INORGANIC CHEMISTRY Laboratory

Chemistry 5192

General Laboratory Equipment Setup Descriptions

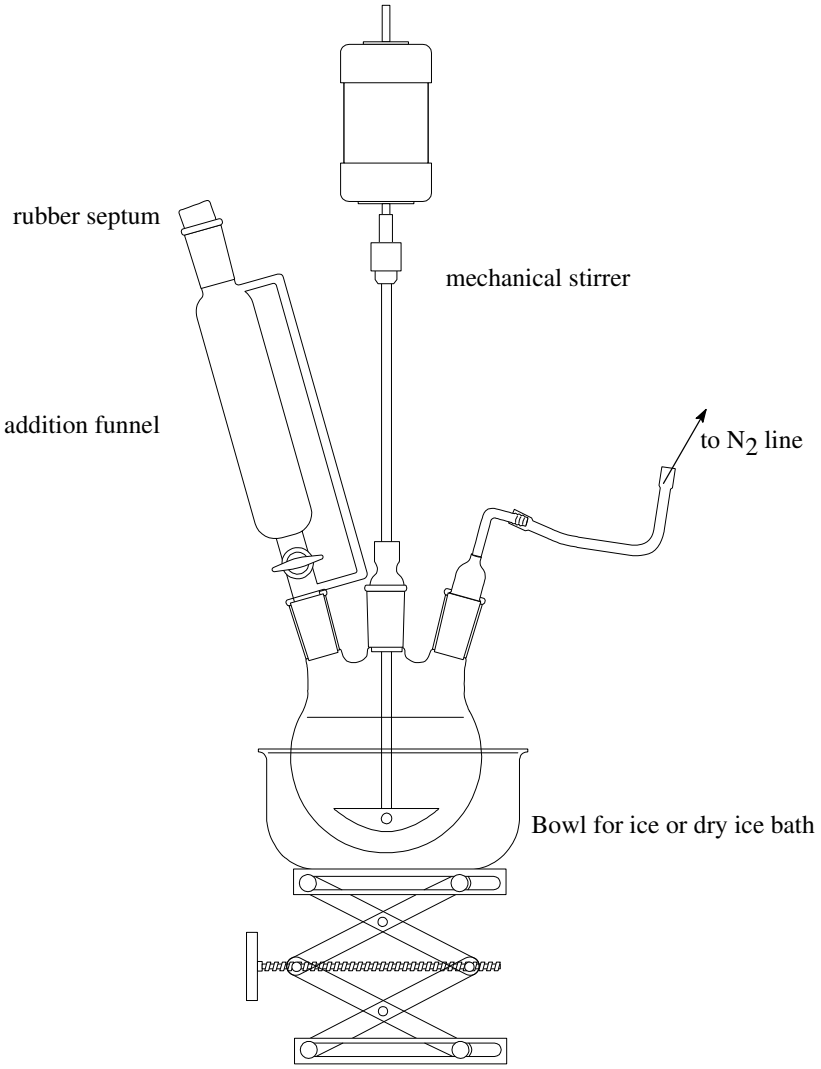
Basic Reaction Setup

Equipment

Reaction flask
Stir bar or mechanical stirrer (paddle)
Magnetic stir motor or mechanical stir motor
Glass gas inlet
Addition funnel
Rubber septum
N_2 inlet/mercury bubbler

Procedure:

Set up as shown in the figure below. (Variations include using a magnetic stirrer, two-necked flasks, etc.) Flush reaction flask and addition funnel with N_2. Add reagent and solvent to reaction flask. Chill or warm as appropriate. Syringe, pour, or cannulate reagent into the addition funnel. If more than one reagent is to be added to the reaction mixture, wash the funnel with solvent between additions. When the last reagent has been added, replace the addition funnel with a glass stopper.



