

## 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.

Inert gas



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.

Inert gas



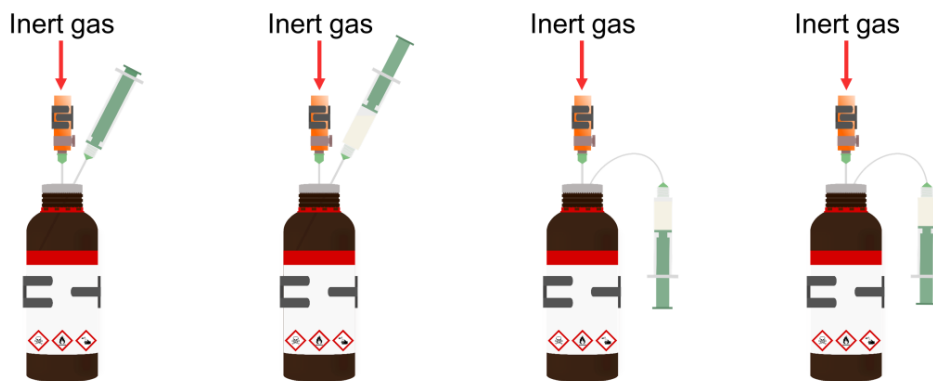
Inserting the gas inlet needle into the septum on the reagent bottle.

### 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

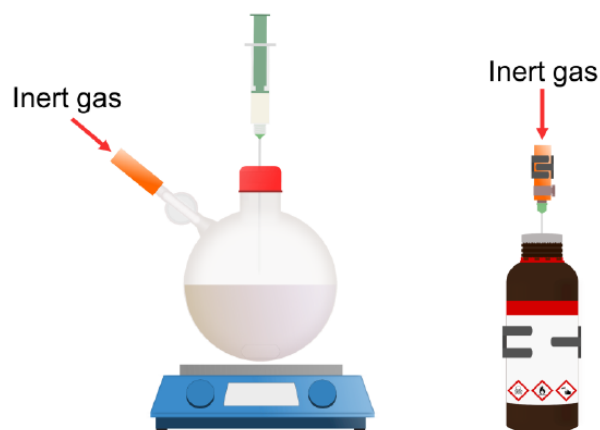
Insert the purged syringe and needle through the septum down into the reagent bottle. Slowly pull the plunger to withdraw the liquid into the syringe, removing slightly more liquid than required. With the needle still pierced through the septum, carefully bend the needle and turn the syringe vertically, 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  $n\text{BuLi}$ , 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 ( $>20\text{ mL}$ ) 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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