RISE Catalysis Center Glovebox Manual

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Introduction

This manual provides an overview of proper operating procedures for the RISE Catalysis Center double-box glovebox. New users will be introduced to the best practices for operating and maintaining gloveboxes. The glovebox provides an oxygen-free environment by virtue of a purifying catalyst bed consisting of activated molecular sieves and copper oxide. There are two gloveboxes that are connected through a T-shaped large antechamber. The box on the left is a fully dry box: no volatiles or solvents are to be used. This box is equipped with a balance, vials, spatulas, kimwipes, and a –30 °C freezer. The right box is a fully equipped "wet" box, equipped with: auxiliary vacuum feedthrough, vacuum desiccator, a multi-well shaker, various auto-pipets, vials, glass pipets, kimwipes, stir plate, ring stands with clamps, celite, alumina, sieves, and a selection of solvents. This manual outlines glovebox operating procedures designed to provide a safe and efficient working environment for all users.

Quick Reference

Note that water and protic solvents (such as MeOH, EtOH) are not allowed in Janus.

Table 1. Guide to chemical compatibility with catalyst.

Functional Group	Examples	Purifier (Open/ Closed)	Notes
Saturated hydrocarbons	Hexanes, pet ether, cyclohexane	Open	
Olefins	Octene, cyclohexene, isobutene	***Closed***	Olefins bind the catalyst tightly.
Aromatic hydrocarbons	benzene, toluene	Open	No haloarenes
Ethers	Diethyl ether, THF, dioxane	Open	
Halogenated hydrocarbons	Dichloromethane, chloroform, chlorobenzene	***Closed***	Chlorinated solvents react with the Cu catalyst to make Cu–Cl bonds.
Strong σ-donors / bases	Phosphines, nitriles (incl. MeCN), pyridines, amines	***Closed***	Any reagent/solvent that is a good ligand for late metals will bind the catalyst.

Table 2. Guide to commonly used solvents.

Solvent	Purifier Open or Closed?
Pentane, hexane, cyclohexane, petroleum ether	Open
Benzene, toluene, xylene	Open
Diethyl ether, THF	Open
Dichloromethane, dichloroethane, chlorobenzene, fluorobenzene	***Closed***
Acetonitrile	***Closed***
Pyridine	***Closed***

Table 3. Purge times.

The glovebox should be purged after using ANY volatile solvent. Below are suggested timeframes for purging of solvent [classes]. *If your solvent of choice is not listed, purge the box for at least 10 minutes.*

Reason for Purge	Length
Halogenated solvents (e.g., CH_2Cl_2, C_6H_5F)	10 min
Coordinating solvents (e.g., PMe_3, MeCN, PhCN)	15 min
Pentane, hexane, cyclohexane, petroleum ether	10 min
Benzene, toluene, xylene	10 min
Diethyl ether, THF	10 min
O_2 spike (10-50 ppm)	10 min
O_2 spike (50-150 ppm)	20 min
O_2 spike (>150 ppm)	40 min (turn off O_2 analyzer)

Please note that often times our analyzer will spike when first turned on. This is a function of the O_2 analyzer and does not require a purge of the box.

Table 4. Glovebox Boxes

Box Side	MBraun Project #	Specialty
Left, dry side	17-128	Rigorously dry atmosphere, solids
Right, wet side	17-128	"Wet" atmosphere

Purge and Antechamber Logs

The use of the logs is critical. Other users need to know when the box is in use, when the antechambers are in use, and why the box is purging. When purging the box, note the time when the purge was started, the duration of the purge, the reason for purging (i.e. "after CH_2Cl_2").

Before pumping anything into the box, check the logbook. If someone has recently exposed the antechamber to air, refilling it with N_2 from the box could compromise the atmosphere (even when coming out of the box)!

Nitrogen Tanks

The liquid nitrogen tank that feeds into the two boxes is stored in room 007 (left tank). The nitrogen feeds through the ceiling across the two rooms. Always check the psi on the tank before purging or using the box. After using the box, note the percentage liquid nitrogen remaining on the tank and write it on the log. Alert Brandie or the TA if the tank is empty and in need of changing. Routine glovebox users can be trained on how to change out the tank as well.

Antechambers.

Standard Procedures:

Always check to see if the antechambers were recently opened to air. Be careful not to refill the antechambers immediately after they have been exposed to air, as this could contaminate the glovebox atmosphere.