Filtering in an Airless System

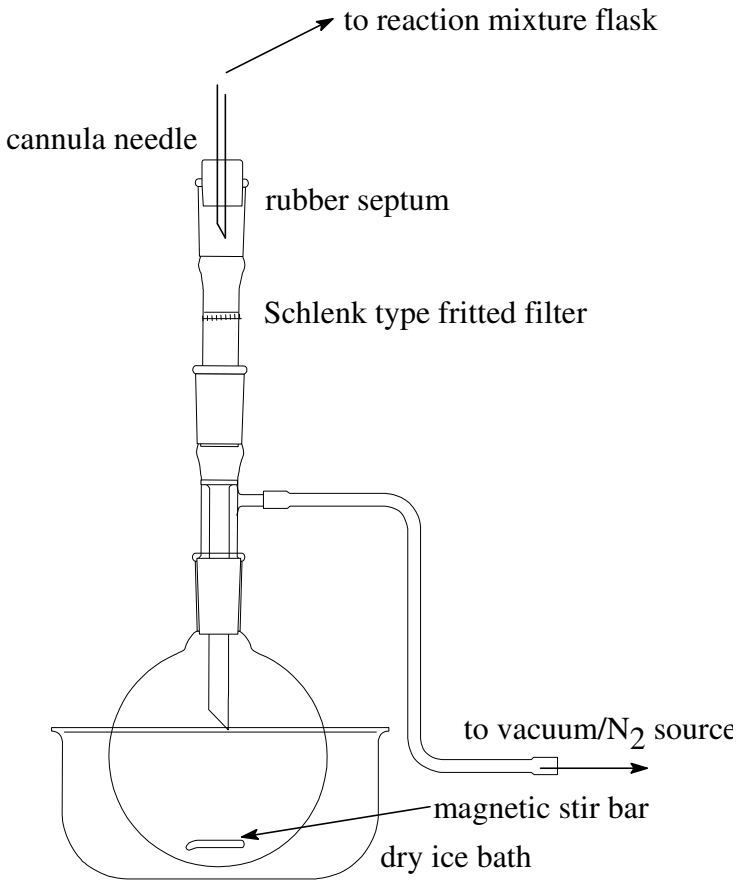
Purpose: To remove unwanted solids from a reaction mixture.

Equipment:

Flask containing reaction mixture with solids
Fritted filter with ground glass joints
Adapter with hose adapter to connect to N_2/vacuum line
Receiving flask with stir bar
Dry ice/acetone bath (-78 °C)
Rubber septum

Procedure:

Set up system as shown in figure. Connect to the vacuum system and evacuate for ca. 5 min. Then fill with N_2. Cool the receiving flask (dry ice bath) and transfer the mixture to the frit using the cannulation procedures described in a later section of this manual. Use only a slight vacuum in the receiving flask to prevent clogging the filter.



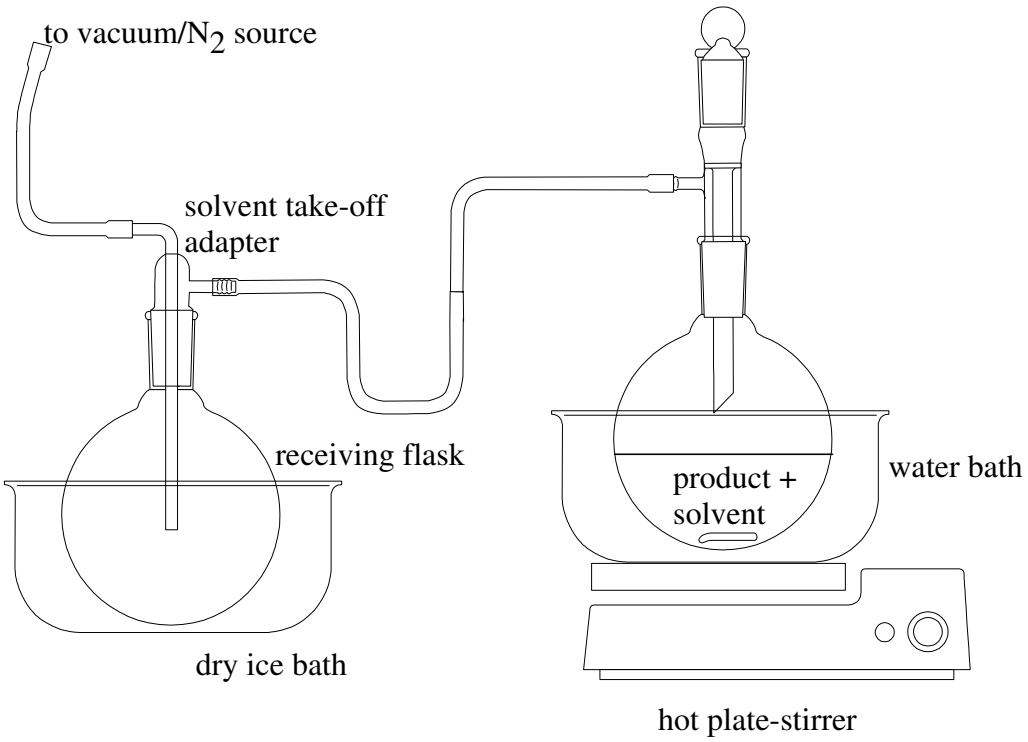
Airless Solvent Removal

Equipment:

