# THE SCHLENK LINE SURVIVAL GUIDE

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# The Schlenk Line Survival Guide

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This text was compiled on 05/09/2026

# TABLE OF CONTENTS


Licensing

## Guides

- 1: The Schlenk Line
- 2: Cycling onto the Schlenk Line
- 3: Performing Sensitive Reactions without a Schlenk Line
- 4: Transferring Liquids
  - 4.1: Cannula Transfer
  - 4.2: Syringes and Sure Seals
- 5: Inert Atmosphere Filtrations
  - 5.1: Cannula Filtration
  - 5.2: Filtration through Celite
- 6: NMR Preparation
  - 6.1: Preparing NMR Samples on a Schlenk Line
  - 6.2: Removing Solvent from NMR tubes
- 7: Distillations
  - 7.1: Static Vacuum Distillation
  - 7.2: Dynamic Vacuum Distillation
- 8: Freeze-Pump-Thaw
- 9: Drying Solvents
- 10: Removing Solvent
- 11: Addition of Solids
- 12: Refluxing Under an Inert Atmosphere
- 13: Gloveboxes

Index

Glossary

Detailed Licensing

# Licensing

*A detailed breakdown of this resource's licensing can be found in **Back Matter/Detailed Licensing**.*

# CHAPTER OVERVIEW

## Guides


1: The Schlenk Line
2: Cycling onto the Schlenk Line
3: Performing Sensitive Reactions without a Schlenk Line
4: Transferring Liquids
4.1: Cannula Transfer
4.2: Syringes and Sure Seals
5: Inert Atmosphere Filtrations
5.1: Cannula Filtration
5.2: Filtration through Celite
6: NMR Preparation
6.1: Preparing NMR Samples on a Schlenk Line
6.2: Removing Solvent from NMR tubes
7: Distillations
7.1: Static Vacuum Distillation
7.2: Dynamic Vacuum Distillation
8: Freeze-Pump-Thaw
9: Drying Solvents
10: Removing Solvent
11: Addition of Solids
12: Refluxing Under an Inert Atmosphere
13: Gloveboxes



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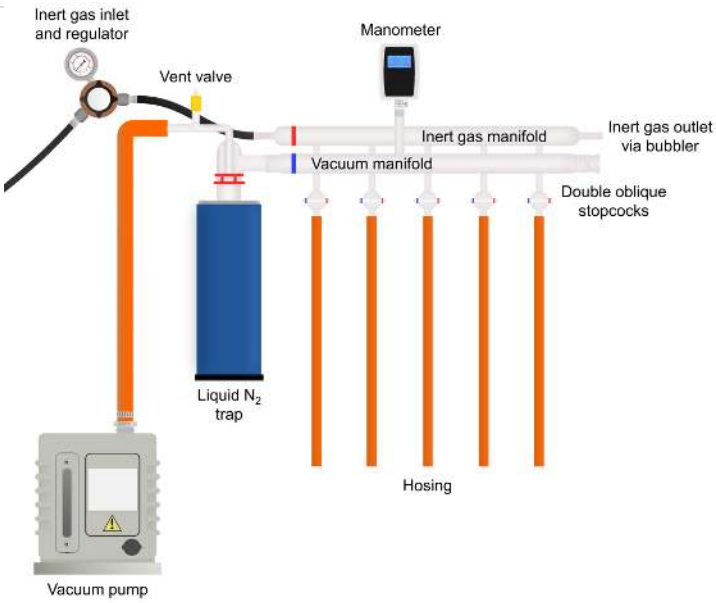
# 1: The Schlenk Line

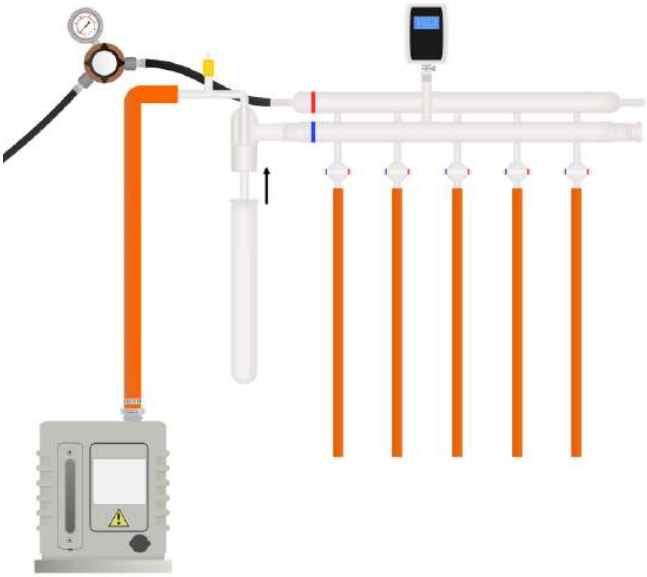



A typical Schlenk line.

Schlenk lines are versatile pieces of laboratory equipment that enable the safe and convenient manipulation of air- and moisture-sensitive compounds. Schlenk lines are dual manifold systems in which the vacuum manifold is connected to a vacuum pump, whilst the inert gas manifold is connected to a sources of purified and dry inert gas (typically nitrogen or argon). The two manifolds have several ports which are interconnected via double oblique stopcocks or Teflon taps, which allow sealed vessels attached to the Schlenk line to be evacuated under vacuum or back-filled with inert gas. Schlenk flasks feature a side-arm (with a greased stopcock or greaseless Teflon tap) that can be attached to the Schlenk line using flexible hosing, alongside a standard ground glass joint to attach other glassware or to simply insert a stopper or septum. Schlenk lines may also be equipped with suitable ground glass adapters to enable the direct attachment of appropriate glassware, without the needed for hosing. A slight over-pressure of inert gas is employed, and this exits the Schlenk line through a bubbler (oil or mercury) which acts a pressure relief system, allows the gas flow rate to be monitored, and prevents the ingress of air back into the Schlenk line. A cryogenic trap (typically liquid nitrogen, -196 °C) is used to condense solvent vapours and other volatiles, which protects and prevents contamination of the vacuum pump. The vacuum manifold may be fitted with a manometer to measure the vacuum pressure – for a typical Schlenk line equipped with a rotary vane vacuum pump, a pressure between $10^{-2}$–$10^{-4}$ mbar is often reached which is suitable for standard manipulations. Schlenk lines are often custom made for specific research laboratories, meaning that numerous designs and adaptations are possible depending on the intended applications.

**Starting up the Schlenk line**

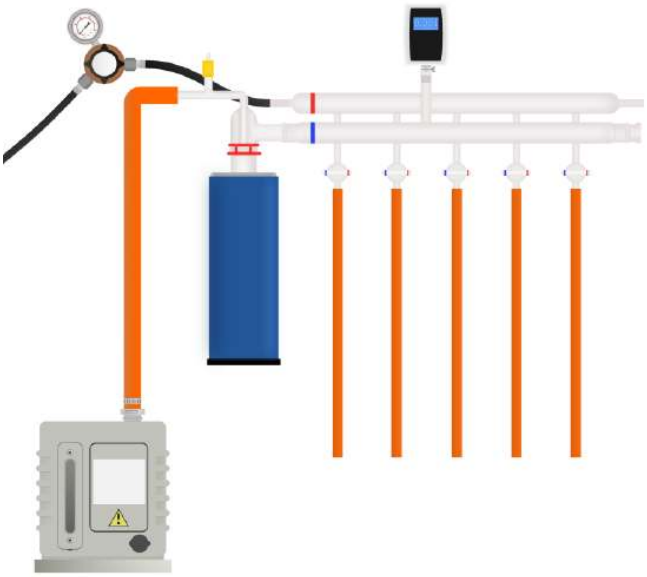
**Step 1:** Attach a clean and dry solvent trap to the Schlenk line ensuring that the male ground-glass joint is sufficiently greased. Twist the solvent trap to evenly coat the grease and then secure it in place with a clip. Solvent traps may instead be connected to the Schlenk line via greaseless O-ring joints.



Attaching the solvent trap to the Schlenk line.

**Step 2:** Ensure that the vent valve and all double-oblique stopcocks or Teflon taps are closed (or turned to inert gas if a vessel is already attached to the Schlenk line hosing). Turn on the vacuum pump.

**Step 3:** Wait for 5–15 minutes for the trap and vacuum manifold to be evacuated before submerging the solvent trap in a Dewar of liquid nitrogen – a manometer reading below 0.1 mbar is recommended to ensure there are no leaks within the Schlenk line assembly. The inert gas supply can now be opened and the Schlenk line is ready for operation; the inert gas supply can also be established independently of the vacuum for other applications (i.e. bubbling inert gas through a solvent to degas it).



Schlenk line equipped with a liquid nitrogen trap.

If the Schlenk line has just been assembled after cleaning, it is recommended to purge the inert gas manifold by passing inert gas through it for 15-30 minutes prior to use. Some Schlenk lines are designed to allow the inert gas manifold to be fully evacuated without any risk of oil suck-back or regulator damage.

**Shutting down the Schlenk Line**

**Step 1:** Ensure that all Schlenk flasks and vessels connected to the Schlenk line are under inert gas and that all other stopcocks and taps are closed if not in use.

**Step 2:** Turn off the vacuum pump then remove the Dewar of liquid nitrogen from the solvent trap. Open the vent valve to quench the vacuum and re-pressurise the vacuum manifold. Schlenk lines may have an additional Teflon tap to isolate the vacuum pump from the solvent trap and vacuum manifold.

**Step 3:** Allow any collected solvent in the trap to thaw before discarding into an appropriate waste container.

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# 2: Cycling onto the Schlenk Line

Glassware used for the manipulation and preparation of air- and moisture-sensitive compounds must be rigorously dried and free of air prior to use. Schlenk flasks and items are typically stored in ovens above 100 ºC (for a few hours or overnight) to remove any residual solvent or adsorbed water.

## Attaching a new Schlenk flask or reaction vessel to the Schlenk line

### Step 1

Remove a Schlenk flask, magnetic stir bar, ground glass stopper and stopcock from the oven using heat resistant gloves.



Schlenk flask, stopcock, stopper and stir bar.

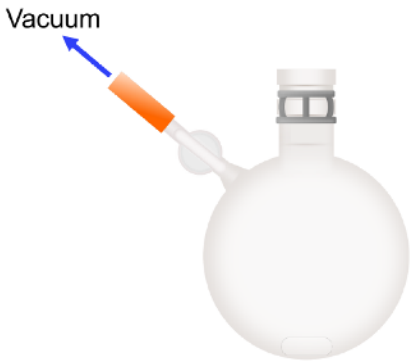
### Step 2

The stopcock and stopper are greased (using a length of silicone, hydrocarbon or polyfluorinated based lubricant) and inserted into the corresponding ground glass joints with twisting to ensure a smooth and uniform seal that is free of bubbles or streaks. Only two thirds of the stopper are greased to prevent excess grease from contaminating the inside of the flask. Ensure that the grease does not block the gas inlet/outlet hole on the stopcock. Appropriate nuts, O-rings, washers and clips can be added to secure the stopcock and stopper in place.

Greased and assembled Schlenk flask.

### Step 3

The assembled Schlenk flask is then connected to the Schlenk line hosing and evacuated under vacuum whilst it cools to room temperature. After a suitable amount of time (depending on the size of the flask, ~5 minutes for a 100 mL vessel), the Schlenk flask is slowly backfilled with inert gas. This corresponds to a single ‘cycle’, and this is repeated two more times to ensure complete removal of air and moisture from the reaction vessel.




