

Techniques

HANDLING OF LIQUIDS, CALIBRATION OF PASTEUR PIPET

The various common operations that Organic chemists use regularly in the laboratory are called “Techniques.” They include extraction, distillation, recrystallization, various forms of chromatography, IR spectroscopy, etc. *The major objective of this first semester Organic laboratory course is to demonstrate most of these common techniques and to encourage the student to understand them and to become proficient in their use. These techniques will be used regularly in the second semester and in succeeding Organic laboratory experiences. The student will be expected to know when to apply them and to remember how to perform them. And, they often appear on lab quizzes. Here are some general and very important things to remember.*

Since you will be handling very small quantities of liquids, special care must be taken to minimize losses. Keep the desktop clean. Work over the glass surface to provide better visibility and to provide a clean and clear surface in case of a spill. Spills can be very deadly. Therefore, work carefully and plan each step. Avoid sudden movements. Support all containers holding liquids by placing them inside another slightly larger container such as a small beaker. This will hold the container erect.

Transfers of liquids will also require care. For any quantity of 5 mL or more, the use of a graduated cylinder will be adequate. However, for quantities below 5 mL, too much liquid would be lost on the sides of the graduated cylinder. It is therefore not appropriate.

Small quantities of liquids are never poured.
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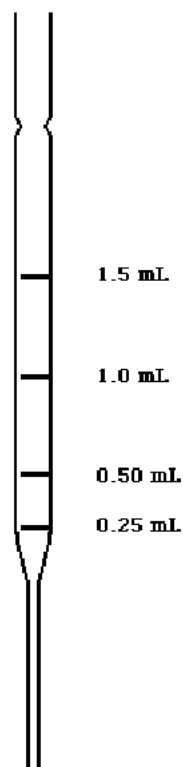
Small quantities of liquids (< 5 mL) will be measured and transferred using a variety of pipets. The simplest is the “Pasteur” pipet. This is a long glass tube, which has been drawn to a fine point at one end. The other end is connected to a rubber bulb to provide a means of suction to draw the liquid into the pipet. Since precision machines produce these, there is little variation of size among them. They can therefore be approximately “calibrated” (see Figure 1 below) and this calibration used to estimate the quantities of material in other pipets *of the same dimensions*.

These Pasteur pipets can also be used to filter small quantities of solid from a small volume of liquid. See the Pasteur Filter Pipets section below.

Calibration of Pasteur Pipets

Very often, you will need to transfer “about 0.5 mL” or “about 1 mL.” The easiest way to do this is to use a “calibrated” Pasteur pipet. The calibration procedure is only approximate but very easy:

- Lay a clean pipet over the following drawing.
- Clearly mark the positions of 0.5, 1.0, 1.5, and 2.0 mL on the new pipet with a crayon or other marking pen. Use this same drawing to “calibrate” other pipets throughout the semester as needed.



Whenever an approximate volume is needed, use your Pasteur pipet, calibrated by comparison to this diagram.

Similar approximate calibrations may be made for pipets of any size.

Figure 1: Approximate Calibration of a 5.75" Pasteur Pipet

PASTEUR FILTER PIPET

As mentioned earlier, when working with liquids on a small scale (*e.g.* 0.3-3.0 mL), it is *never* a good idea to pour. Too much material is lost by just wetting the glass. Furthermore, graduated cylinders are not at all accurate for such volumes. Syringes are excellent, but are expensive and easily broken.

The standard "disposable" or "Pasteur" pipet is excellent for transferring small volumes of non-volatile liquids such as aqueous solutions. However, most organic solvents have a significant vapor pressure at room temperature. This causes them to "dribble" from the pipet as a transfer is attempted.

To alleviate this source of sample loss, the standard pipet can be easily modified to produce an excellent means of transfer of these small quantities of organic liquids. It also provides an excellent method of filtering small quantities of liquid. We will call the modified pipet a "*Pasteur Filter Pipet.*"

All that is required is to place a very small piece of cotton in the tip of the pipet. This technique is sort of an "art" and requires a few trials to get it right. However, once the technique is learned, a new Pasteur Filter Pipet can be prepared in about 30 seconds.

Procedure

Take a very small piece of clean cotton — slightly larger than the diameter of the *tip* of the pipet — and roll it into a ball with your fingertips. Drop it into the larger end of the pipet.

Using about 18" of Copper wire, push the cotton ball down the narrow end of the pipet until it is *just* at the end. This will take some practice. Often the wire will bend as you push. This may mean the piece of cotton is too large. If the cotton moves *very* easily, you probably do not have a large enough piece and it will fall out when you go to use the pipet. Keep trying until you have *mastered* this very important technique.

When you think you have produced a 'good' filter pipet, attach it to a yellow pipet bulb and draw up some reagent grade acetone. Expel the acetone into a small beaker. Repeat this once or twice to test the pipet and to rinse the cotton. This will also help you to determine if the pipet is 'OK' (i.e., it draws the liquid at a reasonable rate, *and* the cotton does not move).

Draw air into the pipet to dry the residual acetone.

Make two or three Pasteur Filter Pipets for your drawer and rinse each with acetone and air as above. You will be using these pipets frequently.

Dispose of the acetone washings in the appropriate hazardous waste container in the hood.