Dry ice/acetone bath
Receiving flask
Flask with solvent and product
Glass gas outlet
Solvent take-off adapter
Vacuum tubing (ca. 12 inches)
Vacuum/N_2 source
Magnetic stirrer/stir bar
Heater/warm water bath

Procedure:

Purge the receiving flask, solvent take-off adapter, and vacuum tubing with N_2. Set up system as shown in figure. While stirring, pull vacuum such that the mixture boils gently. Do not pump continuously or solvents such as ether will escape the -78 °C bath and plug the muck trap on the vacuum manifold. Heat water slightly -- just enough to offset heat loss from solvent evaporation. When the solvent is nearly gone, large bubbles will appear. It may be necessary to place the solvent/reagent mixture in a smaller flask to effectively remove solvent. Transfer product to a small flask for distillation. See "Flask to Flask Transfer" procedure.



Flask to Flask Transfer

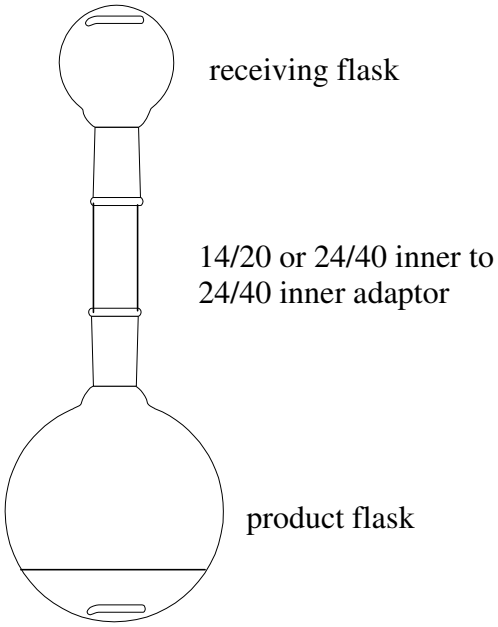
Purpose: Airless flask to flask transfer

Equipment:

Flask with product
Receiving flask
Transfer adapter(s)
Large stir bar if needed
Vacuum adapter if needed

Procedure:

Purge the receiving flask and transfer adapter(s) with N_2. Assemble equipment as illustrated with the product flask on the bottom. Rubber band of clip system together securely. Lift system and gently turn it over allowing product to run into the clean flask. If a stir bar is in the product flask, hold a large stir bar on the exterior of the adapter to prevent bar from falling into the adapter mouth. Clamp system securely if it is to be left standing. If a solvent is present, it may be necessary to use a vacuum adapter attached to a mercury bubbler between the adapter and receiving flask to release pressure buildup that occurs when solvent is shaken (e.g., Et2O).



Vacuum Distillation

Purpose: To purify products that have boiling points that are too low to conveniently condense at room temperature.

Equipment:

Distillation head
Distribution adapter (cow)
Receiving flasks and glass stopper (s)
Dummy flask
Flask with product
Thermometer with ground glass joint
Thermometer adapter if needed
Vacuum line
Hose/vacuum line adapter
Vacuum tubing (2 to 3 feet)
Heating mantle of appropriate size
Variac (Variable transformer)
Stir bar
Magnetic stirrer
Glass Wool
Amber tubing (2 lengths) for water in and out of condenser
Tubing and flask clips

Procedure:

Assemble distillation apparatus as shown in figure on next page. Begin by clamping the large piece (i.e. column and condenser) to the rack. Then connect condenser hoses with clips and then the vacuum hose. Add the thermometer and attach dummy flask with a rubber band or flask clip. Assemble the cow with pre-weighed receiving flasks well-attached with rubber bands or clips. Attach cow with flasks to distillation apparatus and evacuate system to hold cow in place. Further secure cow with rubber bands and/or clips.