Evacuating a Schlenk flask under vacuum.

## Cycling sealed vessels already under an inert atmosphere:

For ampoules and flasks that are already sealed under an atmosphere of inert gas, three vacuum/inert gas cycles are required to evacuate the air within the Schlenk line hosing and flask side arm. Shorter cycles (30-60 seconds) are typically sufficient for these purposes.

The process of performing numerous vacuum/inert gas cycles is akin to performing repeated extractions of an aqueous phase with organic solvents during a typical reaction work-up. Assuming that a 100 mL reaction flask contains approximately 1 mmol of $O_2$ at room temperature and atmospheric–pressure (~1000 mbar), then an initial vacuum cycle down to 0.1 mbar will reduce the quantity of $O_2$ down to 1 x $10^{-4}$ mmol of $O_2$. Backfilling with inert gas and re-evacuating the flask down to 0.1 mbar again reduces the $O_2$ quantity down to 1 x $10^{-8}$ mmol, and a third vacuum/inert-gas cycle further lowers this to 1 x $10^{-12}$ mmol of $O_2$. This is several orders of magnitude lower than performing a single but longer evacuation down to 0.001 mbar, for example, and illustrates that even poor performing vacuum pumps can be sufficient to reach low partial pressures of $O_2$ after repeated vacuum/inert gas cycles.

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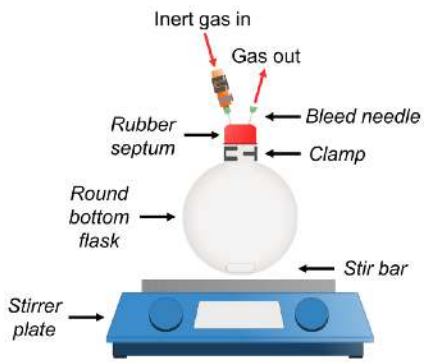
# 3: Performing Sensitive Reactions without a Schlenk Line

Whilst Schlenk lines and Schlenk flasks are the ideal equipment to ensure that reactions are free of air and moisture, it is still possible to perform air- and moisture-sensitive reactions without the need for specialist equipment. This is especially true when the products of the reaction are not particularly air- and moisture-sensitive but the reagents or catalysts used for the reaction are. In these cases, it is often sufficient to use a round bottom flask fitted with a rubber septum, and to have an inert gas inlet, either from an inert gas manifold or simply a balloon filled with inert gas.

A representative reaction (the synthesis of *N*-aryl amidines from secondary amines and benzonitriles – adapted from here) is used as an illustrative guide, but each step can be modified depending on the reaction conditions required and nature of the reagents.

## Step 1

An oven-dried round bottom flask equipped with a magnetic stir-bar is fitted with a rubber septum and clamped securely. A bleed needle is added followed by the inert gas inlet needle, and the flask is flushed for several minutes (depending on the volume) to remove air. *Note: If using a Schlenk line and Schlenk flask then the reaction vessel would instead be cycled onto the line prior to use.*




Flushing a round bottom flask with inert gas prior to use.

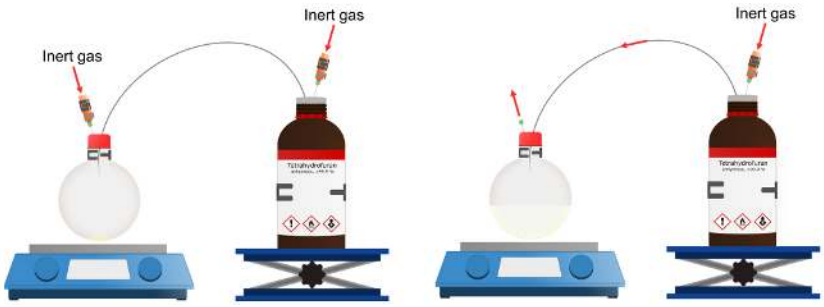
## Step 2

Once the reaction flask has been flushed with inert gas, the bleed needle is removed from the septum. For the addition of liquid reagents (aniline in this specific case), the syringe and needle is first purged three times with inert gas (this is recommended even for non air- and moisture-sensitive reagents) and then the required quantity of liquid reagent is added. For solid reagents, it is recommended to add the required quantity of solid to the flask prior to flushing with inert gas.

Addition of liquid reagents to the flask.

## Step 3

Anhydrous solvent (commercially available bottle equipped with a rubber septum) is added to the reaction flask. This can be achieved *via* cannula transfer (as illustrated below) or more conveniently by using a syringe and needle.



Addition of anhydrous solvent via cannula transfer.

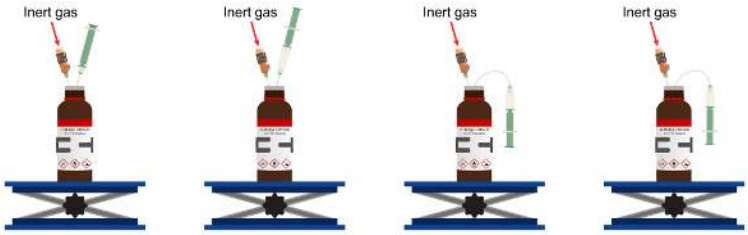
## Step 4

The reaction flask is cooled to 0 ºC in an ice bath. *Note: This is specific to the reaction.*

Cooling the reaction in an ice bath.

## Step 5

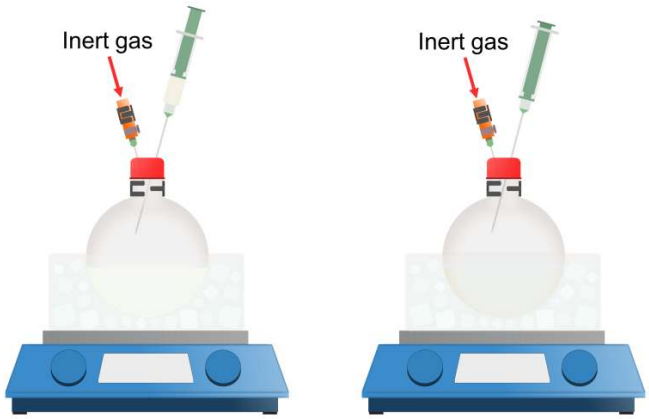
$^{n}$BuLi is dispensed from the reagent bottle using a syringe and needle (see here for full details). *Note: $^{n}$BuLi is prime example of a reagent that requires air and moisture free conditions for its safe handling, whilst the final product obtained after hydrolysis or electrophilic quenching may not be sensitive*.



Dispensing $^{n}$BuLi from the reagent bottle.

## Step 6

The $^{n}$BuLi is added dropwise to the reaction flask.



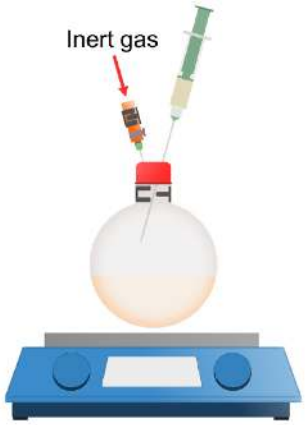

Dropwise addition of $^{n}$BuLi to the reaction flask.

## Step 7

The reaction flask is removed from the ice bath and allowed to stir for 1 hour at room temperature. *Note: If an inert gas balloon is used, it may be necessary to manually release excess pressure from the reaction flask using a bleed needle*.

## Step 8

The third liquid reagent (benzonitrile in this specific case) is slowly added to the reaction flask using a syringe and needle.




Addition of a liquid reagent.

## Step 9

The reaction is stirred at room temperature for 1 hour. The gas inlet is carefully removed, followed by the rubber septum, and the reaction is quenched with ice water. The product (*N*-phenyl benzamidine in this specific case) is obtained after work-up and recrystallisation.

## Tip, Tricks and Safety Notes

- When handling large quantities of pyrophoric reagents, it is strongly advised to use a Schlenk line and rigorously dried Schlenk flasks to ensure that reactions are free of air and moisture, and therefore performed as safely as possible.
- It may be convenient to add air sensitive solids such as catalysts or strong bases to the reaction flask using a glovebox. The flask can be removed from the glovebox fitted with the rubber septum to ensure it remains sealed and free of air and moisture.
- If the reaction is not sensitive to moisture, it may be sufficient to degas the solvent by **sparging** (bubbling inert gas through the solvent) directly in the reaction flask prior to use. Even if anhydrous solvent is used, it may be beneficial to sparge the solvent with inert gas for 5-10 minutes prior to the addition of reagents.

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# SECTION OVERVIEW

## 4: Transferring Liquids


### 4.1: Cannula Transfer

### 4.2: Syringes and Sure Seals



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# 4.1: Cannula Transfer

Cannula transfers are routinely used during the preparation and manipulation of air- and moisture-sensitive compounds to transfer solvents, solutions and suspensions between vessels. Cannulae are typically made of stainless steel or Teflon and come in a range of gauges depending on the volume of liquid to be transferred.

## Step 1

Cycle a solvent ampoule onto the Schlenk line with a minimum of three vacuum/inert gas cycles.

Cycling a solvent ampoule onto the Schlenk line.

## Step 2

Once cycled onto the line, replace the Teflon tap with a rubber septum under a positive pressure of inert gas. Insert a bleed needle (also called an exit or vent needle) into the septum for 5-10 seconds to purge out any air that is introduced into the ampoule. Ensure that the bleed needle only just pierces the septum so that the air/inert gas is forced out of the needle.

A solvent ampoule ready for cannula transfer.

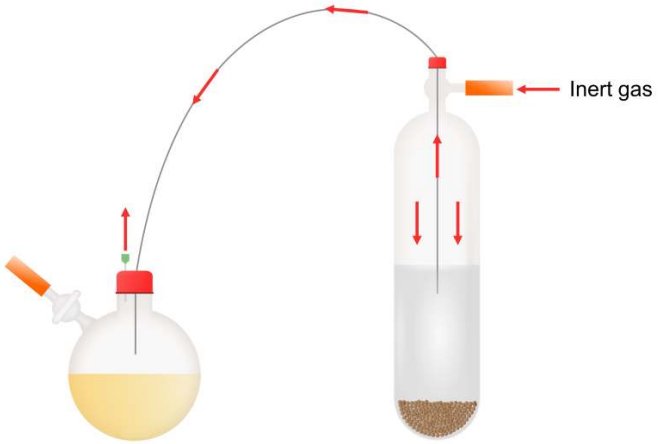
## Step 3

Insert a cannula through the septum on the solvent ampoule and allow inert gas to purge through the cannula for 15-30 seconds before inserting the cannula into the septum on the receiving flask.

Inserting a cannula into the solvent ampoule and receiving flask.

## Step 4

The cannula is lowered down into the solvent, then the stopcock on the receiving flask in closed, and a bleed needle is inserted into the septum on the receiving flask. This establishes a pressure differential which will transfer the liquid from the transfer flask into the receiving flask (inert gas flow marked with red arrows).




Transferring solvent from the ampoule to the receiving flask.