Rewritten entirely:

Small antechamber: There are small antechambers for both sides of the glovebox and they are both refilled from the dry box. Ensure the large antechamber valve and the small antechamber you are not using are in the "closed" position. Refill the small chamber, then turn the valve to the "closed" position and open the outer door. Insert vacuum-ready objects (see "bringing in chemicals" section below), close the door, and turn the valve to the "vacuum" position. Five (5) evacuate/refill cycles should be performed, with each evacuation lasting for at least 30 seconds after reaching full vacuum. After refilling the

antechamber, the valve should be pointed to the "closed" position before opening the inner door. Bring objects into the glovebox, close the door, and don't forget to evacuate the small chamber and return the other small antechamber to dynamic vacuum. To remove objects from the glovebox, first ensure that the antechamber has been sufficiently evacuated since being exposed to air. Then, refill the chamber, turn the valve to "closed" position, and open the inner door. Insert items to be removed from the box, close the door, and then open the outer door. Remove items, close door, and evacuate the chamber. Both small antechambers should remain under dynamic vacuum (valve in evacuate position) when not in use.

Large antechamber: To bring items in through the large antechamber, first turn both small antechambers to the closed position. The antechamber refills from the dry box. The large antechamber should undergo three evacuate/refill cycles, with each evacuation cycle lasting at least 20 minutes. To bring items out of the glovebox, make sure the large antechamber has been evacuated sufficiently since being exposed to air. If the large antechamber was under static vacuum (which is its usual state with the valve in the close position), partially refill the antechamber, then evacuate to full dynamic vacuum for a few minutes.

Overnight evacuation: Anything that will be stores in the box long-term that has not bee oven-dried, such as plastic, paper, wood, or rubber should be pumped into the box overnight. This includes electronics(fans' stirplates) and especially paper products. Kimwipes should be heated to 60 ºC for 48 hours before pumping in overnight.

It is important to observe the logbook, and be aware of recent antechamber activity. Remember that the large antechamber connects both sides of the glovebox and if the wet side of the glovebox is not clean, care must be taken to ensure that the dry side atmosphere is not contaminated from moving between the two sides. If the large antechamber door is opened to the wet side and there is solvent in the atmosphere, the antechamber must be evacuated and refilled before reentering the dry side through the large antechamber.

Once all work is complete in the large antechamber, fully evacuate the chamber and then change the valve to closed. This puts the antechamber under static vacuum, which is how it should remain when not in use (note this is different from the small antechambers).

Maintaining A Good Atmosphere

Each glovebox is equipped with a separate activated catalyst bed (a.k.a. purifier) which is able to remove O_2 and H_2O. For the dry box, the levels are <0.5 ppm, and <0.1 ppm for the "wet" box. In normal operation the purifier is recirculating the N_2 atmosphere of the glovebox over the catalyst bed repeatedly. This means that N_2 in the glovebox should be improving over time, as more O_2 and H_2O are removed from the atmosphere with each pass over the catalyst bed. A good catalyst is essential to maintaining a healthy glovebox. Users can help maintain the efficacy of the catalyst and preserve the quality of the atmosphere by using care when working in the glovebox: only use the solvents compatible with the catalyst bed when the purifier is operating, operate the antechambers as prescribed above, and use care when incompatible solvents or chemicals must be used in the glovebox. Details on these procedures are found below. Check the atmospheric oxygen levels regularly by looking at the O_2 analyzer readout on the touch screen. O_2 levels should not be above 5 ppm during regular use. ***Note: The O_2 analyzer reading is only accurate if the purifier is on.*** If the O_2 level spikes above 5 ppm, try to determine what caused the spike (antechamber not fully pumped down, hole in glove, etc), and purge as appropriate to reduce the O_2 level. Note the O_2 level and (if known) what caused the spike in the log. If the problem is persistent and the cause is not readily apparent, notify Brandie or a TA to start an investigation.

Noises

Gloveboxes make a variety of distinctive noises. The user should familiarize him or herself with the common noises that a functional glovebox makes. Unfamiliar noises should be noted; unusual noises are

very often a symptom of problem. When the large chamber pumps down, there is a very loud sound. If the problem cannot be detected and remedied, notify Brandie or the TA. Because of the importance of recognizing unusual noises, headphones must not be worn while working in the glovebox, and radios or CD players should be kept at a reasonable level. Common sources of unusual noises include vacuum pumps, the circulation blower, pneumatic valves, and the refrigeration apparatus.