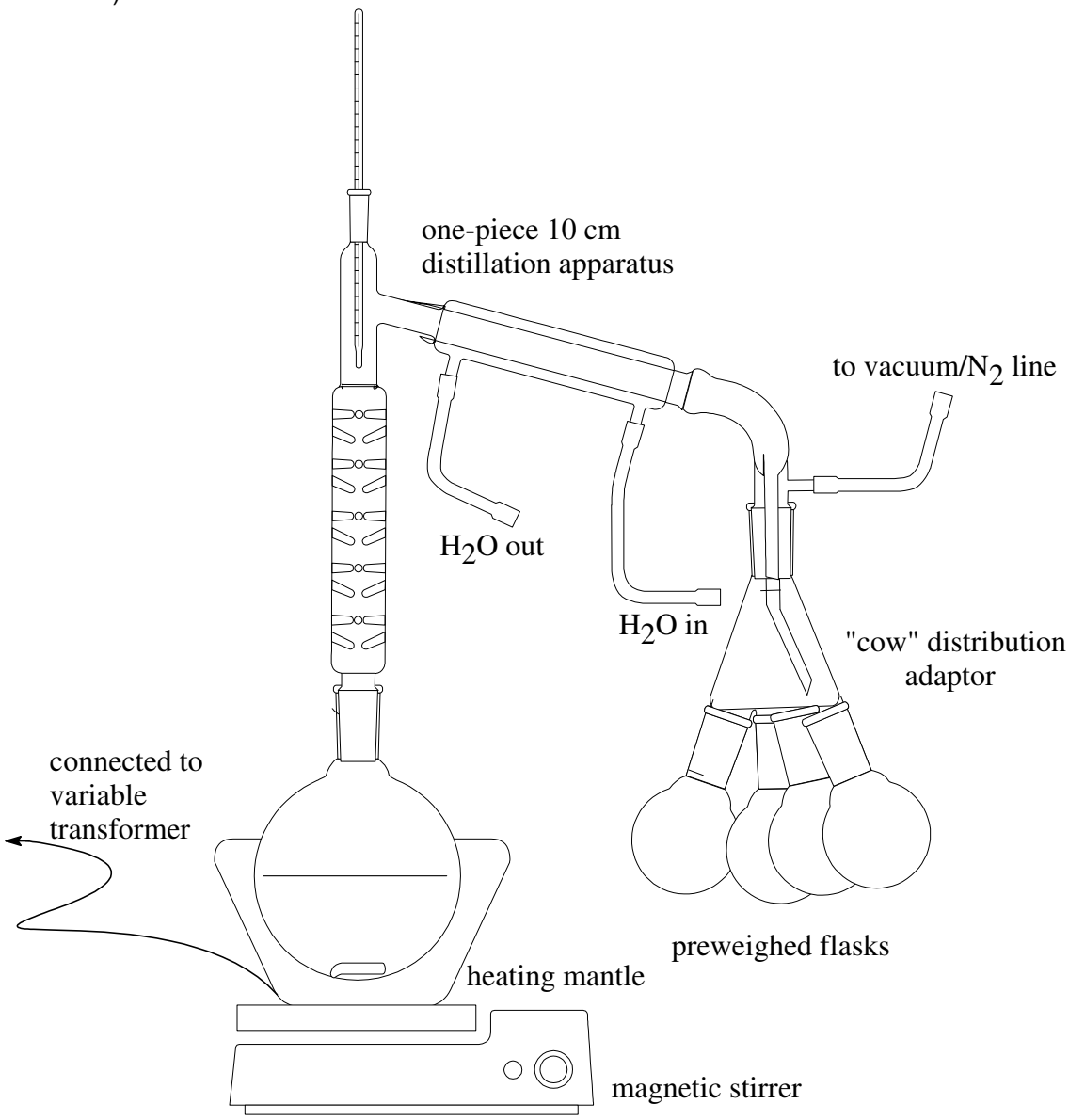
Evacuate the system for 5 to 10 minutes. Check the vacuum to see if it is as good as it was before attaching this apparatus. Then slowly fill the vacuum manifold and distillation apparatus with N_2, monitoring pressure with the manometer. Replace the dummy flask with the flask containing the product to be distilled and a stir bar. Partially evacuate the system (ca. 700 Torr) to hold it together. Place heating mantle and stirrer beneath distillation flask and turn on magnetic stirrer. Also turn on water to condenser. Close stopcock between vac line and distillation apparatus. Evacuate line.