## Step 5

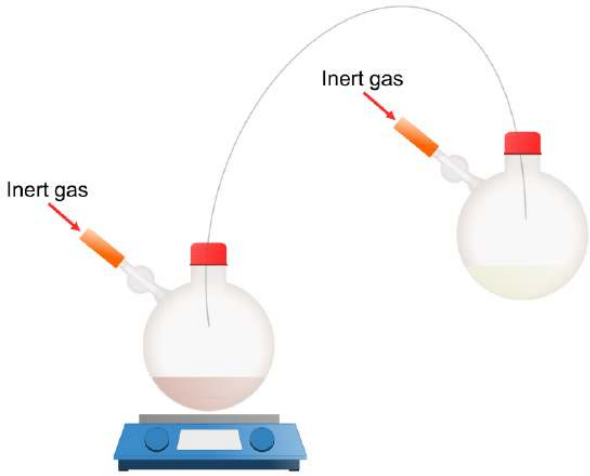
Once the desired volume of solvent has been transferred, the cannula is raised above the liquid level, the stopcock on the receiving flask is opened to inert gas, and the bleed needle is removed. The cannula is then removed from the receiving flask and transfer flask, and the septa are replaced with the corresponding stopper or Teflon tap.

This same technique can also be used to transfer solutions, slurries or liquid reagents between flasks. In these cases, the cannula should be cleaned immediately after use (typically with acetone and water).

## Dropwise Addition

### Step 1

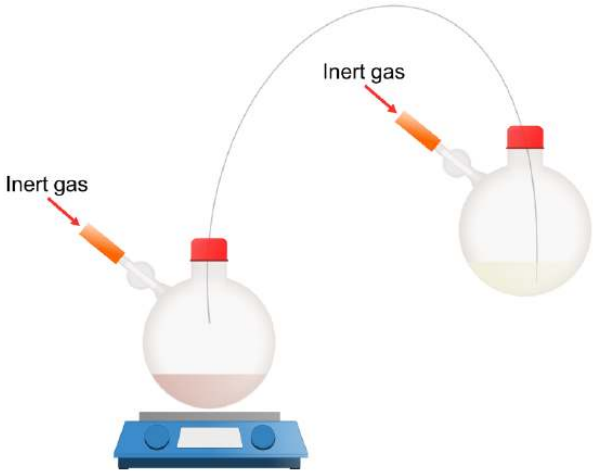
Replace the glass stoppers on the transfer and receiving flask with rubber septa under a positive pressure of inert gas. Insert a cannula in through the septum of the transfer flask and purge for 15-30 seconds before inserting through the septum of the receiving flask.




Transfer and receiving flasks prior to dropwise addition.

### Step 2

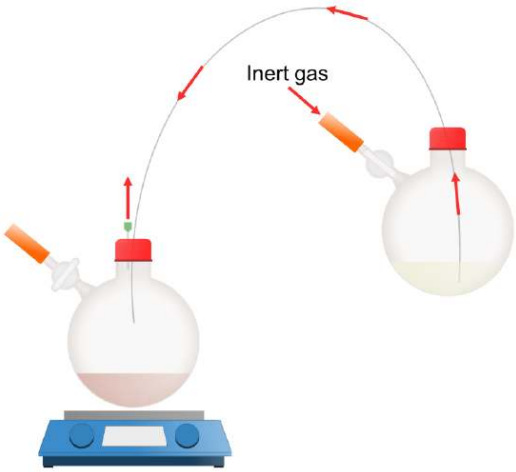
Lower the cannula into the solution of the transfer flask. Ensure that the bottom of the cannula in the transfer flask is higher than the bottom of the cannula in the receiving flask.




Lowering the cannula into the solution to be transferred.

### Step 3

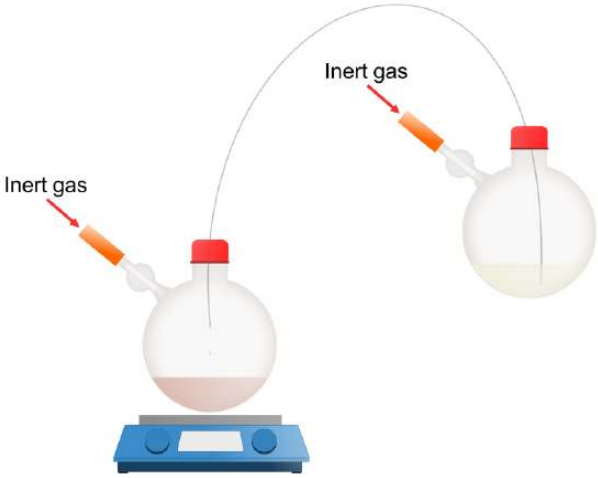
Insert a bleed needle through the septum on the receiving flask and close the stopcock to stop the flow of inert gas and initiate the cannula transfer. As soon as the first drop of solution passes through the cannula, open the stopcock on the receiving flask and remove the bleed needle to re-equalise the gas pressure and create a syphon.




Initiating the transfer by establishing a pressure differential.

### Step 4

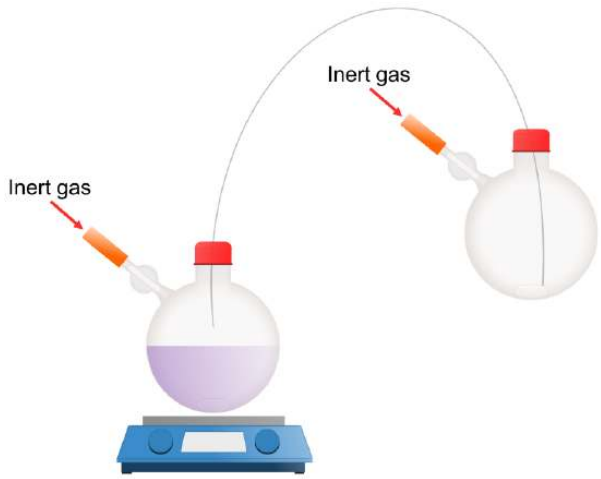
The rate of addition can be controlled by adjusting the relative height of the transfer and receiving flasks – raising the level of the transfer flask or lowering the cannula into the receiving flask will increase the rate of addition and *vice versa*.




Dropwise addition via cannula transfer.

### Step 5

Once all of the solution from the transfer flask has been added, remove the cannula from both flasks and replace the rubber septa with greased ground glass stoppers.

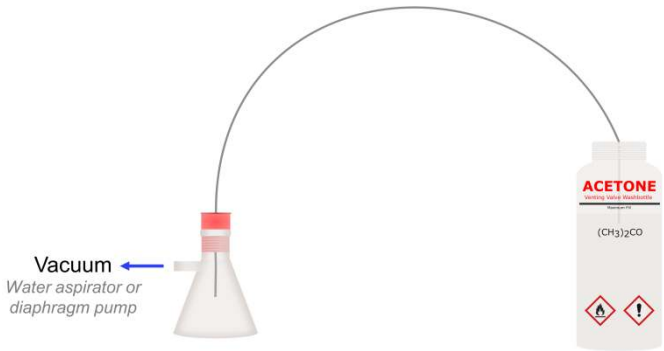



Reaction flasks after addition.

## Cleaning Cannulae

Cannulae can be easily cleaned by flushing them with suitable solvents or quenching solutions. For typical organic reaction mixtures, acetone and water are used, whilst cannulae used to transfer phosphines or thiols are typically washed with bleach. In any case, cannulae should be cleaned immediately after use to prevent blocking.

Using a water aspirator or diaphragm pump to pull a vacuum provides a simple way of pulling the cleaning solvents/solutions through the cannulae. Ensure that the final rinse is with a low boiling organic solvent (typically acetone) and allow to air dry before placing into the lower shelf (or away from clean and dry cannulae) of an oven.




Cleaning a cannula with acetone.

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# 4.2: Syringes and Sure Seals

Recommended reading: Safe Handling of Cannulas and Needles

Many air- and moisture-sensitive reagents or anhydrous solvents that are commercially available are stored under an inert atmosphere in a sealed bottle containing a penetrable rubber septum. Withdrawing the liquid from these sealed vessels requires an inert gas inlet to re-equalise the pressure and to prevent a partial vacuum; this could introduce atmospheric air and moisture through the compromised rubber septum and degrade the contents. When using a syringe and needle to transfer liquid reagents, and particularly when dispensing pyrophoric substances, it is essential that an appropriately sized syringe and needle gauge is used

## Step 1

A clean short (40–60 mm) needle is first inserted into the Schlenk line hosing using an appropriate screw-thread (Luer-lock) adapter and hose clamp. The needle and hosing are purged with inert gas for 15-30 seconds. During this time, the hosing and reagent bottle can be clamped in place.

Purging a gas inlet needle with inert gas.

## Step 2

Unscrew the cap from the reagent bottle, then remove the protective sheath from the needle and pierce the needle through the rubber septum.

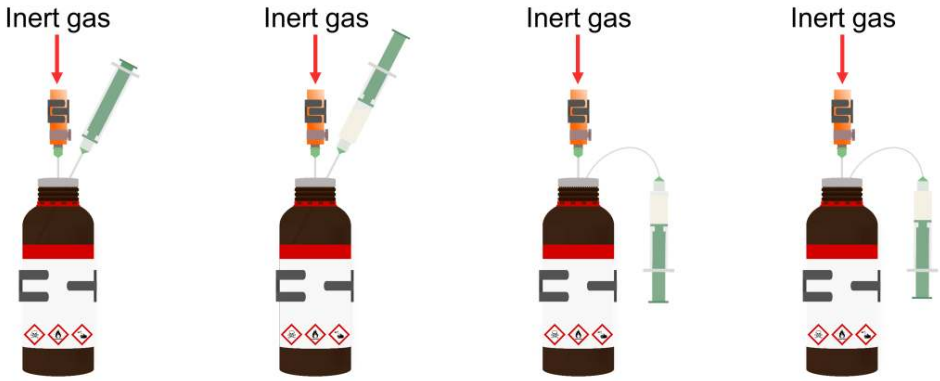
Inserting the gas inlet needle into the septum on the reagent bottle.

## Step 3

The disposable syringe and long (100–120 mm) needle used to measure the reagent is first purged by piercing it through the rubber septum on the receiving Schlenk flask, withdrawing inert gas into the syringe, removing the needle from the septum, and then expelling the gas. The user should monitor the bubbler when withdrawing gas or liquid into the syringe – if the bubbler stops then a local vacuum may have been introduced which compromises the inert gas atmosphere. This purging process is repeated two more times.

## Step 4

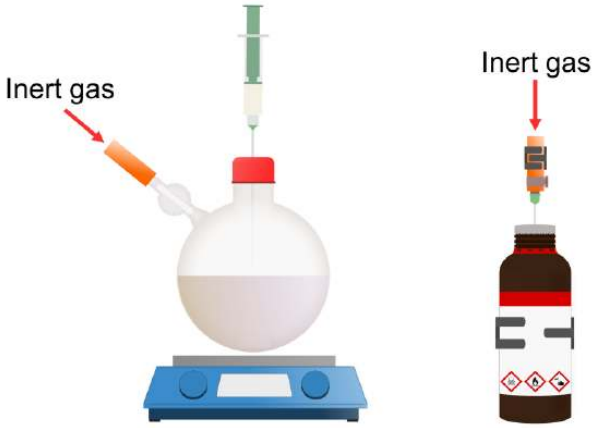
Insert the purged syringe and needle through the septum down into the reagent bottle. Slowly pull the plunger to withdraw the liquid into the syringe, removing slightly more liquid than required. With the needle still pierced through the septum, carefully bend the needle and turn the syringe vertically, then depress the plunger to remove the gas bubble. Continue depressing the plunger until the desired volume of liquid has been reached. *Note: It may also be desirable to withdraw a small pocket of inert gas into the syringe to protect the reagent or solvent from exposure to the atmosphere during transfer.*




Withdrawing liquid using a syringe and needle.