Group Supplies

As of now, supplies will be restocked by glovebox users. Please be mindful of other users and take responsibility for items that you have used up that need to be replaced.

Vials, Pipets, etc.

The common tools for inert atmosphere glovebox reaction chemistry will be provided for group use. Reusable tools such as spatulas, pipet bulbs, and filtration devices should be replaced where they belong and kept clean. Disposable items will be maintained in the box as well, including vials (20, 4 mL, GC), corresponding caps, glass pipets, plastic syringes, disposable needles, micropipette tips, kimwipes, and grease. Replacement vials and pipets should be kept in the oven, ready to be pumped into the box. Kimwipes should be warmed (carefully) in a cooler oven if possible, for 1-2 days; then, they are to be pumped on overnight in the large antechamber. Leaving kimwipes to pump in during the weekend is a good practice. Kimwipes retain an incredible amount of water.

Celite / Sieves / Alumina

These staple chemicals will be available for use by the group. They are dried by heating to 200 °C overnight under vacuum. Due to the variety of coordinating solvents used in the glovebox, the ***utmost care*** should be taken to avoid exposing these containers to solvents (see Solvents section). Keep the celite, sieves, and alumina closed if solvents are present in the box atmosphere. Plan ahead: take the celite you need before you start a reaction with a volatile/solvent. Consider carefully what is in the atmosphere before opening these canisters, so that they remain useful tools for the group. If contaminated, you and your fellow users' chemistry will be compromised.

Solvents

Solvents available for use include diethyl ether, tetrahydrofuran, benzene, toluene, methylene chloride, and acetonitrile. Solvents will be stored in 0.5 L glass bottles equipped with activated 3 Å molecular sieves. Users are responsible for bringing solvents into the box. If a user empties a solvent flask, he/she must refill it. There is a solvent log next to the glovebox system, please log where you refill the solvents from. If a user plans on using a large (>100 mL) amount of solvent, the user should bring in their own solvent, and not use group supply. Solvents taken from the activated alumina solvent columns should be briefly degassed, and then tested with a purple solution of sodium benzophenone ketyl radical (except halogentated and coordinating solvents). See ketyl radical recipe below.

Procedure for testing solvents: Fill a 4 mL vial with solvent. Add one drop of ketyl solution, and stir/shake the vial. If the solution stays purple, it is dry. If it goes colorless or blue, add one drop at a time until it turns purple. The general guidelines for dryness are as follows: pet. ether, benzene, and toluene, no more than 1 drop to purple; Diethyl ether, up to 2 drops to purple; THF up to 4 drops to purple. If a solvent does not pass, try degassing more, then testing again. If it still does not pass, but is relatively close, the solvent can be run through alumina in the box, and tested again. If it is not close, remove the solvent from the box. The user bringing in the solvent is responsible for the solvent being dry. If it is not dry, do not leave it in the box!

In order to keep the atmosphere clean, and to maintain a box that is suitable for everyone, only certain solvents will be allowed to be open at the same time, and thus allowed to intermix slightly. These "good" solvents are petroleum ether, diethyl ether, tetrahydrofuran, benzene, and toluene. These solvents can be used while the catalyst is open, and can be opened at the same time or one after another without purging. Chemicals in the same class, that are not common solvents, such as hexanes, dioxane, or xylenes, can be used with the same guidelines. Oxidizing, coordinating, or otherwise damaging solvents are considered "bad" solvents. These solvents have the ability to a) reduce catalyst lifetime and b) react unfavorably with other users' compounds. Therefore, when methylene chloride, acetonitrile, or amines are used, the catalyst must be closed, and the solvents must be kept isolated from each other, and especially the "good" solvents. Under no circumstances should a "good" solvent be opened when the atmosphere is contaminated with a "bad" solvent. If a user wishes to use these two classes of solvents at the same time, it is recommended that the user keep a 20 mL vial filled with the "good" solvent, only to be used when bad solvents are in use. When a "bad" solvent is in use, the user should make a public note of that, either on the white board or with sticky notes, so that other users do not unwittingly open their chemicals when the atmosphere is bad. Finally, the freezers and other group supplies such as celite, sieves, and alumina, should not be opened when "bad" solvents are in the atmosphere.

