

Ball Lab Standard Operating Procedure 1

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Scope: This SOP details the procedure and safety considerations for the Air-Free Flask Technique.

Kind of SOP: Technique.

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Introduction and Purpose:

The Air-Free Flask Technique is a way of removing air from a flask and replacing it with a more desirable gas (e.g. an inert gas such as Nitrogen that will not react with materials).

Uses:

This technique is particularly useful for organic reactions that you want to take place in the presence of an inert gas so as to avoid reactions between the air and the organic materials.

General Procedure:

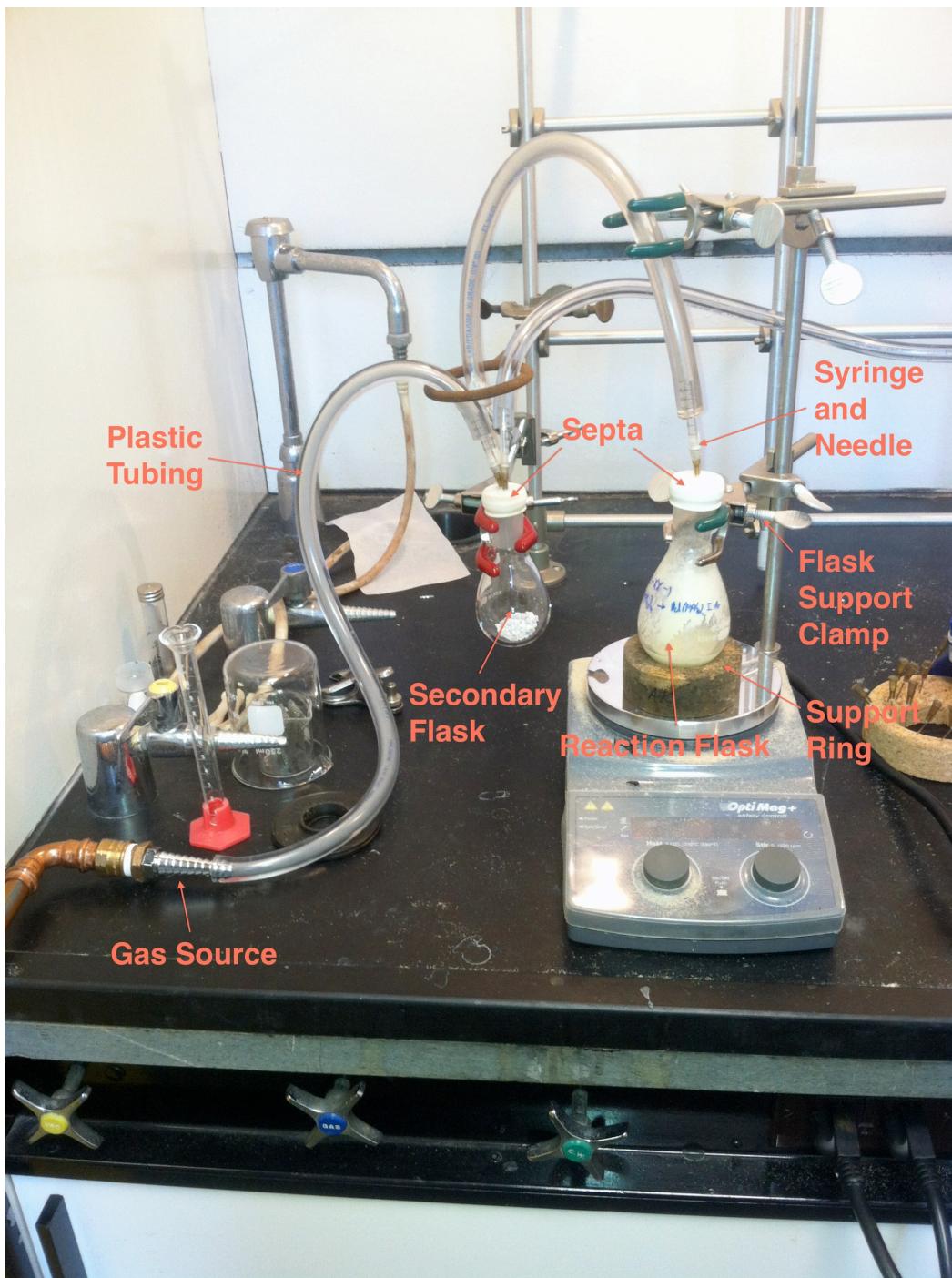
Necessary Materials:

- Appropriately-sized round-bottom flask for your reaction mixture (a good rule of thumb is for the total volume of the flask to be 3 times the volume of your reaction mixture)
- Secondary round-bottom flask
- 1 mL syringes
- Plastic tubing
- Needles

- Support ring for the round bottom flask
- Ring stand
- Support clamp for flask
- Gas source

Step-by-step setup: (Note: see picture on next page to see setup)

1. First you will want to setup your secondary round-bottom flask using a flask support clamp and ring stand. Arrange the secondary round-bottom flask close enough to the gas source so that the round-bottom and gas source can be connected with your plastic tubing.
2. Place drierite into your secondary round-bottom and then cap the round-bottom with a septum.
3. Now, to setup the tube connecting your secondary round-bottom flask and gas source, you will need a 1 mL syringe, a **capped** needle (needles should always be capped until you are ready to use them), and plastic tubing.
 - First, take the 1 mL syringe and remove the plunger. Then, cut off the end piece (the non-needle end) of the syringe (anywhere around 0.7 mL is fine).
 - Then, layer acetone onto the end of the plastic tubing. This should soften the tubing enough so that you can insert the end of the syringe into the plastic tubing. Insert the end of the syringe into the tubing. The syringe should now be securely lodged in the tubing.
 - Place a **capped** needle onto the needle hub of the syringe. Then, secure the needle to the syringe using parafilm. Now you are ready to connect the gas source to your secondary round-bottom!
4. Now, fasten the plastic tubing to the gas source. Then, uncap your needle and insert it into the septum covering the secondary round-bottom flask.
5. Next, once again using a ring stand and a clamp, arrange your reaction round-bottom flask close enough to the secondary round-bottom flask so that they can be connected with one of your plastic tubes. Then, cap your reaction round-bottom with a septum.
6. Now, take another one of your plastic tubes and, using the procedure detailed above, insert a syringe and needle into each end of the plastic tube.
7. Uncap the needle on one end of the tube and insert this needle into the septum covering the secondary round-bottom. Now you can turn on the gas so that the air is flushed out of the secondary round-bottom and it is filled with the gas.
8. Now you are going to connect your secondary round-bottom to your reaction round-bottom by inserting the needle at the other end of the tube into the septum covering the reaction round-bottom. However, before you do this, have a needle (capped until you are ready to use!) at the ready to insert into the reaction round-bottom's septum. This will serve as ventilation so that the gas can flow through the reaction round-bottom.
9. However, **it is important to note** that the ventilation needle must be inserted very quickly **AFTER** inserting the needle attached to the tube. This will allow the desired gas to flush the reaction round-bottom and flow through it.



Safety Considerations and Precautions:

1. ALWAYS keep ALL needles CAPPED until they are ready for use. At any time that the needle is exposed make sure to RECAP it (alternatively, you could stick the needles into a cork ring).

2. After turning the gas source on, make sure that all of your flasks have some sort of ventilation to avoid pressure building up.