## Step 5

Carefully remove the syringe and needle from the reagent bottle and pierce it through the septum on the reaction flask. Slowly depress the plunger to expel the liquids. After addition, the needle and syringe are removed and disposed into an appropriate sharps and waste container. For pyrophoric reagents, the needle and syringe should be quenched with iso-propanol/toluene prior to disposal.




Adding the liquid to the reaction flask.

## Step 6

Remove the inert gas inlet needle from the reagent bottle and replace the screw cap.

For frequently used reagents such as $^{n}$BuLi, it is advised to transfer the contents from the bottle into an ampoule with a Teflon tap *via* cannula transfer since the rubber septa are prone to leaking after repeated use. When large amounts (>20 mL) of reagent from a sure seal bottle are required, it is recommended to directly cannula transfer the liquid into a graduated dropping funnel.

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# SECTION OVERVIEW

## 5: Inert Atmosphere Filtrations


### 5.1: Cannula Filtration

### 5.2: Filtration through Celite



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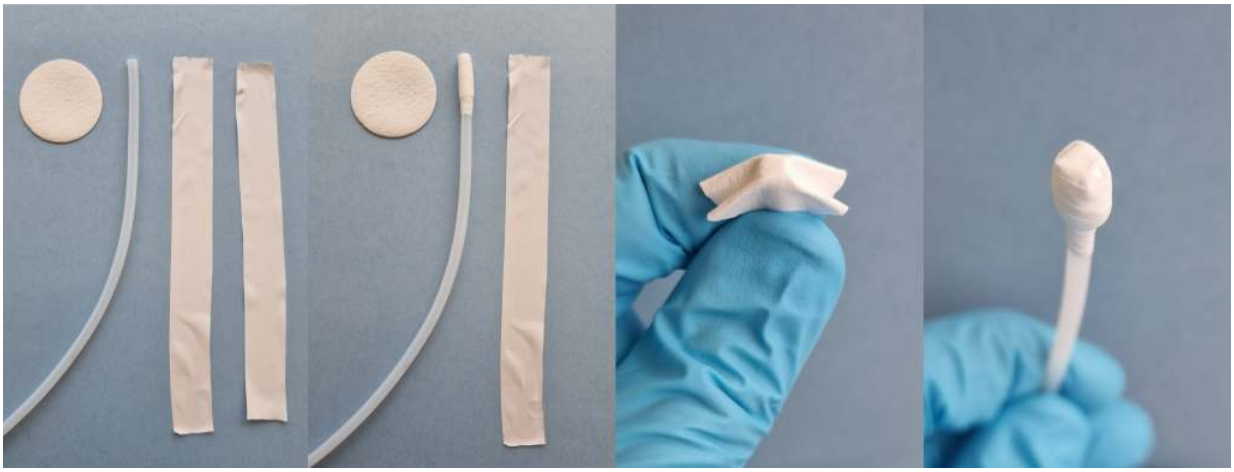
# 5.1: Cannula Filtration

Cannula filtrations are a convenient and routine method employed in Schlenk line chemistry. This method is recommended in situations where the crystallised or precipitated solids readily settles out of solution; fine solids or suspensions on the other hand can easily block the filter cannula. For large crystals, it may be sufficient to simply decant off the supernatant using a long needle and syringe, or to perform a standard cannula transfer.

**Preparing the Filter Cannula**

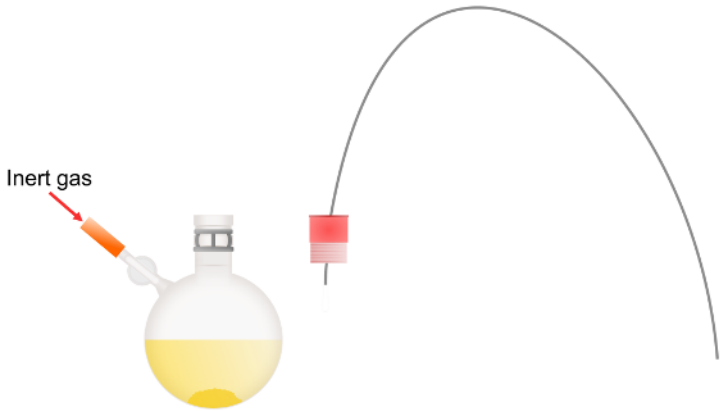
Cannulas used for filtrations should contain one end which is flat or suitably modified to allow a small glass microfibre filter to be secured to the cannula using PTFE tape. Firstly, a ~10 cm length of PTFE tape is wrapped around a flat-ended cannula, making sure to not obstruct the hole. A 125 mm diameter glass microfibre filter is then carefully folded around the cannula, ensuring that the filter remains flat and flush with the end of the cannula. Finally, a second ~10 cm length of PTFE tape is wrapped around the filter to secure it to the cannula. The filter cannula is then stored in an oven prior to use. It is also possible to purchase premade filter attachments (available in different sizes and porosities) that connect to the cannula and can be secured in place using PTFE tape.



Preparing a filter cannula.

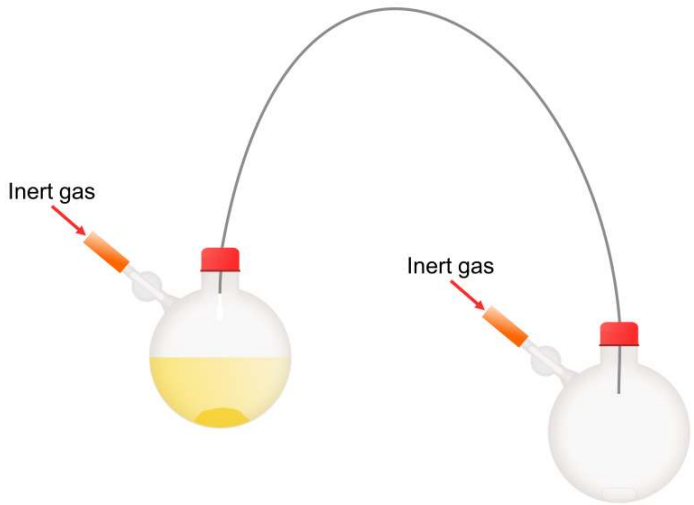
**Performing a Cannula Filtration**

**Step 1:** Pierce the sharp end of the prepared filter cannula through the bottom of a rubber septum and pull it along so that the end of the cannula containing the filter is close to the bottom of the septum.




Inserting the filter cannula and septum into the Schlenk flask.

**Step 2:** Replace the ground glass stopper with the rubber septum/filter cannula under a positive pressure of inert gas. Purge the cannula for 15-30 seconds before inserting the sharp end of the cannula into a receiving Schlenk flask that has previously been cycled onto the Schlenk line.




Setup prior to filtration.

**Step 3:** Insert a bleed needle into the septum on the receiving flask and close the stopcock to establish a pressure differential. Lower the filter cannula down into the supernatant. For cannula filtrations, it may be necessary to increase the inert gas over-pressure and raise the height of the transfer flask to help speed up the filtration.

Performing the cannula filtration.

**Step 4:** Once the filtration is complete, the filter cannula is raised above the solids to allow any remaining filtrate to pass through the cannula. If necessary, it is possible to add additional solvent to the transfer flask to wash or extract the solids (by syringe or cannula transfer) and repeat the filtration.

Completed cannula filtration.

**Step 5:** The stopcock on the receiving flask is opened to inert gas and the bleed needle is removed from the septum. The cannula is removed from the septum of the receiving flask and then from the transfer flask along with the septum and replaced with a greased stopper.

Transfer and receiving flasks after filtration.

Clean the cannula immediately after use to prevent blocking. The solids can now be dried under vacuum (if desired) or discarded/quenched, and the filtrate is ready for further manipulations.

This technique is also amenable to being performed at low temperatures by simply submerging the transfer flask in a suitable cooling bath. Hot filtrations using cannula filters may lead to undesirable crystallisation or precipitation of solids within the cannula or on the filter paper and should therefore be used cautiously.

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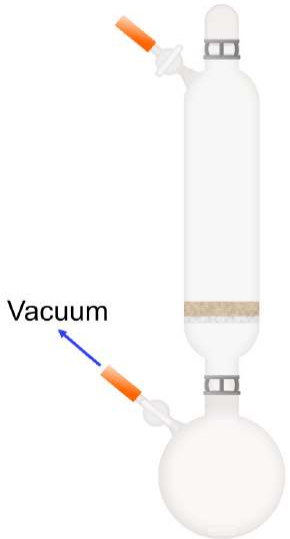
# 5.2: Filtration through Celite

Filtrations through Celite are commonly employed in synthetic inorganic chemistry to remove fine solids such as metal salts from reaction mixtures. They are particularly useful for large scale reactions where a standard cannula filtration would take too long.

## Step 1

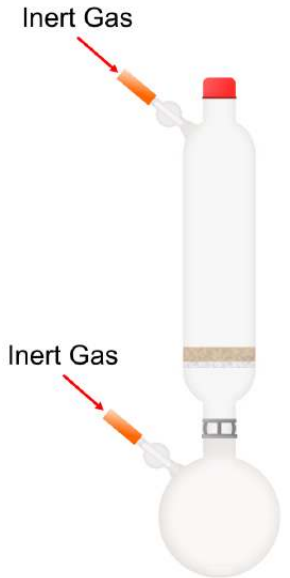
A Schlenk flask equipped with a stir bar, glass filter frit with a layer of Celite (pre-dried in an oven), and Schlenk cap is greased, assembled and cycled onto the Schlenk line. It is recommended to leave the assembly under vacuum for 30-60 minutes for the first cycle to ensure the frit and Celite is thoroughly dried.




Cycling a filter frit and receiving flask onto the Schlenk line.

## Step 2

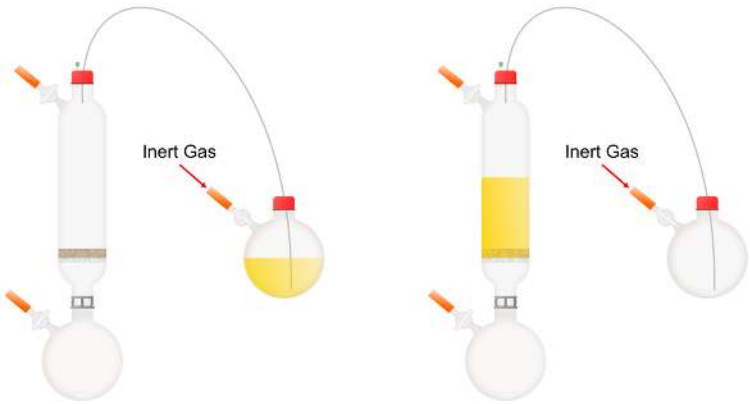
Once cycled onto the Schlenk line, the apparatus is **carefully** backfilled with inert gas – pulling vacuum from the receiving Schlenk flask sidearm and then backfilling from the filter frit sidearm (ensuring that the other stopcock is closed) is recommended to avoid disruption of the Celite layer. The Schlenk cap is replaced with a rubber septum under a positive pressure of inert gas.




Filter assembly under inert gas.