Deuterated Solvents

All deuterated solvents should be dried on a personal need basis, and kept in personal storage. Remember that deuterated solvents react the same as their proteo analogues, so CD_2Cl_2 is a "bad" solvent and requires the catalyst to be closed during use and a purge after use.

Purging

The box should be purged if the atmosphere is contaminated. A researcher using "bad" solvents (see above) or other chemicals that would react with the catalyst (e.g., volatile acids and phosphines, amines, chlorinated solvents) is responsible for closing the catalyst bed during use and purging the glovebox thoroughly after contaminating the atmosphere. The table at the beginning of this document can be used as a guide, but purge time should be adjusted based on the amount of contamination (i.e., how much solvent was used and for how long).

Suggested purge times:

≥10 minutes after volatile "bad" solvents (e.g. CH_2Cl_2)

≥15 minutes after less volatile "bad" solvents (e.g. CH_3CN)

Table 4. Purge times.

The glovebox should be purged after using ANY volatile solvent. Below are suggested timeframes for purging of solvent [classes]. *If your solvent of choice is not listed, purge the box for at least 10 minutes.*

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Halogenated solvents (e.g., CH_2Cl_2, C_6H_5F)	10 min
Coordinating solvents (e.g., PMe_3, MeCN, PhCN)	15 min
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Benzene, toluene, xylene	10 min
Diethyl ether, THF	10 min
O_2 spike (10-50 ppm)	10 min
O_2 spike (50-150 ppm)	20 min
O_2 spike (>150 ppm)	40 min (turn off O_2 analyzer)

Please note that often times our analyzer will spike when first turned on. This is a function of the O_2 analyzer and does not require a purge of the box.

The glovebox can be used during a purge, but this is only recommended when absolutely necessary. The N_2 regulator should not need to be adjusted during purges (or at any other time). However, it is possible that the problems in the N_2 supply (bad tank, pressure drop, etc) could cause over or under-pressurization of the box. Therefore, it is essential that users pay attention to purges and listen for clicks and other sounds that may indicate a problem.

Pressure limits

*Recommended **working** pressure limits:*

2 – 6 mbar.

The pressure should be between 6 and 8 mbar during a purge. Avoid negative pressure, mostly commonly encountered when the large antechamber is filled too quickly. Likewise, avoid large positive pressure, as this could lead to breaking the plexiglass or the gloves popping off! This could be due to a malfunction of the footswitch or the electronic controller. Use the footswitch when necessary; it is a good idea to use the footswitch to raise pressure when filling the large antechamber. In cases of emergency: if the glovebox is overpressurizing, and the foot pedal is not working, refill the large antechamber, which will take N_2 out of the glovebox and into the chamber, lowering overall pressure OR you can shut off the nitrogen flow by closing the valve on the wall beside the dry box. Seek the cause quickly, as this is only a temporary fix. If the box is underpressurized, use the foot pedal, or insert your hands into the box to increase the pressure. Check that the N_2 tank is not empty.

Purification and Circulation

The purifier is controlled by the touch screen (under Functions). When the purifier button is shaded green, it is on, as further indicated by an audible whirring sound. Press the button to turn off the circulation and close the catalyst bed / purifier. The purifier should always be closed when using "bad" chemicals (chlorinated reagents, amines, phosphines, etc). After using "bad" chemicals, the glovebox should be purged before the purifier is opened.

Auxiliary Vacuum

The box will be equipped with an auxiliary vacuum pump for filtrations and removing solvents. A desiccator equipped with a Kontes valve for slow depressurization is available in the "wet" box. This allows for the safe and efficient removal of solvents from multiple vials at a time. Different sized vial-holders are also provided. The first user to need vacuum should set up the trap, cooled with liquid N_2. That user will be responsible for the trap for the day, until responsibility is passed on to another user, or the trap is dropped. When the trap is put up, put your initials on the white board, and if responsibility is passed on, the new user should change the initials to their own. Before evacuating "bad" solvents or other chemicals that may contaminate other compounds that are under vacuum, one should consult with the other users of the box. In case they cannot be found, their containers should be closed to vacuum before pumping on contaminating mixtures. Users pumping on contaminating mixtures should leave a note on the white board mentioning the nature of the contaminant (i.e. CH_2Cl_2 in VAC). The last person to work in the box should check that the trap was taken down. All the valves inside the box and the valve next to the trap must be closed at the end of the day. If the trap is to be left up overnight, make sure the dewar is completely full before you leave, and remember to refill it in the morning. Specific instructions for operating the auxiliary vacuum are outlined below.

