Section 5.10 Title: Cannula Transfers **Revision Date:** 11/01/19
Prepared By: Mike Aristov and Michael Roy **P.I.:** Prof. John F. Berry

Prior Approval: This procedure is NOT considered hazardous enough that prior approval is needed from the Principal Investigator.

Involves Use of Particularly Hazardous Substance (PHS)? No
___ Carcinogen ___ Reproductive Toxin ___ High Acute Toxicity
Does this procedure require medical surveillance? No
Does this require use of a fit-tested respirator? No

Brief Description of Procedure:
The use of a cannula to transfer a liquid or suspension between two air-free flasks.

Location: *List the locations (buildings/rooms) where this procedure may be performed. For use of a PHS indicate a more precise location within the room, if appropriate, as a designated area.*
Daniels Chemistry - All Berry group labs

Chemicals Involved:

Chemical	Physical or Health Hazard (e.g. carcinogen, corrosive)
Organic solvents	Consult relevant SDSs for more details

Other Hazards: *Include hazards, other than chemical, that may be present during operation of the procedure.*
Sharps (cannulas and needles)

Exposure Controls: *(Check all that apply)*
PPE: _X_ Safety Glasses ___ Face Shield ___Chemical Splash Goggles
___Chemical Apron _X_ Gloves (Nitrile) _X_ Lab Coat
___Respirator (type) ___Other:

Engineering Controls:
_X_ Fume Hood ___Biosafety Cabinet ___ Glove box
___ Vented gas cabinet ___Other:

Administrative Controls: *List any specific work practices needed to perform this procedure (e.g., cannot be performed alone, must notify other staff members before beginning, etc.).*

N/A

Task Hazard Control Table: *For procedures involving numerous steps, it may be convenient to indicate specific requirements for individual tasks in the table below:*

N/A

Waste Disposal: *Describe any chemical waste generated and the disposal method used.*

Dispose of the reagents involved as appropriate. Consult SDSs for more details.

Accidental Spills: *Describe the procedure for handling small chemical spills that may occur during this procedure. Note that for large spills it may be appropriate to call 911.*

Small spills may be cleaned with an absorbing material. The material should be placed in a fume hood to dry after the spill has been cleaned.

Decontamination Procedures (required for PHS use): *Describe the procedure for decontamination of personnel and equipment.*

N/A

Training: *Describe any training needed prior to performing this procedure. Include training performed in-lab and any required demonstrations of competency.*

Training on needles and syringes is required before performing cannula transfers. No further formal training or documentation is required. New lab members should consult with senior members before handling cannulas and may wish for informal training or supervision.

Principle Investigator Approval: I have reviewed this procedure and approved it for use. Note: Modifications to the procedure may require update to this form.

Name: John F. Berry Signature: _____ Date: _____

Cannula Transfers

Overview: A cannula transfer is a method to transfer solutions between two air-free flasks without exposing either to air. There are three main ways to perform a cannula transfer – positive pressure, negative pressure, and siphon. Of these techniques, siphon is the slowest but safest, positive pressure is medium speed but risks bursting the septum off the flask, and negative pressure is the fastest but risks drawing in air to the flask through the porous septa.

Technique

General Starting procedure:

1) Set up your desired two flask system and identify the flask containing the solution to dispense (hereafter known as the starting flask), and the flask/frit/round bottom where you want the solution to be received (hereafter known as the receiving flask).
2) Position the two flasks such that the receiving flask is lower or at least level with the starting flask.
    a. Potential energy plays a large roll in all cannula transfers. Even with high positive pressure, if the solvent is viscous or dense (like water), then you will not be able to start the transfer if there is too great a difference in height between the two flasks or if the cannula arches too high.
3) Check to ensure all flasks are securely clamped.
4) Place the starting and receiving flasks under a high positive pressure and put a rubber septum on each flask
    a. Be sure to 'burp' the septa, as air can get caught in the cavity of the septum.
5) Select an appropriate cannula from the oven. Make sure the cannula is hot and work quickly to avoid condensation on the metal.
    a. If you are performing a cannula transfer on a suspension, use a thick gauge cannula to avoid clogging the linkage.
    b. For transferring solvent, a medium gauge cannula is plenty.
    c. If you are trying to layer solvent, use a small gauge cannula to avoid transferring solvent too quickly and thus destroying the layer.
6) Grip the flask to hold it in place and carefully pierce the cannula into the starting flask. Be very careful to not press the cannula in so far that it becomes submerged into your solution, as this will cause you to start spraying your solvent/reaction onto the fume hood.
    a. If the flask is under sufficient pressure, you should be able to hear the flow of gas through the cannula.
    b. Under no circumstances should you ever point the cannula at your ear or face to hear this sound. A needle blowing gas can be just as deadly, so treat it with respect and direct it away from the body.
    c. If working under a lower pressure and you are concerned about the flow of gas, place the tip of the cannula close to a liquid and check for ripples in the surface.
7) At this step, you can begin either positive, negative, or siphon transfer, and all three techniques are described below.