## Step 3

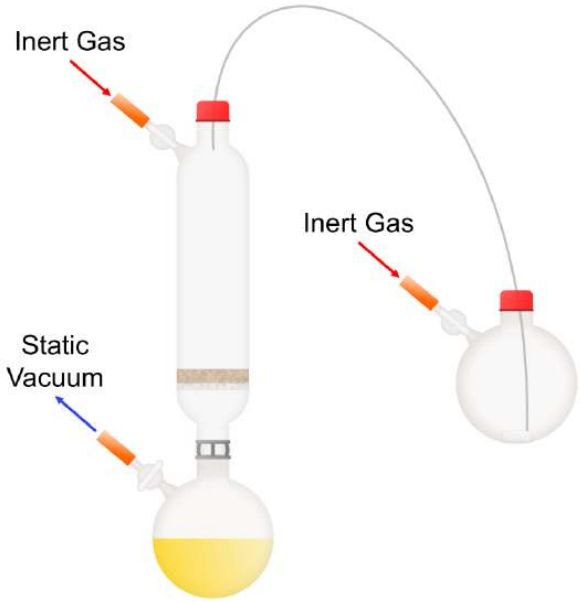
The reaction mixture to be filtered is transferred to the filter frit *via* cannula transfer.




Cannula transferring the suspension into the filter frit.

## Step 4

Once the reaction mixture has been transferred to the filter frit, the bleed needle is removed but the cannula is kept in place since this facilitates any further washing or extraction steps. If the filter frit does not have an in-built sidearm, then it is essential to keep the cannula in place to provide a constant feed of inert gas. A pressure differential is required to initiate the filtration, which can be achieved by establishing a static vacuum in the Schlenk line hosing attached to the receiving flask. This may have to be periodically refreshed to speed up the filtration.




Completed filtration through Celite.

## Step 5

Once the filtration is complete, additional solvent can be transferred either into the original reaction flask or directly into the filter frit to further extract the solids. The receiving flask is then put back under inert gas, the cannula is removed and cleaned, and the filter frit is replaced with a greased glass stopper ready for further manipulations.

**Hints and tips**

- For reactions mixtures that are too thick to be transferred by cannula, it may be necessary to attach the filter stick and receiving flask directly to the transfer flask and carefully turn the set-up 180° to pour the reaction mixture through the glass frit. A small plug of oven-dried glass wool can be placed above the Celite in the filter stick to prevent it from moving.

- The same technique can also be used with $SiO_2$ or $Al_2O_3$ as a means of removing polar impurities.

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# SECTION OVERVIEW

## 6: NMR Preparation

6.1: Preparing NMR Samples on a Schlenk Line

6.2: Removing Solvent from NMR tubes

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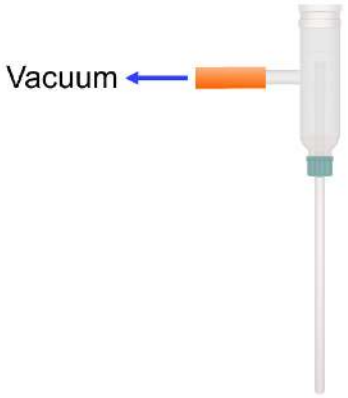
# 6.1: Preparing NMR Samples on a Schlenk Line

Air- and moisture-sensitive samples to be analysed by NMR spectroscopy can be readily prepared using Schlenk line techniques. NMR tubes used for sensitive samples typically contain a screw-thread Teflon tap (i.e. J. Young's) which ensures an airtight seal to prevent contamination by atmospheric air or moisture. Although it is often more convenient to prepare NMR samples inside of a glovebox, it may be desirable to add the deuterated solvent on a Schlenk line (to minimise solvent use in a glovebox or prevent contamination by other volatiles in the glovebox atmosphere) or to directly take an aliquot of the reaction mixture.

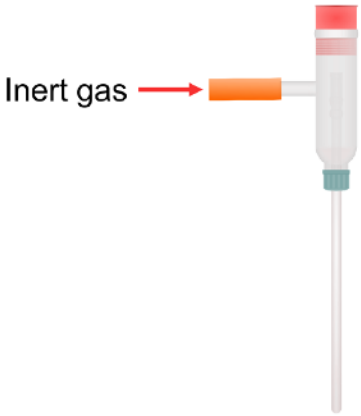
**Preparing an NMR sample to analyse a reaction mixture**

**Step 1:** Cycle a J. Young's NMR tube onto the Schlenk line with a minimum of three vacuum/inert gas cycles using an appropriate NMR tube adapter. A thermometer adapter or a rubber septum with a 5 mm hole at the bottom of the adapter provide a sufficiently air-tight seal.



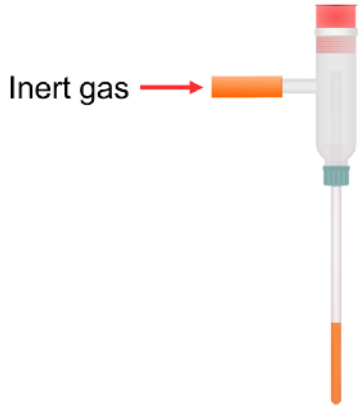

Cycling a J. Young's NMR tube onto the Schlenk line.

**Step 2:** Replace the glass stopper with a rubber septum under a positive pressure of inert gas. Insert a bleed needle for 5-15 seconds to purge out any air that enters the system.



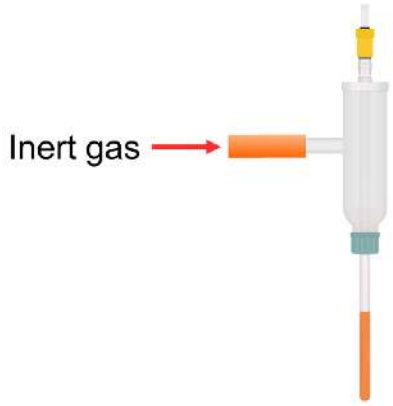

Fitting the rubber septum to the NMR tube adapter.

**Step 3:** Add an aliquot of the reaction mixture (approximately 0.5-0.6 mL) to the NMR tube *via* cannula transfer (using a suitably gauged cannula) or with a syringe and needle.




Adding an aliquot of the crude reaction mixture.

**Step 4:** Under a positive pressure of inert gas, secure the Teflon valve onto the J. Young's NMR tube. Close the stopcock or Teflon tap supplying the inert gas from the Schlenk line into the NMR tube adapter.

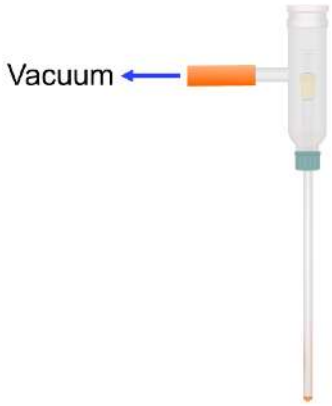



Sealing the NMR tube under inert gas.

The *protio*-solvent in the NMR tube can now be removed under vacuum and the sample later redissolved in an appropriate deuterated solvent. Alternatively, the NMR sample can be run directly provided that the NMR spectrometer is setup for *protio*-solvents – this is particularly useful for heteronuclei such as $^{31}P$ NMR spectroscopy.

**Adding solvent or liquids to a sealed J. Young's NMR tube on the Schlenk line**

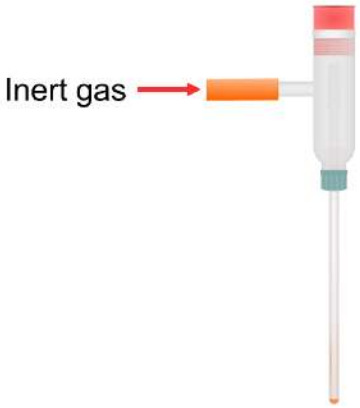
**Step 1:** Cycle the sealed J. Young's NMR tube onto the Schlenk line using an appropriate adapter.




Cycling a sealed J. Young's NMR tube onto the Schlenk line.

**Step 2:** Under a positive pressure of inert gas, remove the glass stopper, raise the NMR tube and remove the J. Young's Teflon valve. Quickly lower the NMR tube and seal the adapter with a rubber septum. Insert a bleed needle through the septum for 10-15

seconds to purge out any air that is introduced into the system.




Removing the Teflon valve under inert gas.

**Step 3:** Add the desired solvent or liquid reagent to the NMR tube *via* cannula transfer or needle and syringe.

**Step 4:** Under a positive pressure of inert gas, secure the Teflon valve onto the J. Young's NMR tube. Close the stopcock or Teflon tap supplying the inert gas from the Schlenk line into the NMR tube adapter.

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# 6.2: Removing Solvent from NMR tubes

Solvent can be removed directly from NMR tubes under vacuum by using the tapered glass adapters that are supplied with J. Young's NMR tubes.

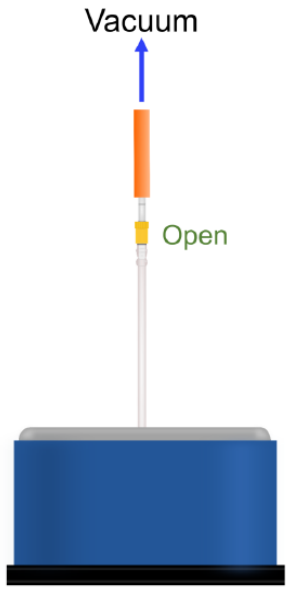
## Step 1

Attach the J. Young's NMR tube directly to the Schlenk line hosing using the tapered glass adapter and place under vacuum.

Attaching the NMR tube to the Schlenk line hosing.

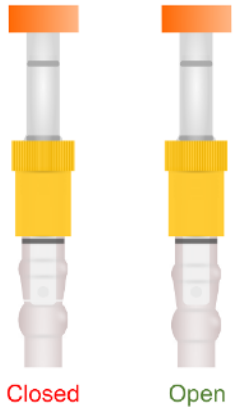
## Step 2

The contents of the NMR tube are cooled (or frozen) in a dry ice/acetone bath (-78 °C) or a Dewar of liquid nitrogen. Once the solvent is frozen, open the Teflon valve carefully by twisting it anti-clockwise, and evacuate the headspace for 30-60 seconds.




Evacuating the headspace of the NMR tube.

The J. Young's valves are hollow, allowing the NMR tube to be opened or closed to vacuum or inert gas whilst connected to the Schlenk line hosing.




Closed and open positions of a J. Young's NMR valve.

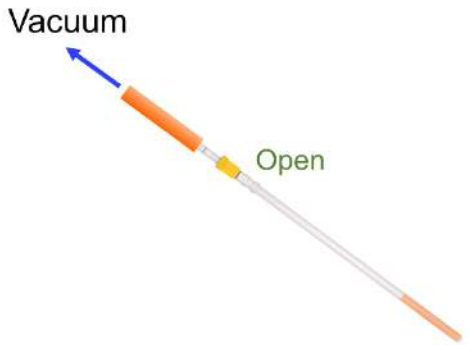
## Step 3

Close the Teflon valve and remove the NMR tube from the cooling bath. Carefully thaw the solvent from the top down to the bottom by rubbing the tube between your thumb and fingers. This degasses the solvent which helps prevent bumping whilst under dynamic vacuum.

Thawing the contents of the NMR tube under static vacuum.

## Step 4

Repeat step 2 then remove the NMR tube from the cooling bath and thaw the solvent with the Teflon valve open. As the NMR tube warms to room temperature, the solvent will slowly evaporate. Holding the NMR tube at an angle with gentle agitation helps to increase the surface area to facilitate evaporation of the solvent. It may be necessary to close the Teflon tap and repeat this process if the solvent removal becomes uncontrolled.




Removing solvent from an NMR tube under dynamic vacuum.

## Step 5

Keep the NMR tube under vacuum for 5 or so minutes to remove all traces of solvent and volatile material. Backfill the NMR tube with inert gas and then close the Teflon valve. The NMR tube is now ready to transferred into a glovebox or cycled back onto the Schlenk line for further manipulations.