Trash

Glass waste and other waste trash bins are available outside of the box. As with other common group containers, these should not be exposed to "bad" atmosphere or glassware used with "bad" solvents. Contaminated glassware and kimwipes should be brought out after use, and before the "after bad solvent" purge. If contamination accidentally occurs, the box should be purged with the trash containers open. *To avoid accidents, waste contaminated with pyrophoric material should be handled by the person generating it.* Trash should be stored open in the hood for 24 hours before disposal. Empty trash containers must be pumped in overnight.

User Chemicals

Bringing in chemicals

Solids: To bring in dry, air exposed solids, cover the container with kimwipe secured with a rubber band. Make sure the solids are not prone to sublimation (e.g., ferrocene, Co_2CO_8); if they are a sublimation risk, degas in a Schlenk flask cooled with dry ice/acetone, and bring the sealed vessel into the box. Reactions run in Schlenk flasks can be dried under vacuum to leave a solid residue before being brought into the box. Vials or glass bottles from other boxes should be tightly sealed with electrical tape before exposure to air. These bottles can be usually be safely evacuated (although beware odd-shaped glassware or very large bottles).

Liquids: Purify and degas liquids by standard procedures and store outside of the box in a Teflon-stoppered flask (*no ground glass stoppers!*). Vessels containing liquids can be pumped in under pressure or vacuum if a Teflon-stoppered vessel (e.g., "bomb" or "Straus flask") is used. Schlenk flasks are not recommended for bringing liquids in the box. If you must, make sure that the stopper is taped tightly to the flask; *watch and listen for implosions*! To bring in Sure-Seal bottles, seal the cap with electrical tape, then pump in the bottle, turn off the purifier, take the cap off, purge, and turn on purifier.

All chemicals should be labeled with the date when they were opened (in or out of the box), the date they were brought into the box, and the initials of the person bringing them in. Chemicals that are going to be a risk to the box (acids, chlorinated, etc) should be taped tightly at all times, to prevent leakage and contamination.

Freezers

Each group will have a designated shelf or part of a shelf. This will facilitate easy access to personal chemicals without moving others' vials. Large scale reaction mixtures stored on the tall shelves should be worked up and removed as soon as possible to allow other users to use these shelves. Our freezer is located on the "dry" side of the glovebox so there shouldn't be "bad" atmospheric conditions present at ANY time…however, the following is included as a precaution for box usage:

Freezers should not be opened if the box atmosphere is contaminated with a "bad" chemical (moisture, acid, chlorinated solvents, amines, phophines, etc.). If the freezer gets contaminated, the box should be purged with the freezer open. Be careful when accessing your chemicals, as your labmates may have crystallizations set up in the freezer.

Storage

In order to allow efficient storage, stackable containers should be used. Chemicals should be stored on the shelves rather than on the floor, if possible (exceptions: large or unwieldy bottles). Containers with tools and materials for general use (spatulas of various types, grease, tape, caps of

various sizes, stir bars, rubber stoppers, etc.) will be available. Be mindful to others use of the glovebox if removing any of these items from the box.

Any materials for personal use should be stored in personal storage space. Chemicals that are sensitive to common atmospheric contaminants should be well taped. Chemicals that are themselves atmospheric contaminants should be well taped to prevent leakage into the atmosphere. As a courtesy to others, do not store large equipment or reaction apparatus on the floor for extended periods of time.

Pump maintenance

Pump oil in all glovebox pumps should be changed ***every six months.***

Pump oil in the antechamber pump should be changed ***after every glovebox regeneration***.

Regeneration

Regeneration should be performed when the purifier fails to keep the atmosphere clean. Purifier should be changed when regeneration fails to reactivate it. Regeneration is performed with 90/10 N_2/H_2 mixture, and takes about 800 psi of gas to regenerate the catalyst. The pump oil must be changed after regeneration.