Positive pressure transfer

1) Insert the cannula into the receiving flask.

2) Place a bleed needle of the same gauge or smaller than the cannula into the receiving flask. Close any other inlets to the flask.
3) Submerge the needle into the solution of the starting flask, and the cannula transfer should start within seconds.
    a. If the transfer does not start immediately, try lowering the receiving flask/raising the starting flask.
    b. If it still doesn't transfer, see troubleshooting section
4) Transfer the desired amount of solution/solvent and remove the tip of the cannula from the solvent/solution so that the transfer stops. Do not remove the cannula at this point.
    c. It will take a few seconds for the transfer to stop, as the solution in the canula will still need to be pushed out.
5) If using a Schlenk flask or other glassware with a nitrogen inlet, open that inlet.
6) Remove the bleed needle from the receiving flask, and then remove the cannula from the receiving flask.
7) Remove the cannula from the starting flask and place it out of the way.
8) Replace rubber septa with greased glass stoppers to prevent air leaking into the flasks as needed.
9) Burp any stoppers which are replaced throughout the producer, as these can introduce small pockets of air into the reaction otherwise.
10) Once the flasks are stabilized and sealed, clean the cannula. Do not leave a dirty cannula overnight.

Siphon Transfer

1) Insert the cannula into the receiving flask and raise the starting flask as high as you reasonably can relative to the receiving flask.
2) Close any inlets on the receiving flask while leaving the starting flask open to the line.
3) Press the cannula bellow the solvent level in the starting flask.
    a. At this point **no** solvent should transfer.
4) Like in the positive pressure transfer, insert a bleed needle that is the same gauge or smaller than the cannula into the receiving flask until you see solvent start to flow through the cannula.
5) Once solvent starts to transfer, simultaneously remove the bleed needle and open the receiving flask to the line.
    a. The transfer of solvent will slow, but not stop.
    b. You can control the transfer speed by raising and lower the starting flask.
    c. If the transfer stops, then you will need to restart the solvent flow by repeating steps 2 through 5.
6) Once the desired amount of solvent has been transferred remove the tip of the cannula from the solvent/solution so that the transfer stops. Do not remove the cannula at this point.
    a. It will take a few seconds for the transfer to stop, as the solution in the canula will still need to be pushed out.
7) Remove the cannula from the receiving flask, and then from the starting flask, placing it to the side.
8) Replace rubber septa with greased glass stoppers to prevent air leaking into the flasks as needed.

9) Once the flasks are stabilized and sealed, clean the cannula. Do not leave a dirty cannula overnight.

Negative pressure

1) Insert the cannula into the receiving flask.
2) Lower the cannula into the solution in the starting flask.
3) Have the starting flask under a dynamic flow of nitrogen, and then gently apply vacuum to the receiving flask.
    a. The transfer should start quickly.
    b. Not much vacuum is required to draw solution over, so only brief application of vacuum is sufficient to transfer most solutions.
    c. Too much vacuum can cause your solution to bump, evaporate, and freeze inside of the cannula.
4) Monitor the receiving flask and continue to apply brief periods of vacuum until the desired amount of solution is transferred.
5) One the desired quantity of solution has been transferred, open the receiving flask to dynamic nitrogen, and the cannula transfer should stop quickly.
6) Replace rubber septa with greased glass stoppers to prevent air leaking into the flasks as needed.
7) Once the flasks are stabilized and sealed, clean the cannula. Do not leave a dirty cannula overnight.

Cleaning Cannulas

Cannulas come in contact with four main types of solutions; pure solvents, metal-containing solutions, organic solutions, and suspensions. The general procedure for cleaning a cannula involves drawing solvent through the cannula to flush out any contaminant. Dirty cannula can ruin a reaction, and rust can cause a cannula to become clogged, leading to a potential pressure build-up.

1) Start by placing a rubber septum on an Erlenmeyer with side arm connected to a water aspirator, then pierce the cannula into a rubber septum.
    a. If the cannula only transferred pure solvent, wash with acetone.
    b. If the solution transferred contained metal, rinse with acetone, then water, then 1 M HCl solution, then water, then acetone. The last rinses with water and acetone should be clear. If these rinses are not clear, repeat the procedure.
    c. If the solution transferred contained only organic ligand, rinse with the solvent that the ligand was dissolved in, then acetone.
    d. If the solution transferred was a suspension, rinse with water, acetone, and hexanes.
2) Wipe the outside of the cannula with a lab wipe wetted with acetone to remove residue. Allow the cannula to fully dry before putting it in the oven.