NMR tube after removal of solvent.

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# SECTION OVERVIEW

## 7: Distillations

7.1: Static Vacuum Distillation

7.2: Dynamic Vacuum Distillation

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# 7.1: Static Vacuum Distillation

Static vacuum distillations (also called vacuum transfers) are frequently employed for the purification and/or drying of small volumes of solvents or liquid reagents with relatively low boiling points (<150 °C). Static vacuum distillations often employ custom-made greaseless vessels and distillation bridges which prevents grease contamination in the purified distillate.

## Step 1

An oven-dried Teflon tapped ampoule with a 90° side-arm, equipped with a stir bar, is cycled onto the Schlenk Line using a suitable O-ring or ground-glass adapter.

Cycling a Teflon tapped ampoule onto the Schlenk line.

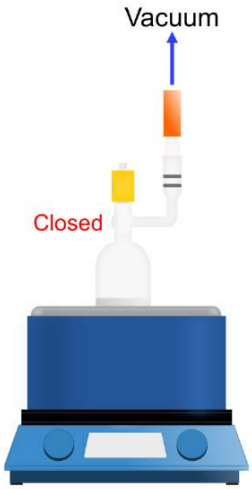
## Step 2

The ampoule is backfilled with inert gas and then the desiccant is added followed by the solvent (or liquid reagent); this can be introduced via syringe or cannula transfer, or simply poured into the flask with a funnel under a flow of inert gas. The contents are then stirred overnight under an inert gas atmosphere: the flask can be sealed or periodically opened to the Schlenk line to release $H_2$ build-up (from the reaction of the desiccant with water or protic impurities).

Drying the solvent or liquid reagent over a dessicant.

## Step 3

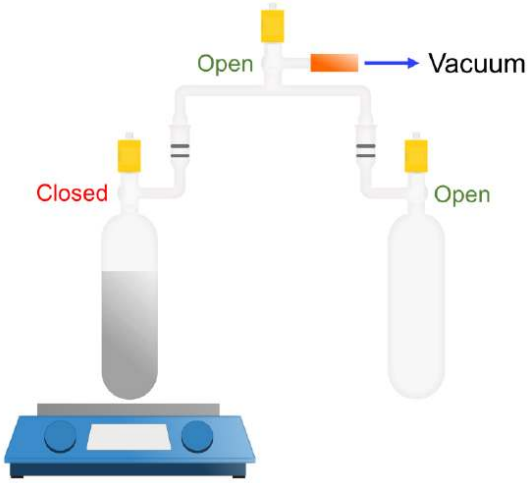
The Teflon tap is closed, and the ampoule is slowly submerged in a -78 °C dry-ice/acetone bath. Once completely frozen, the headspace is evacuated under dynamic vacuum for 5 minutes. The Teflon tap is then closed, and the ampoule is removed from the cooling bath to slowly thaw to room temperature. This corresponds to one freeze-pump-thaw cycle, which is then repeated once more.




Performing a freeze-pump-thaw to degas the contents of the ampoule.

## Step 4

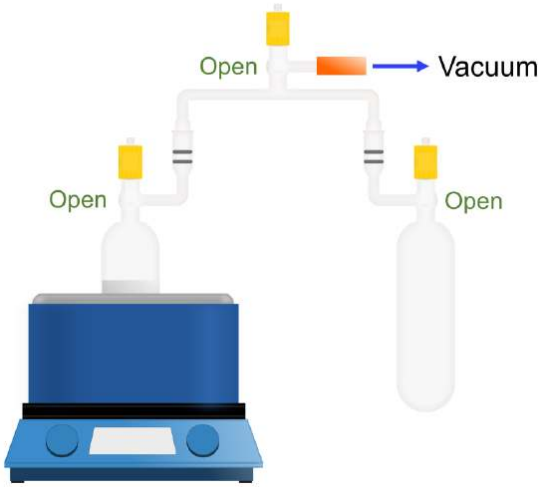
The ampoule is then connected to a greaseless distillation bridge (this may be built directly into the Schlenk or high vacuum line, or be a separate unit that can be connected to the Schlenk line hosing), and a second receiving ampoule is connected to the bridge and evacuated under vacuum. Oven-dried glass wool can be placed within the distillation bridge or ampoule side-arm to prevent solid desiccant from accidentally contaminating the purified distillate.




Connecting a receiving ampoule to the distillation bridge.

## Step 5

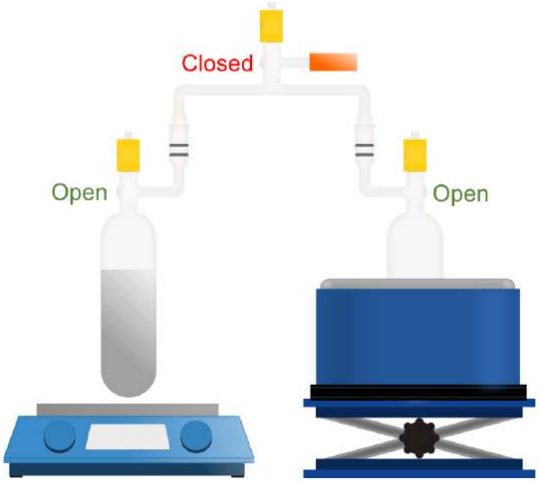
The ampoule containing the liquid is cooled to -78 °C using a dry-ice/acetone bath, and then evacuated under vacuum. Liquid nitrogen cooling baths should be avoided where possible for vacuum transfers as this presents a significant risk of liquid oxygen condensation. Performing this third freeze and pump cycle whilst attached to the distillation bridge ensures that the best possible vacuum is established within the distillation apparatus. A manometer is beneficial here to confirm a suitable vacuum is established prior to beginning the distillation.




Establishing the static vacuum.

## Step 6

The Teflon tap on the distillation bridge is closed, then the transfer ampoule is removed from the -78 °C cooling bath whilst the receiving ampoule is submerged in the -78 °C cooling bath . As the liquid slowly thaws, it will begin to evaporate under the reduced pressure and condense in the receiving flask at the lower temperature.

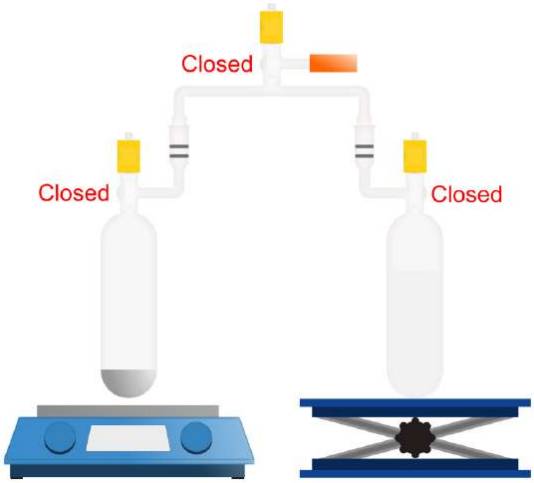



Performing a static vacuum distillation.

It may be necessary to refresh the static vacuum periodically by closing the Teflon tap on the transfer ampoule, then opening the Teflon tap on the distillation bridge to dynamic vacuum. After several minutes, or until the manometer reading is sufficiently low, the Teflon tap on the distillation bridge is closed and the Teflon tap on the transfer ampoule is slowly opened to the continue the distillation.

## Step 7

Once the distillation is complete, the Teflon taps on both ampoules are closed and the receiving flask is removed from the cooling bath. The liquid in the receiving flask is allowed to thaw before being backfilled with inert gas (depending on the setup this may need to be first disconnected from the distillation bridge and then cycled back onto the Schlenk line). The freshly distilled liquid can now be transferred to a suitable flask for storage. The transfer ampoule is also cycled onto the Schlenk line and backfilled with inert gas, and then quenched with iso-propanol/toluene to safely destroy the excess desiccant.




Completed static vacuum distillation.

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# 7.2: Dynamic Vacuum Distillation

Dynamic vacuum distillations are routinely employed to purify high-boiling liquids (> 150 °C) and some low-melting solids. This method is well suited for commercially available reagents or compounds prepared on a large scale in the laboratory in which the (often known) impurities are non-volatile and so remain behind after distillation.

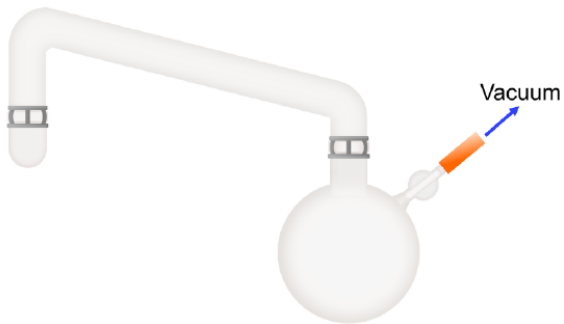
## Step 1

The impure material is transferred to a suitable Schlenk flask equipped with a magnetic stir bar. For commercially available reagents to be purified in bulk, this can be added to a Schlenk flask (that has already been cycled onto the Schlenk line) under a positive pressure of inert gas. For "in-house" prepared compounds, the crude material will typically remain in the Schlenk flask after removing the solvent and volatiles *in vacuo*.

Crude material in a Schlenk flask.

## Step 2

A Schlenk flask, distillation bridge and Schlenk cap are greased, assembled and cycled onto the Schlenk line. Note: The style of distillation bridge (single-piece) illustrated is designed specifically to purify high-boiling liquids under high-vacuum. This differs from a typical distillation setup with a distillation head, thermometer adapter, and water-cooled condenser.




Cycling the distillation bridge and receiving flask onto the Schlenk line.

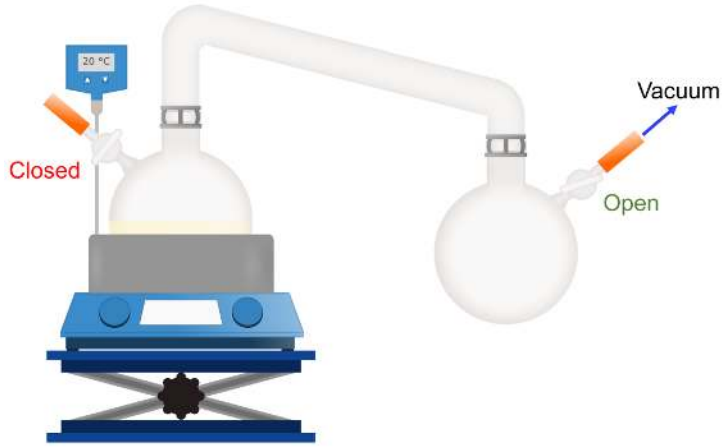
## Step 3

Once the receiving Schlenk flask and distillation bridge has been cycled onto the line and back-filled with inert gas, it is connected to the Schlenk flask containing the crude material. This may require a brief helping hand to remove clips and stoppers. Ensure that inert gas is flowing into both flasks during this process to minimise exposure to atmospheric air and moisture.

Setup prior to distillation.