Benzophenone Ketyl Radical Recipe (Courtesy: Prof. Theo Agapie, Caltech)

Recipe: In the glovebox, weigh 28 mg (1.22 mmol, 1.6 equiv) sodium metal into a 20 mL scintillation vial. Add 0.137 g (0.752 mmol) benzophenone (Ph_2CO), 20 mL THF, and a magnetic stir bar. Stir vigorously overnight. The clear, colorless solution should darken steadily to a blue color before eventually turning inky purple. The excess sodium will keep the indicating solution active and accurate for longer.

Derivation:

We want to test for < 10 ppm water in our solvents. What concentration of ketyl radical do we need?

For any solvent, we can choose the following parameters:

Desired maximum level of water impurity: p x 10^{-6} (p ppm)

$d_{solvent}$ = density of solvent to be tested

$FW_{solvent}$ = formula weight of solvent to be tested

FW_{Ph2CO} = 182 g mol^{-1} = formula weight of benzophenone indicator

Volume of solvent to be tested: V mL (normally 4 mL – full small vial)

Moles of solvent = $(V \text{ x } d_{solvent}) / FW_{solvent}$

⇨ Max moles of water = $(p \text{ x } V \text{ x } d_{solvent}) / FW_{solvent}$

Prepare 20 mL solution of indicator to have the appropriate concentration to discolor when one drop is added to V mL of a solvent containing more than p ppm water.

Mass of Ph_2CO in a drop of solution: $(p \text{ x } V \text{ x } d_{solvent} \text{ x } FW_{Ph2CO}) / FW_{solvent}$

Volume of one drop (disposable Pasteur pipet) ~ 0.013 mL (153 drops for 2 mL of THF)

Mass of Ph_2CO for 20 mL indicator solution:

$(p \text{ x } V \text{ x } d_{solvent} \text{ x } FW_{Ph2CO} \text{ x } 153 \text{ x } 10) / FW_{solvent}$

For testing THF:

$FW_{solvent}$ = 72 g mol^{-1}

$d_{solvent}$ = 0.889 g mL^{-1}
V = 4 mL
p = 10 x 10^{-6} = 10 ppm

Hence, $10x10^{-6}$ x 4 x 0.889 x 182 x 153 x 10 / 72 = 0.137 g Ph_2CO for 20 mL indicator solution. Excess sodium was used (28 mg, 1.6 equiv).

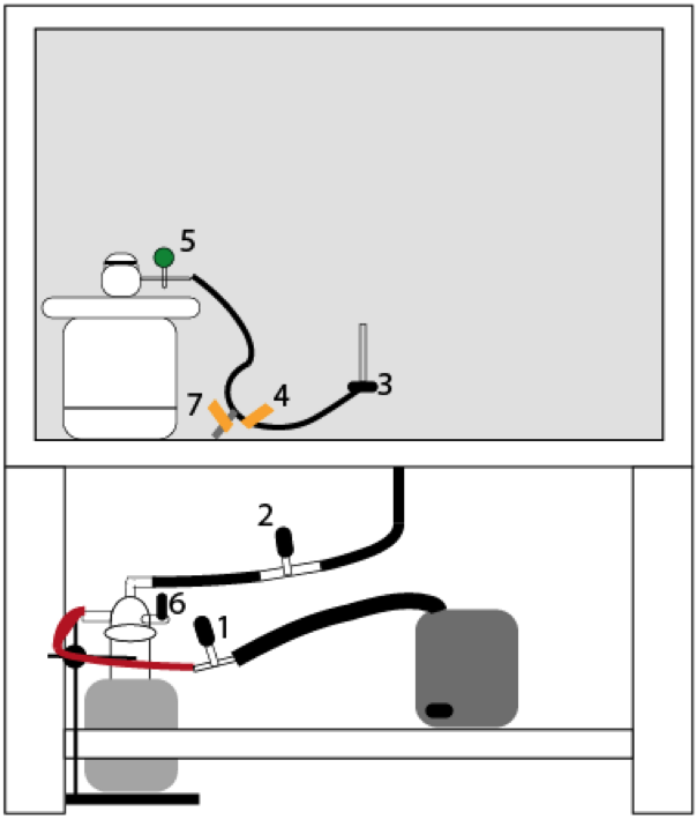
Concentration of Ph_2CO in solution is 0.0377 M (moles L^{-1}).
For a different solvent, if the solution stays purple, max content of water is:
$p' = C_{Ph2CO}$ x 0.000013 x $FW_{solvent}$ / (V x $d_{solvent}$)

For pentane:
$FW_{solvent}$ = 72 g mol^{-1}
$D_{solvent}$ = 0.63 g mL^{-1}
V = 4 mL
⇨ p' = 14 ppm

Given that the solution prepared above precipitates a little solid, the concentration of ketyl radical is slightly lower, so the concentration of water in the solvent is even smaller. If even more rigorously dry solvents are required, the volume of solvent tested could be doubled.