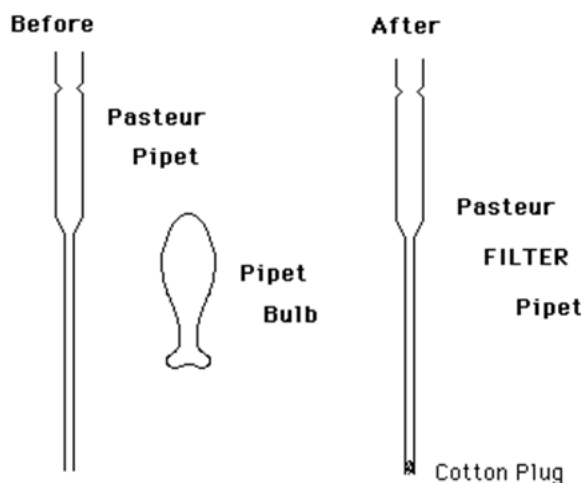


Figure 2: Pasteur Pipet

HEATING REACTIONS

During the course of this semester, there will be many occasions to heat a reaction. In every case, you will use a hot plate/magnetic stirrer to supply the heat. This avoids the use of the flame of a Bunsen burner, which is much more dangerous. Unless specifically directed, the use of a flame is *never* appropriate in this course.

The hot plate heat control needs to be turned slowly. If turned to 'HI' and allowed to remain there it will exceed 400°C in about 15-20 minutes. Also, once set to a high reading, the hot plate will

usually keep heating for some time, even after the control has been turned down. This “ballistic” heating is not a wise procedure in this lab. Time will generally be tight, so cooling down a set-up and reheating more carefully to get the desired temperature will waste a lot of precious time, since once the hot plate has been overheated it may take 30-45 minutes for it to cool down.

The best rule of thumb is to carefully set the heat and be patient. It may take 10-15 minutes to heat up on a lower setting, but the temperature will not rise ballistically and overshoot the desired temperature. (Remember, the hot plate is very slow to cool down once it has been heated.)

Many of the drawings in this book show the use of an aluminum heating block to transfer the heat of the hot plate to the reaction vial. However, the aluminum has a high heat capacity and stays hot long after the reaction. An aluminum block can be easily cooled to room temperature by picking it up *with a pair of crucible tongs* (**NOT** your fingers) and placing the block under running cold water for a minute or so. After drying with a towel, it can be reheated or replaced on the side shelf where the blocks are stored.

This same cooling procedure should be used for the aluminum collars that are used with the blocks.

CAUTION: NEVER touch a HOT aluminum block or collar.

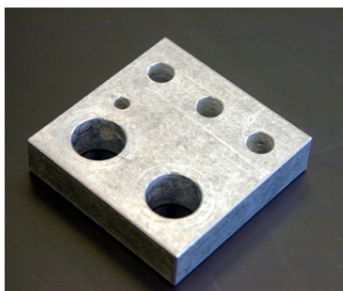


Figure 3: Aluminum Heating Block

MELTING POINT

Most solids have a characteristic temperature at which they change from the solid to the liquid state. This temperature is known as the “Melting Point” or mp. In fact, this is rarely a very sharp ‘point,’ but rather is usually a *range* of about 0.5 to 2 or 3°C. Nevertheless, the name “Melting Point” remains in common use rather than the more accurate “Melting Range.”

The presence of an impurity in a solid has the effect of depressing the melting point and increasing the temperature range over which the sample changes from solid to liquid, *i.e.*, melts. Conversely, the purer the sample, the sharper the melting point.

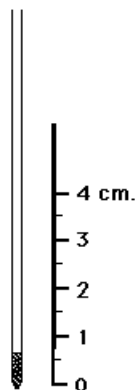


Figure 4: Loaded Melting Point Capillary

One of the best and easiest ways to estimate the purity of a solid sample is to take a melting point. All that is required is a few milligrams of the sample and a glass capillary tube. The sample is ground to a fine free flowing powder and placed in the capillary to a height of about 0.5-0.7 cm.

The powder is scraped into the tube by pushing the open end of the capillary through the freshly ground powder. To insure good packing of the solid, the loaded capillary is dropped down a 12-15" length of 5-6 mm id glass tubing and allowed to bounce on the bench top. This is repeated once or twice and then the capillary is placed into a *cold* ($< 50^{\circ}\text{C}$) 'Mel-Temp'TM device. The light is turned ON and the Voltage Control is set at about 35 (or 3.5 depending on the dial).

As the temperature rises, the sample is observed through the built-in magnifying device. The temperature at which the corners of the micro crystals begin to round is taken as the beginning of the 'Melting Point.' When the last trace of solid disappears, the end of the range of the 'Melting Point' is recorded. Thus, a typical 'Melting Point' might be recorded as mp 134.4-135.7°C. As soon as the sample has melted, the voltage control should be returned to 0, and the device allowed to cool.

The rate at which the sample is heated has a great effect on the observed mp. The temperature should not be rising more than about 1-2°/minute from 10° below the mp up through the mp. If the temperature is rising much faster it is not possible to obtain an accurate mp. If your first trial results in too fast a temperature rise, prepare a new sample in a clean glass capillary and start again.

Patience is a necessity.

When testing the purity of a solid that has been recrystallized, it is good practice to determine the mp of the solid *before and after* recrystallization. If the mp does not rise and become sharper (*i.e.*, extend over a smaller temperature range) after recrystallization, purification was not effected and a different recrystallizing solvent should be tried.