septum. Carefully expel the nitrogen until the first drop of the solvent appears.
8. With volume-difference liquid transfer, carefully add the solvent and rinse down the side of the reaction flask. Use the markings on the needle to determine the volume added.
9. Once sufficient solvent is added, withdraw some inert gas from the reaction flask by pulling the syringe handle.
10. Keeping the syringe upside-down, remove needle.
11. Expel any remaining solvent into the waste container.
12. Rinse the needle and syringe with acetone by withdrawing up the needle. Discard the syringe in the solid waste container and place the needle in the rightmost tray in the oven to dry.

Safety Considerations and Precautions:

- Always ensure minimal pointing of syringe needle towards any lab personnel
- Keep the syringe inverted while holding the solvent to prevent spilling