Janus Vacuum Desiccator SOP

Written by Hannah Starr 10/15/18



Description of valves:

1. Valve that isolates vacuum from the trap
2. Valve that isolates the inside of the glovebox from the outside. THIS VALVE SHOULD ALWAYS BE CLOSED UNLESS YOU ARE ACTIVELY UNDER VACUUM.
3. Connecting valve from the tube that brings vacuum into the box. Valve is closed when it is turned perpendicular to the metal tube.
4. Additional connecting valve
5. Green circular valve that isolates the desiccator
6. Trap release valve
7. Interior vacuum release valve

Vacuuming down samples:

At least 10 minutes before you are ready to remove solvent, assemble trap and turn on vacuum pump. Do not add liquid nitrogen yet. This process removes any oxygen from the trap. You will want **valve 1** open for this process and all other valves closed.

After complete evacuation of the trap, lower the trap into the empty dewar and then pour liquid nitrogen around the trap. The trap should be mostly in the dewar, but still clamped to the stand. See the diagram above.

Add your samples (in a vial holder) to the desiccator and close the lid. Use secondary gloves when handling any of the greased components! There is a round glass top (that contains **valve 5)** that you will place over the top of the desiccator. Be sure the holes from the top of the desiccator and the additional

glass piece are aligned. To secure the top, there is a large black O-ring (usually kept in the yellow bin above the shaker) that keeps the glass top secure and in place.

Next, open **valve 2**. This evacuates up to **valve 3**. Note that this line is kept under static vacuum, so you should not expect to hear any major changes from the pump when you open this valve. Now open **valve 3**. This evacuates to **valve 4**. Check to be sure **valves 5** and **7** are closed. Open **valve 4.** The system is now evacuated to the desiccator. Slowly open **valve 5.** There should be a noticeable change in the sound coming from the pump. It will take a while to evacuate the full desiccator. The strength of the vacuum on the samples is controlled by **valve 5**. Solvents that are highly susceptible to bumping (i.e DCM, pentane) should be monitored closely. Solvents less susceptible to bumping (i.e. toluene) can usually tolerate a much stronger pull. Be sure to check on the liquid nitrogen level periodically and add as needed.

Turning the pump off:

This process is essentially the previous steps in reverse. Begin by closing **valve 5**. Next, close **valve 4**, followed by **valve 3**. Next, you will want to open **valve 7**. This will vent the line between **valves 4** and **5**. Begin opening **valve 5** to release vacuum inside the desiccator. Remove the O-ring and lift the glass piece containing **valve 5** from the top of the desiccator. Samples can now be removed from the desiccator.

To lower the trap and turn off the pump, close **valve 2**. This valve must remain closed when the vacuum is not in use. Next, raise the trap out of the liquid nitrogen dewar and quickly check for liquid oxygen (it will be blue). If there is no liquid oxygen, turn off the pump and open **valve 6**. You can now disassemble the trap and place it in the hood by the glovebox.

Troubleshooting:

If it does not appear that the pump is sufficiently pulling vacuum on your samples, first make sure all the proper valves are open (1,2,3,4,5 open and 6,7 closed).
Users have noted: if the interior green circular valve tubing is at a sharp angle, the top part can slowly turn and decrease the vacuum actually being pulled on the desiccator

Ensure that there is not a leak somewhere with the desiccator. If there is a leak, you should notice the pressure in the box decreasing if you hold still or remove your arms. If you find a leak here, close **valve 4**, vent **valve 7**, reassemble the desiccator, and try again.

If you had strong vacuum to begin with, but it appears that you have lost some or all, it is likely that you have frozen solvent clogging the tube within the trap. To fix this problem, you will have to go through all the steps to take down the trap and turn off the pump, and then clean out the tube and trap and reassemble (going through all the initial steps to set up the pump).