Distillation at Lowest Pressure: Carefully open stopcock between vac line and distillation apparatus to de-gas material and to remove last traces of solvent. Be very careful in this step to avoid foaming or pumping of impure product into the clean condenser and flasks. When foaming has stopped, open system completely and then begin heating. [Never heat a closed system!! This system is closed until it is completely opened to the vacuum manifold which has the Hg manometer/pressure release opened.] Monitor pressure with the McLeod gauge. Collect the first few drops in a small flask. When constant temperature is reached, turn the cow in order to collect product in a tared flask. Record boiling point range and pressure.

Distillation at Higher Pressure. [This should be used with very volatile compounds which have boiling points near or below room temperature at the pressure of the

vacuum system.] With the stopcock to the distillation apparatus closed, slowly bleed nitrogen into vacuum system while keeping vacuum source valve opened. Monitor the pressure with McLeod gauge. If incorrect, re-adjust N_2 flow rate. (Never change the N_2 flow while mercury is in the column of the McLeod gauge. This could force Hg through the gauge causing it to crack.) When the desired pressure is obtained, slowly open stopcock to the distillation apparatus, being careful to avoid bumping and foaming. When foaming stops, begin heating. Continue as described for low pressure distillation.

General: Usually the b.p. will drop after one fraction has distilled and will rise again as another product begins to distill. Change flasks during the temperature drop. You may need to wrap the column with glass wool to speed up the distillation. A variac setting of ca. 30 is usually sufficient for most distillations. When distillation is complete, remove the heating mantle and let the system cool. Do not open the system while it is hot because compounds are more likely to ignite or decompose at high temperatures. Then slowly fill with N_2 and immediately remove collection flasks. Disassemble apparatus immediately before ground joints freeze. (i.e., today not tomorrow).



Cannula Transfer

Purpose: To transfer a large quantity of an air or moisture sensitive liquid in an airless environment

Equipment:

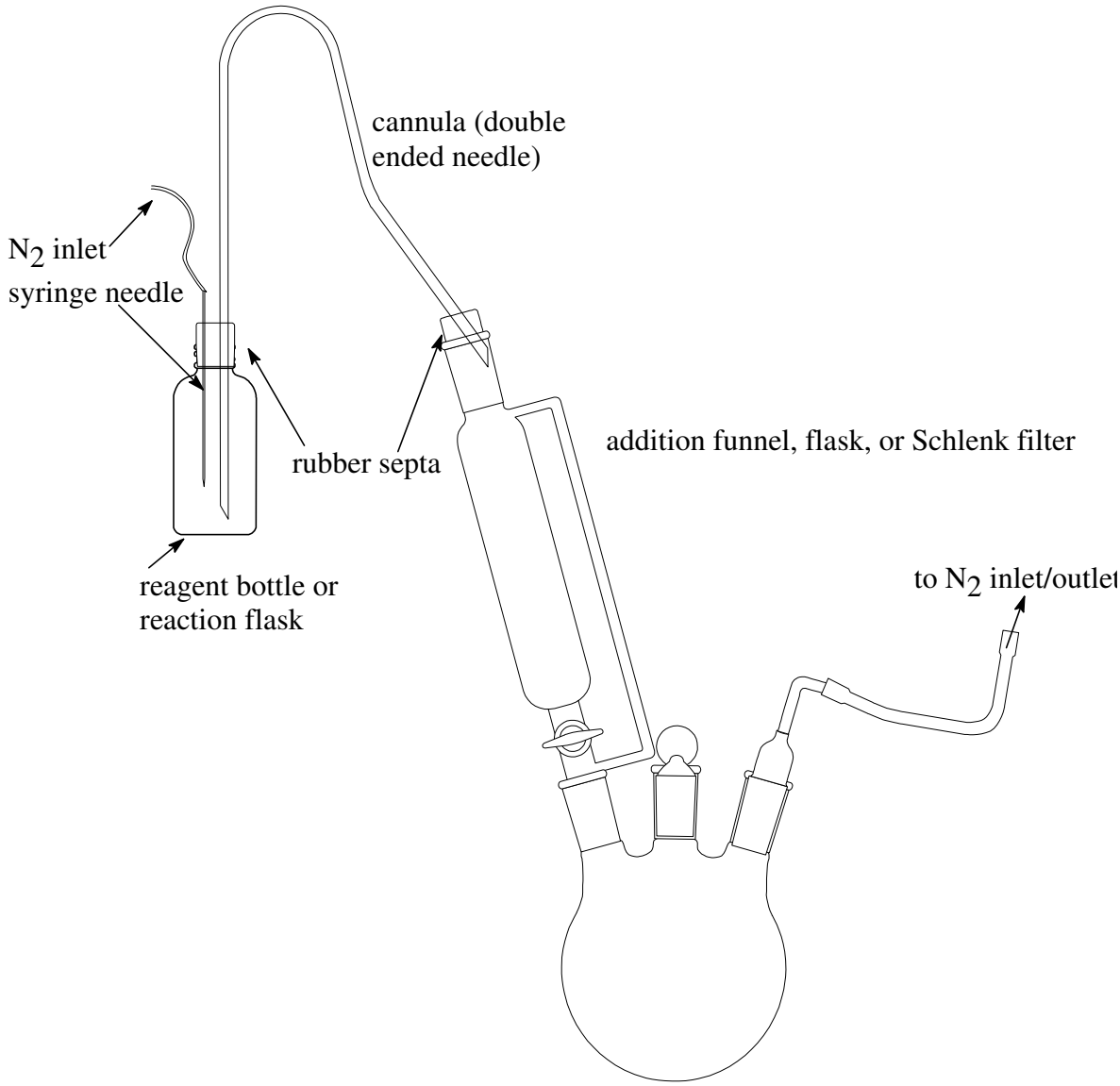
Cannula or Flex needle
2 rubber septa
Reagent container
Receiving container (N_2 purged)
N_2 source with needle attached
Outlet - mineral oil bubbler or second Hg manometer
Gloves (should be standard anyway)

Procedure:

If an exact quantity is to be transferred, pre-measure the receiving container and mark the level needed or use calibrated glassware, e.g. addition funnel. (Use acetone, then dry receiver in oven for 20 min. before transfer. You may also use the reaction solvent and simply add it to the reaction flask. Note: In Chem 5192, you will not need a premeasured quantity because this procedure will only be used to transfer a reaction mixture to a airless fritted filter.) Connect outlet (see figure). With N_2 source off, place N_2 needle in the septum above liquid level of the substance to be transferred. Place the long needle of the cannula in reagent bottle also above liquid level. Flush with N_2. With N_2 off, place short needle into the receiving container and the long needle below the liquid level of liquid to be transferred. Slowly turn N_2 on and liquid will be transferred by pressure. Turn N_2 off just before liquid is at marker and pull the long needle above liquid level but not out of bottle. Practice this step while liquid is being transferred to approximate amount contained in cannula.

If a mineral oil outlet is used, remove needle from oil, turn off N_2, remove cannula from receiver, remove needles from reagent container. If needle in mineral oil is not removed first, the oil may back up in reaction.

Clean cannula immediately with a solvent in which the transferred substance is soluble. Use and aspirator to draw this through the cannula. Rinse with water then acetone. Dry cannula by first blowing N_2 through it, then placing on house vacuum for ca 5 minutes. Store with corks on needles to prevent injuries.


cannula (double
ended needle)
N_2 inlet
syringe needle
rubber septa
addition funnel, flask, or Schlenk filter
reagent bottle or
reaction flask
to N_2 inlet/outlet

Vacuum Line Transfer
(Gas to Liquid)

Purpose: To transfer gases or exceptionally volatile liquids

Equipment:

Vacuum line
Collection vessel or flask
Cold bath of desired temperature
Lecture bottle and valve, Teflon tape two wrenches large clamp
Stopcock adapter