## Step 4

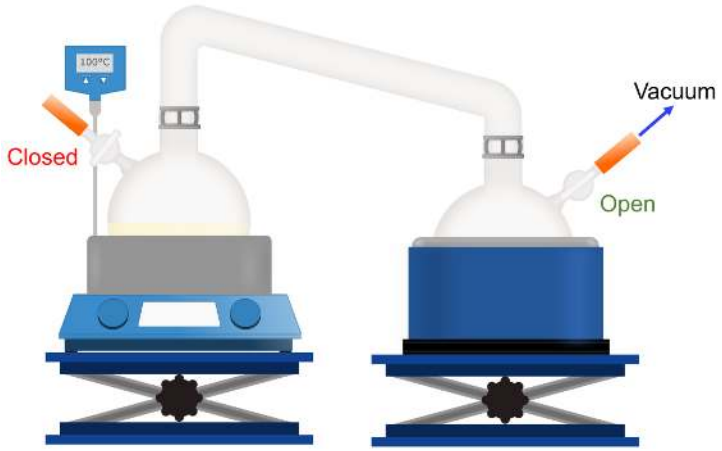
The stopcocks on both Schlenk flasks are closed and the distillation flask is lowered into a suitable heating mantle or oil bath. With stirring, the stopcock on the receiving Schlenk flask is slowly and carefully opened to vacuum. This serves to degas the crude material and remove any residual solvent or volatile impurities. *Note: The bulk material should be sufficiently high-boiling so that it does not evaporate at ambient temperatures whilst under vacuum. An external liquid nitrogen trap can be used between the receiving flask and Schlenk line to condense any volatile compounds.*




Evacuating the assembly prior to heating.

## Step 5

Once a good vacuum (*i.e.* low pressure) has been established within the distillation setup, and the crude material is fully degassed, the temperature on the heating mantle can slowly be increased. Since the crude material does not evaporate at ambient temperature, an ice bath is generally sufficient to cool the receiving flask and condense the distillate, however a dry-ice/acetone bath can also be used. During the distillation, it may be necessary to insulate the flask and part of the bridge with aluminium foil, or to briefly heat the glassware with a heat-gun.




Performing a dynamic vacuum distillation.

## Step 6

Once the distillation is complete, the stopcock is closed on the receiving flask. The heating mantle is lowered to allow the distillation flask to cool to ambient temperature, and the cooling bath is removed from the receiving flask to allow it to thaw or warm to ambient temperature.

Completed distillation.

## Step 7

When the distillation apparatus is at room temperature, the system is slowly backfilled with inert gas. If an external trap was used, it is necessary to disconnect this first and cycle the receiving flask back onto the Schlenk line.

## Step 8

Under a positive pressure of inert gas, the distillation bridge can be removed from the receiving flask and replaced with a clean, greased ground-glass stopper. The purified material can now be transferred to a suitable ampoule for storage *via* cannula transfer, or used directly for further manipulations.

**Hints and tips**

- An approximate distillation temperature can be calculated using the known boiling point of the compound (at ambient pressure) and the pressure within the Schlenk line (if a manometer is being used).
- For complex mixtures of species to be separated by vacuum distillation, a more elaborate setup containing a vigreux column, a thermometer adapter, and a ‘pig’ receiver is generally required to collect multiple fractions.

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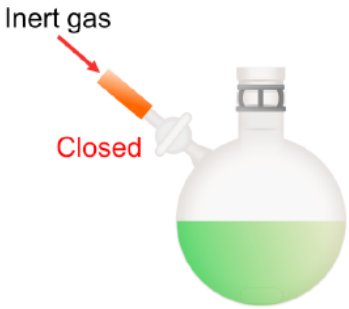
# 8: Freeze-Pump-Thaw

The freeze-pump-thaw (FPT) method is an effective way of degassing solvents, solutions, or liquid reagents.

## Step 1

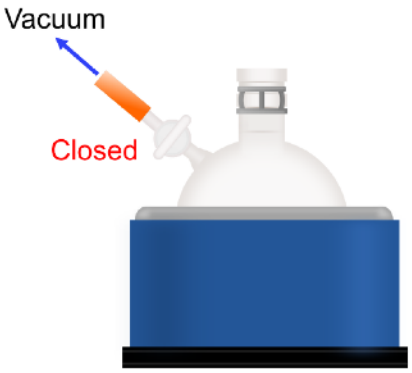
Close the stopcock on the Schlenk flask to seal the contents under inert gas. It is essential that the Schlenk flask is not open to inert gas whilst being frozen in liquid nitrogen (see Schlenk Line Safety for more information).




Sealing the Schlenk flask under inert gas.

## Step 2 – Freeze

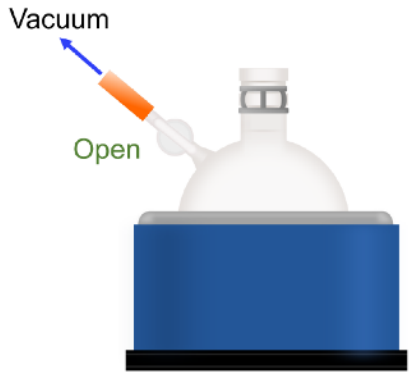
Cool the contents of the flask in a -78 °C dry ice/acetone bath. Cooling the solvent to low temperatures prevents or minimises the amount of solvent that evaporates when placed under dynamic vacuum. Liquid nitrogen can be used to completely freeze the contents of the flask, but this can lead to condensation of liquid oxygen if there is a leak present whilst the flask is under static vacuum. Liquid nitrogen should therefore only be used for more valuable liquid reagents or deuterated solvents.




Cooling or freezing the contents of the flask.

## Step 3 – Pump

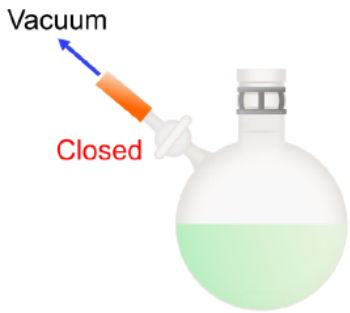
Once the contents of the Schlenk flask have cooled down or frozen, open the stopcock to evacuate the headspace. Keep under vacuum for 3-5 minutes whilst in the cooling bath.




Evacuating the headspace under dynamic vacuum.

## Step 4 – Thaw

Close the stopcock to seal the flask under static vacuum and remove from the cooling baths. As the liquid slowly warms or thaws, any dissolved gas will escape into the headspace of the flask. Avoid applying external heat (warm water bath or heat gun) to make this process faster, since this risks cracking the flask and causing an implosion.




Thawing the contents of the flask.

## Step 5

This process is repeated two more times for a total of three freeze-pump-thaw cycles – this ensures that all of the dissolved gas or any gas trapped within the frozen solid is removed. Once warmed back to room temperature after the final cycle, the flask can be backfilled with inert gas, or even a reactive gas such as $H_2$, CO or $CO_2$. It is essential to never backfill a flask with inert gas (nitrogen or argon) whilst frozen in liquid nitrogen since this can lead to condensation of the inert gas, which, if the flask is then sealed, may result in an explosion as the temperature increases.

## Other Degassing Methods

Other methods of degassing solvents include: (i) sparging with inert gas; and (ii) the boil-degas method. For sparging, inert gas is simply bubbled through the solvent for an appropriate amount of time to displace dissolved air and oxygen. For the boil-degas method, the solvent is placed under dynamic vacuum for an appropriate amount of time (with a suitable solvent trap) to remove dissolved gases. Both methods can be equally as effective as the freeze-pump-thaw method but do result in significant evaporative loss of the solvent, and therefore is only recommended for bulk organic solvents and should not be employed for degassing expensive deuterated solvents or volatile liquid reagents.

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# 9: Drying Solvents

A key aspect of Schlenk line chemistry is the purification and drying of organic solvents. Traditionally, this has been achieved through the use of solvent stills in which large volumes (>1 L) of solvent are refluxed over a suitable desiccant (typically alkali metals or metal hydrides) prior to distillation and collection. Solvent stills pose an enormous safety risk however, and therefore have been phased out of many synthetic laboratories, but are still occasionally used when necessary.

Solvent purification systems (SPS) are a safe and convenient solution for the purification and drying of organic solvents. Using inert gas pressure, the organic solvent is passed through a series of columns; one containing activated alumina to remove water and protic contaminants, and one containing a supported copper catalyst to remove trace oxygen. The solvent is first degassed by bubbling inert gas through the solvent reservoirs, however it may also be necessary to degas the solvent after collection using the freeze-pump-thaw method.

The use of activated molecular sieves alone has also been shown to be sufficient to dry a range of organic solvents. Water content can be quantitively measured using a Karl Fischer apparatus, or qualitatively assessed using the ketyl radical test.

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# 10: Removing Solvent

Schlenk lines allow solvents and other volatiles to be readily removed under vacuum without exposing the reaction vessel to atmospheric air and moisture. The low pressures offered by rotary vane vacuum pumps means that even high boiling solvents such as toluene can be removed at room temperature, which may be crucial for the isolation of temperature-sensitive compounds. When evaporating a small volume of solvent (>50 mL) or simply drying a compound under vacuum, the stopcock on the Schlenk flask is first closed, the stopcock on the Schlenk line is turned to vacuum, and then the stopcock on the Schlenk flask is slowly turned to vacuum, ensuring that the solution is adequately stirred to prevent bumping (i.e. rapid or uncontrolled bubbling/boiling).

A secondary external liquid nitrogen trap should be used when removing large volumes of solvents and is recommended when removing corrosive or reactive volatiles. Schlenk lines may also be equipped with a bui °C) can instead be employed over liquid nitrogen to help prevent blockages in the external trap.

## Step 1

Close the stopcock on the Schlenk flask to seal the contents under inert gas then disconnect it from the Schlenk line hosing. Attach the flask to an external solvent trap, then connect the external trap to the Schlenk line hosing and place under vacuum.

Attaching an external trap to the Schlenk flask.

## Step 2

After a few minutes of being evacuated, carefully lower the external trap into a Dewar of liquid nitrogen. Slowly and partially open the stopcock on the Schlenk flask, ensuring that the solution is stirring to prevent bumping. It may be necessary to open the tap fully or use a warm water bath to fully remove all solvent or volatiles.

Removing solvent under vacuum.

## Step 3

Once all of the solvent has been removed, close the stopcock on the Schlenk flask followed by the Teflon tap or stopcock connecting the external trap to the Schlenk line. Disconnect the Schlenk flask from the external trap and then cycle it back onto the Schlenk line, slowly opening the stopcock when backfilling with inert gas. Disconnect the external trap from the Schlenk line hosing and remove it from the Dewar of liquid nitrogen. Allow the collected solvent to thaw before discarding the contents into an appropriate waste container.

Schlenk flask after removal of solvent.

This same approach can also be used to partially concentrate solutions *in vacuo* prior to crystallisation.

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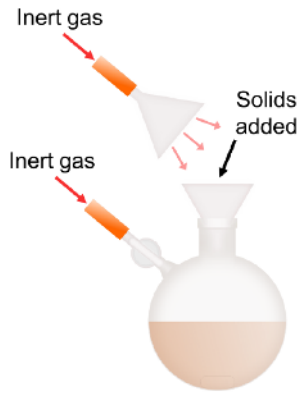
# 11: Addition of Solids

It may be more convenient (or even necessary) to add solid reagents directly to the reaction flask instead of first dissolving it in a separate Schlenk flask and performing a cannula transfer.

For less sensitive reactions involving air- and moisture-stable solids, it is often sufficient to simply add the solid through a powder funnel (to avoid it sticking to the greased ground glass joint) under a positive flow of inert gas. An inert gas blanket can also be employed – this is achieved by inserting the stem of an inverted funnel into the Schlenk line hosing and flushing inert gas over the Schlenk flask during the addition of the solid. This method works best when using argon as an inert gas since it is denser than both air and $N_2$. This method is particularly useful for the portion-wise addition of air- and moisture-stable solids.




