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| **Brueckner Lab-Specific Standard Operating Procedure (LSOP):**  **Flame Drying and Activation of Molecular Sieves** | |
| **Principal Investigator(PI):** Christian Brueckner | |
| **Building:** Chemistry | **Lab(s) Covered by LSOP:** R413/R415 |
| **Department:** Chemistry | **Lab Phone Number(s):** 6-6596/6-6598 |
| **SECTION 1 – PROCESS and HAZARDS INVOLVED** | |
| Flame drying and activation of molecular sieves involves the heating of glassware (with or without molecular sieves) using an open flame of a high-temperature het gun under high vacuum  Hazards: Open flame, hot glass wares, glassware under vacuum | |
| **SECTION 2 – ADMINISTRATIVE CONTROLS** | |
| * Anyone using the chemicals and procedures described herein needs to have undergone the annual EH&S [Chemical Hygiene Training](http://www.ehs.uconn.edu/Chemical/?p=training): * Be aware of the applicable safety data sheets (SDS) if molecular sieves are involved: <http://www.msds.com> * [Working Alone](http://policy.uconn.edu/2012/07/30/working-alone-policy/) is not permitted when using chemicals or processes described in this LSOP | |
| **SECTION 3 – ENGINEERING CONTROLS** | |
| Use adequate general or local exhaust ventilation to keep heat from building up | |
| **SECTION 4 – WORK PRACTICES** | |
| * Preferably perform the flame drying procedures inside a fume hood * Like all experiments involving high vacuum, flame drying procedures should also be performed during normal business hours (i.e., 8:00 am-5:00 pm Mon-Fri), if possible * Keep away from sources of ignition and combustible materials and remove any excess clutter and any flammable solvents that are not needed for the process * Fill the liquid nitrogen reservior prior to use of the oil pumps of the double manifold * Follow the step-by-step procedures outlined below | |
| **SECTION 5 – PERSONAL PROTECTIVE EQUIPMENT (PPE)** | |
| * Flame/heat resistant gloves must be worn while handling – we keep sets of leather work gloves in the bottom of the tool cabinet | |
| **SECTION 6 – STORAGE** | |
| * Molecular sieves should be stored in the oven | |
| **SECTION 7 – SPILL AND ACCIDENT PROCEDURES** | |
| * If breakage of the glassware occurs, let cool and sweep up, including the molecular sieves.   **Report any incident to the PI and fill out the** [**accident form**](https://www.google.com/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0ahUKEwiF3bPe1dPXAhVRRN8KHX4wDf4QFggmMAA&url=https%3A%2F%2Fchemistry.uconn.edu%2Fwp-content%2Fuploads%2Fsites%2F1259%2F2015%2F09%2FIncident-Report-Form.doc&usg=AOvVaw3Uov8IQ2Z-Kan) | |
| **SECTION 8 – FIRST AID PROCEDURES** | |
| * Treat any minor skin burns caused by contact with the propane torch, hot glassware, or burning rubber, cork, or cloth by washing under cool water, application of burn ointment, antibiotic ointment, and a bandage if necessary (see our first aid kit in the storage room between R413 and R415). * For major skin burns, immediately move to safety shower or other water source and begin rinsing affected area(s). Accompanied by another person, see the infirmary. * In case of massive burns, have another person from the lab dial 911and specifically mention the type of exposure. * Same for cuts stemming from breaking glassware: Treat any minor cuts with an antibiotic ointment and a bandage (see our first aid kit in the storage room between R413 and R415). * For major cuts, bandage and see the infirmary; go only accompanied by another person. * In case of severe cuts, have another person from the lab dial 911and specifically mention the type of injury.   **Report any incident to the PI and fill out the** [**accident form**](https://www.google.com/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0ahUKEwiF3bPe1dPXAhVRRN8KHX4wDf4QFggmMAA&url=https%3A%2F%2Fchemistry.uconn.edu%2Fwp-content%2Fuploads%2Fsites%2F1259%2F2015%2F09%2FIncident-Report-Form.doc&usg=AOvVaw3Uov8IQ2Z-Kan) | |
| **SECTION 9 – WASTE MANAGEMENT** | |
| N/A | |
| **SECTION 10 – DECONTAMINATION PROCEDURES** | |
| N/A | |
| **SECTION 11 – SPECIFIC PROCEDURE** | |
| **Molecular Sieve Activation:**  Molecular sieves must be activated (dried) before use. To check if molecular sieves are dry, you may put a bit in the palm of your hand and add a touch of water. If they generate a good amount of heat, they are dry. Otherwise, follow the procedure below:  Add molecular sieves to a dry flask and heat to 120 °C with an oil bath under high vacuum overnight (no stirring needed) using a flow control adapter (T-joint). Refill with argon or dry nitrogen, and use as needed (remember to flush flask out by vacuum-nitrogen-vacuum-nitrogen after opening each time to keep water out). Check sieves periodically for heat generation and dry as needed.  **Glassware Flame drying - Procedure for less sensitive materials:**  High vacuum will remove the majority of the air and moisture, but it will leave behind the moisture (hydroxyl groups) and air absorbed on the glass itself. In order to remove them, the glassware has to be heated with a heat gun (or Bunsen burner) while purging it (make sure that you remove all flammable materials from the area to prevent fires). The heat guns in the laboratory are industrial scale heat guns. They allow temperatures up to 500 °C. Upon completion of the heating; the filament inside the heat gun has to cool down to prevent it from melting. The heat gun should be placed away from any flammables even after the nozzle cooled down a little.  **Glassware Flame drying - Procedure for highly sensitive materials**   1. Prepare the clean glassware that is to be flame dried by placing a dry magnetic stir bar in the flask (if necessary for a reaction or distillation). Dry the flask above 130 °C in an oven overnight. Once out of the oven, seal the flask with a rubber septum. Completely fold the rubber septum over the neck of the flask. If removing hot glassware from the oven, handle the glassware with hot gloves before attempting to attach the septum, and complete steps 4-8 while the glassware is hot. 2. Open the main valve on the inert gas tank that serves the fume hood and double manifold to be used. 3. Slowly open valve to introduce gas flow to the double manifold. Bubbles emanating from the bubbler near the manifold indicate that a positive pressure of inert gas is present. For flame drying a small (less than 250 mL) flask, a high flow rate of about 3-5 bubbles per second is desired. A higher bubbling rate (5-8 per second) is desired when flame drying larger flasks. 4. Puncture the septum with an unobstructed needle attached to one of the Tygon tubes connected to the double manifold. 5. Turn the stopcock valve on the double manifold connected to your tubing so that the blue glass tip points up to introduce gas flow through the tubing. 6. Puncture the septum with a second unobstructed, disposable needle ("the vent needle"). Use a hole that is already present in the septum if possible. 7. Ensure that a positive pressure of inert gas is emanating from the vent needle by holding the vent needle up to a sensitive area of the skin such as the moistened underside of the wrist or the lips. 8. Turn the three-way valve connecting the manifold to the bubbler so that inert gas no longer flows to the bubbler. Bubbling should cease. 9. Ignite the propane torch and set it in the fume hood. Ensure that no flammable solvents or combustible materials are nearby, and that the lit torch is not pointed toward you or anyone else. 10. Pick up the piece of glassware by the septum or the needle caps and hold the bottom of the glassware at an angle in the hottest part of the torch flame. 11. Initially, moisture will appear on the inside wall of the glassware. Rotate the glassware and move it through the flame until no more moisture appears on the inside. Do not put the septum in the flame or it will melt/burn. Use a cork ring to brace against the bottom of the glassware to aid in control of the glassware while moving it along the flame. Do not put the cork ring in the flame or it will catch fire. 12. Once all the visible moisture appears to be gone, keep heating the flask until the gas emanating from the vent needle is warm. One to five minutes of heating may be required depending on the glassware size. 13. Place the glassware on a cork ring in the fume hood to cool to room temperature under positive inert gas pressure. Cooling may take between five to fifteen minutes. The higher initial flow of inert gas prevents air from rushing into the flask as it cools because of the decrease in temperature. If additional glassware needs to be flame dried, repeat steps 3-7 and 10-13. Then continue to step 14. 14. After placing the hot glassware on the cork ring, turn off the propane torch and store it properly. 15. When the glassware has cooled to room temperature, quickly remove the vent needle and then immediately turn the three-way valve to resume inert gas flow to the bubbler. Failure to adjust the three way valve quickly will cause pressure to build up in the manifold. Excessive gas pressure from a manifold is most commonly alleviated by popping the septum off of the glassware and/or forcibly ejecting one of the glass stopcocks toward the user. This is to be avoided. Store the vent needle in a plastic cover for future use. If the vent needle is or becomes contaminated or obstructed, dispose of it in the sharps waste and use a new disposable needle. 16. Adjust valve A to decrease the rate of inert gas flow to one bubble every 3 to 5 seconds if continued inert gas use is needed. If inert gas is no longer needed, close this valve to finger tightness. 17. The piece of glassware is now ready for use.   See also: "The Chemist's Companion: A Handbook of Practical Data, Techniques, and References" A. J. Gordon and R. A. Ford, John Wiley and Sons, New York, 1972 – book is in the library I R415) | |

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| **SECTION 12A. APPROVAL** | | | |
| I have reviewed, understood and agreed to follow this lab-specific standard operating procedure (LSOP) for flame drying/molecular sieve activation*.* Failure to follow this LSOP or lab-specific training guidelines is a violation of the [*University Health & Safety Policy*](http://policy.uconn.edu/2011/05/19/health-and-safety-policy/) and [*University Code of Conduct*](http://policy.uconn.edu/2011/05/17/employee-code-of-conduct/).  Further approval and/or review of this LSOP by the PI/Supervisor is required if any of the following events occur:   * A significant change in amount (i.e., doubling of the scale of reaction) or substitution of the chemicals in the procedure is planned * A major change in the agreed-upon experimental set-up is planned (heating instead of room temp, etc.) * Any signs of a failure in safety design or equipment are observed * Any signs or symptoms of a chemical exposure to any personnel are observed * Unexpected and/or potentially dangerous experimental results occur (e.g., fire, uncontrolled buildup of heat and/or pressure, etc.) | | | |
| **Researcher Name/Signature** | **Trainer Name/Signature** | **Training Date** | |
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| **SECTION 12B. PRINCIPAL INVESTIGATOR CERTIFICATION** | | | |
| I approve the contents of the lab-specific standard operating procedure listed above. | | | |
| **PI Signature:** | | | **Date:** |
| **A HARD OR ELECTRONIC COPY (https://bruckner.research.uconn.edu/safety-resources/) OF EACH LAB-SPECIFIC STANDARD OPERATING PROCEDURE MUST BE READILY AVAILBALE IN THE LAB.** | | | |