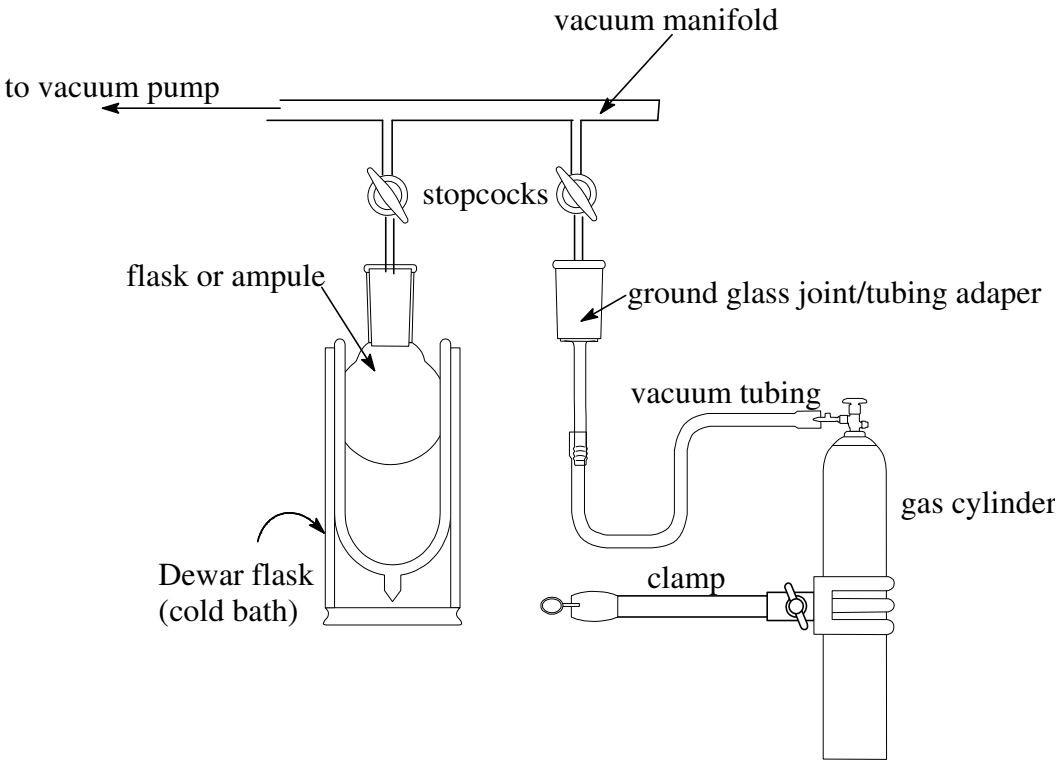
Procedure:

Attach vessel with stopcock (ampule or flask with stopcock adapter). Evacuate the assembled system until a good vacuum is achieved. Setup dewars to chill vessel with cold bath. Use the gas laws and the volume of the vacuum system to calculate the pressure of the gas that should be expanded into the system to get the proper molar quantity of the gas.

Prepare needle valve threads with Teflon tape, then attach to lecture bottle (finger tight plus a bit of a turn with the wrench). Attach hose to valve and vacuum line. Support lecture bottle well and clamp loosely. Evacuate needle valve by opening both the vacuum line stopcock and the valve. Check for leaks by closing vacuum system and watching manometer Hg level for several minutes. When thoroughly evacuated, close the valve. Next, close the stopcock between the muck trap/vacuum pump and the main manifold of the vacuum line. Make sure that the mercury manometer is opened to measure the pressure of the gas to be transferred. Chill the collection vessel with cold bath, but leave the stopcock closed.

[CHEM 5192: **The following procedure is to be done only by the instructor**]. Carefully open the main valve on the lecture bottle and the needle valve and watch the mercury as the pressure increases. When the precalculated, desired amount has been expanded into the system, close the valve on the lecture bottle, the main valve to the actual bottle, and the stopcock between the main manifold and the lecture bottle. Open the stopcocks to the chilled collection vessel and wait for the gas to condense into the vessel. When completely condensed, close the stopcocks to the vessel and open the vacuum system to the muck trap/vacuum pump. Evacuate the valve (to remove corrosive or toxic residual chemical) before disconnecting the lecture bottle from the vacuum system.

Hint: Think carefully throughout this procedure. It is all common sense gas laws.


vacuum manifold
to vacuum pump
stopcocks
flask or ampule
ground glass joint/tubing adaper
vacuum tubing
gas cylinder
Dewar flask
(cold bath)
clamp