*Using an inert gas blanket to add solids.*

The addition of sensitive solid reagents can be achieved by using a solid addition tube – these are similar to test tubes but typically contain a bend (~45°) and a ground glass male joint with can be sealed with a Schlenk cap, small round bottom flask or a rubber septum. Solid addition tubes may also contain a gas inlet sidearm with a ground glass stopcock or Teflon tap to control the inert gas atmosphere.

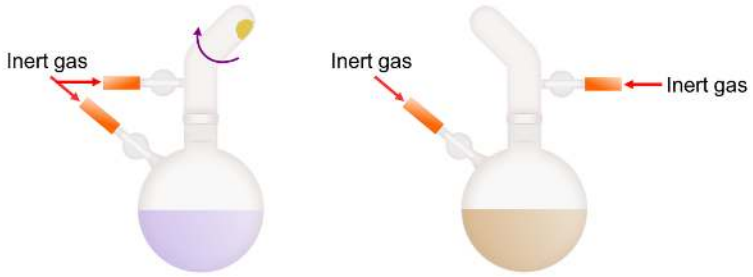
## Step 1

The solid reagent is first added to the solid addition tube inside a glovebox, and this can then be cycled onto the Schlenk line.

*Cycling the solid addition tube onto the Schlenk line.*

## Step 2

Under a positive flow of inert gas, the stopper on the Schlenk flask is removed and the Schlenk cap is quickly removed from the solid addition tube to allow it to be connected to the Schlenk flask. Rotating the solid addition tube and gently agitating it will encourage the solid to enter the reaction flask. This method is best suited for the addition of crystalline solids or salts that will not stick to the solid addition tube.




*Before and after addition of the solids.*

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# 12: Refluxing Under an Inert Atmosphere

## Method A

### Step 1

Cycle a reflux condenser onto the Schlenk line. Attach a gas inlet adapter to the top of the condenser, and a Schlenk cap or small round bottom flask to the bottom of the condenser, greasing the male joints and ensuring a uniform seal. Evacuate under vacuum.

Cycling a reflux condenser onto the Schlenk line.

### Step 2

Once the reflux condenser has been cycled onto the Schlenk line, backfill with inert gas and attach to the Schlenk flask under a positive pressure of inert gas.

Attaching the condenser to the Schlenk flask.

### Step 3

Close the stopcock supplying inert gas to the Schlenk flask and lower the flask into a heating mantle or oil bath. Connect the water tubing to the reflux condenser and begin heating.

Refluxing under an inert atmosphere.

The inert gas flow rate, which can be monitored by the frequency of bubbling, can be lowered once the desired temperature is reached, but should be increased once the flask cools back to room temperature to prevent oil suck back. It is generally recommended to not perform other reactions on the Schlenk line during a reflux to prevent contamination with solvent vapours.

## Method B

If the reflux is expected to liberate nasty by-products that may contaminate the Schlenk line, then it advised to use an external bubbler.

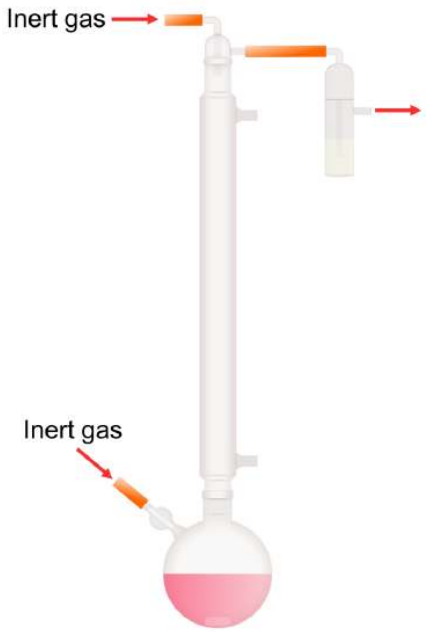
### Step 1

Assemble a reflux condenser and Dreschel head (gas inlet/outlet adapter), greasing the male joints and ensuring a uniform seal. Connect an external oil bubbler to the gas outlet and then attach the gas inlet to the Schlenk line hosing. Open to the inert gas and purge the glassware for 30-60 seconds.

Purging a reflux condenser equipped with an external bubbler.

### Step 2

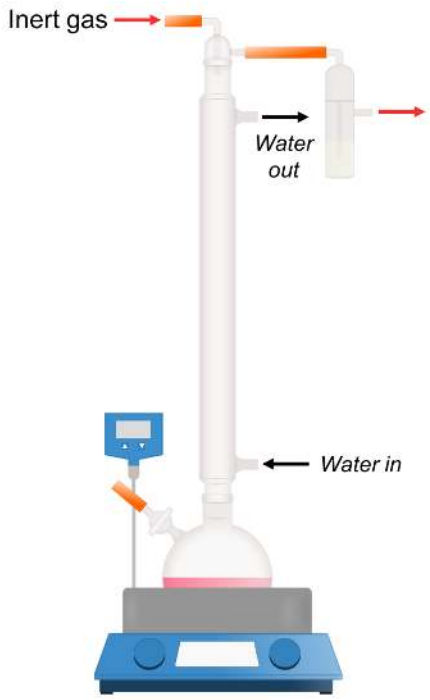
Attach the reflux condenser to the Schlenk flask under a positive pressure of inert gas. The external bubbler will begin bubbling at this point – adjust the gas flow so that it bubbles once every few seconds.




Attaching the condenser to the Schlenk flask.

### Step 3

Close the stopcock supplying inert gas to the Schlenk flask and lower the flask into a heating mantle or oil bath. Connect the water tubing to the reflux condenser and begin heating. It may be necessary to adjust the inert gas flow rate as the system approaches to the desired temperature.




Refluxing under an inert atmosphere with an external bubbler.

### Step 4

After the desired duration of heating, cool the reaction mixture to room temperature. Increase the inert gas flow rate whilst cooling to prevent oil suck-back. Once cooled to room temperature, open the stopcock on the Schlenk flask and replace the reflux condenser with a greased stopper under a positive pressure of inert gas.

## Method C

Reactions that require prolonged heating, and do not liberate gaseous by-products, can be heated directly in Teflon tapped ampoules without the need for a reflux condenser. Since this method does build up pressure within a sealed vessel, it is essential that the reaction vessel is suitable for such use. It is advised not to heat beyond the boiling point of the solvent unless specialist heavy-walled pressure vessels are used in tandem with a blast shield.

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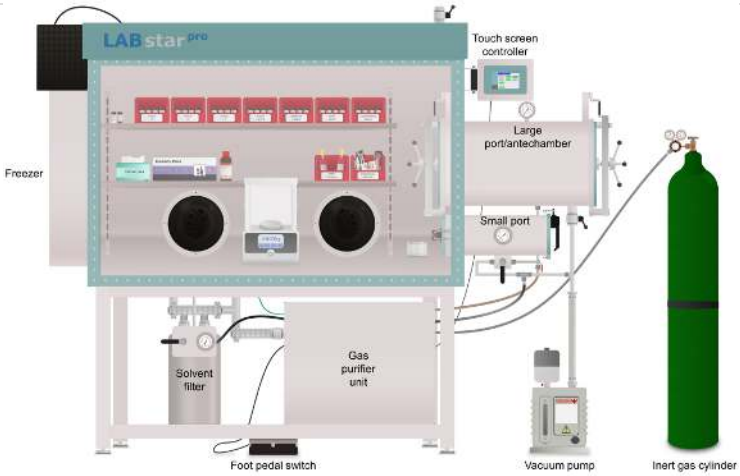
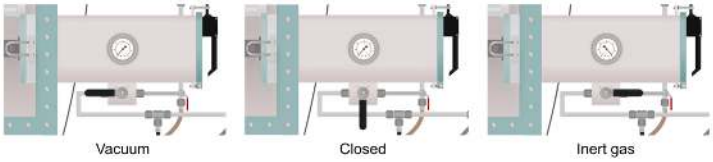
# 13: Gloveboxes




A typical glovebox.

Gloveboxes are large sealed containers that allow sensitive or hazardous materials to be handled under an inert atmosphere. In most chemistry laboratories, gloveboxes are primarily used for the storage of air and moisture sensitive solids, which would be difficult to manipulate using conventional Schlenk line techniques. It is common to prepare samples for NMR spectroscopy or to run test-scale reactions in the glovebox for convenience. Larger scale operations can also be performed inside the glovebox if suitable solvent filters and external vacuum pumps are employed. The inert gas (nitrogen or argon) is passed through a series of purifiers to remove oxygen and water, which maintains an atmosphere within the glovebox typically containing less than 0.1 ppm of oxygen and water.

Items are introduced into the glovebox through the small or large antechambers, and require three vacuum/inert gas cycles, similar to Schlenk line practices. It is recommended to have three 5 minute cycles for the small antechamber, and three 15-20 minute cycles for the large antechamber. The latter can be automated for some glovebox models. Glassware, including vials and pipettes, introduced into the glovebox should be dried in an oven prior to use.




Cycling items into the glovebox using the antechamber.

For a comprehensive guide and video on the principles of glovebox chemistry and standard operating procedure, consult the following JoVE.

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# Index


## C

cannulae

4.1: Cannula Transfer

Celite

5.2: Filtration through Celite

## S

sparging

3: Performing Sensitive Reactions without a Schlenk Line

# Glossary

**Sample Word 1** | Sample Definition 1

# Detailed Licensing

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## By Page


- The Schlenk Line Survival Guide (Borys) - *CC BY-NC 4.0*
  - Front Matter - *CC BY-NC 4.0*
    - TitlePage - *CC BY-NC 4.0*
    - InfoPage - *CC BY-NC 4.0*
    - Table of Contents - *Undeclared*
    - Licensing - *CC BY-NC 4.0*
  - Guides - *CC BY-NC 4.0*
    - 1: The Schlenk Line - *CC BY-NC 4.0*
    - 2: Cycling onto the Schlenk Line - *CC BY-NC 4.0*
    - 3: Performing Sensitive Reactions without a Schlenk Line - *CC BY-NC 4.0*
    - 4: Transferring Liquids - *CC BY-NC 4.0*
      - 4.1: Cannula Transfer - *CC BY-NC 4.0*
      - 4.2: Syringes and Sure Seals - *CC BY-NC 4.0*
    - 5: Inert Atmosphere Filtrations - *CC BY-NC 4.0*
      - 5.1: Cannula Filtration - *CC BY-NC 4.0*
      - 5.2: Filtration through Celite - *CC BY-NC 4.0*
    - 6: NMR Preparation - *CC BY-NC 4.0*
      - 6.1: Preparing NMR Samples on a Schlenk Line - *CC BY-NC 4.0*
      - 6.2: Removing Solvent from NMR tubes - *CC BY-NC 4.0*
    - 7: Distillations - *CC BY-NC 4.0*
      - 7.1: Static Vacuum Distillation - *CC BY-NC 4.0*
      - 7.2: Dynamic Vacuum Distillation - *CC BY-NC 4.0*
    - 8: Freeze-Pump-Thaw - *CC BY-NC 4.0*
    - 9: Drying Solvents - *CC BY-NC 4.0*
    - 10: Removing Solvent - *CC BY-NC 4.0*
    - 11: Addition of Solids - *CC BY-NC 4.0*
    - 12: Refluxing Under an Inert Atmosphere - *CC BY-NC 4.0*
    - 13: Gloveboxes - *CC BY-NC 4.0*
  - Back Matter - *CC BY-NC 4.0*
    - Index - *CC BY-NC 4.0*
    - Glossary - *CC BY-NC 4.0*
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