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Proper Titration Requires Skill

Dear Advice & Counsel,

We have a simple passivation line and also do chromating of aluminum. Our vendor of chemicals has provided analytical procedures for each processing solution, but we get inconsistent results. We are not chemists and have had little experience in doing titrations or even pipeting liquids. How are accurate titrations conducted?

Signed, P. Mohr,

Dear Ms. Mohr,

You've touched on a subject that I first learned about almost 40 years ago, when I started working at Scientific Control Labs. While it seems easy when you watch someone doing a titration, there is a significant amount of skill involved, and I hope to help you develop it.

First, without giving you a whole lecture on safety, be sure you wear eye and skin protection when doing a titration and preparing a sample for titration, as most likely you will be handling some highly corrosive and toxic chemicals.

Next, let's discuss methods of measuring volumes, because titrations are most accurate and repeatable, if we measure volumes of liquids accurately. Volumes can be measured using volumetric flasks, graduated cylinders, and pipets. Your analytical procedure will typically state what type of measuring device you should use. If the text states "add 5 mL of XYZ," you can feel secure in using a (relatively) crude measuring device such as a graduated cylinder. If the text states "pipet 5 mL of XYZ," we will need to use a pipet, which can be considerably more accurate.

Pipets come in varying designs. One is the Mohr pipet, which is graduated along its length to various volumes (see fig 1). It is tempting to use these, because they appear to be more versatile, allowing you to use one pipet to deliver 1, 2, or even 1.2 mL of liquid. However, Mohr pipets require you to measure a volume by allowing the liquid to flow from one gradation to another. Depending on your eyesight, skill level, and attention span, you may be able to deliver 5.0 mL of reagent XYZ, but more likely you will deliver more or less than 5 mL. Therefore, I do not recommend using these pipets for accurate measurement of volumes. They can be used, when accuracy is not critical.

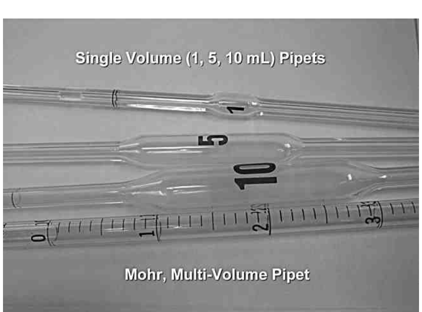

Fig. 1.

A far more accurate method of measuring volumes is the single volume pipet (also shown in fig. 1). These come in two styles; "to deliver" and "to contain." They also come in class "A" and class"B" accuracy levels. For most any plating lab, either class A or B will suffice. Always use a "to deliver" pipet, as it is impossible to blow out all of the liquid in a pipet, which will affect accuracy.


Fig. 2.

Fig. 2a.

The "to deliver" pipet will still have some liquid in the tip after it drains. This is normal. Do not shake or blow out this liquid. Do not use any pipet that has a damaged tip (fig. 2a), as this will affect its accuracy.

While old timers like me remember pipeting a sample by using the vacuum supplied by their mouth, this is a big NO-NO now. Safety concerns (and hygiene) dictate that a sample be drawn up a pipet using some form of vacuum tool instead of your mouth. There are numerous devices for sale that will work reasonably well. In our laboratory, we have found a rubber bulb with a plastic end for drawing the sample works extremely well and is efficient (see fig 2).

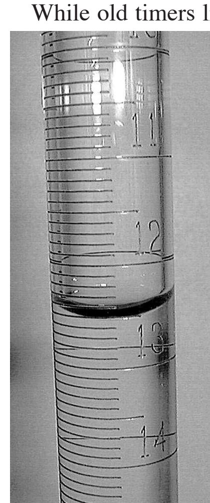
Fig. 3.

Since we will be working with liquids in glass tubes (pipets and burets), we need to cover the topic of a meniscus. When a liquid is placed into a narrow vessel, surface tension effects cause the liquid to deform into a curl called a meniscus (fig 3). Pipets and burets are designed so that the bottom of the meniscus produced by contained liquids is used for volume measurement.

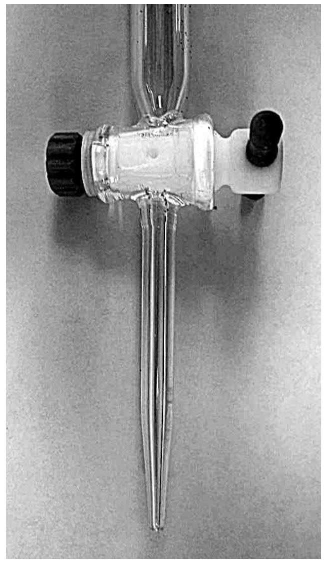

Fig. 4.

When a sample is pipetted from the sample container, the liquid is drawn past the meniscus line on the pipet. The vacuum device is removed and a (gloved) finger is placed over the end of the pipet. Enough pressure needs to be applied to prevent the liquid from leaving the pipet. Before the liquid is transferred to another vessel, the finger pressure on the pipet needs to be slightly loosened so that the liquid slowly drains back to its original container until the liquid reaches the meniscus line. When the bottom of the meniscus is lined up with the meniscus line, the finger pressure at the end of the pipet needs to be increased to stop the pipet from draining. If any partial drop of liquid hangs onto the pipet tip, touch the side of the sample container to remove this excess volume. The pipet will now deliver an exact volume to the next container.

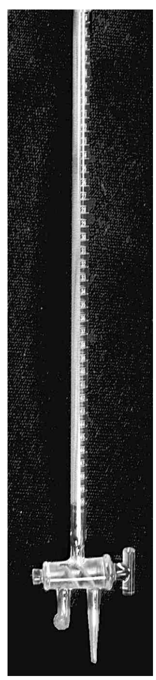

Fig. 5.

Once a sample is measured and prepared for titration, it is brought over to a buret stand to conduct the titration. We now need to discuss burets. They too come in numerous styles and designs. First, there is the "plain" buret (fig. 5). It is basically a graduated glass tube with a spigot (properly called a stopcock). You fill the buret by pouring the reagent from a beaker into the top of the buret (make sure the stopcock is in the "closed" position). This can be messy and may require you to use a funnel.

The stopcock on a buret is critical to a successful titration, yet is often ignored when choosing a buret. Avoid glass stopcocks (fig. 6). They require grease (stopcock grease which is a silicone based lubricant) to make a water tight seal. If the grease is not replaced frequently the glass may seize preventing you from turning the stopcock. A pair of pliers is usually applied to the frozen stopcock with disastrous results.

A far better choice for stopcock material is Teflon™ (fig 7.) . No grease is needed. Just make sure that the pressure knob applies enough pressure to the plastic washer (fig 4), so that the stopcock does not leak and yet turns smoothly with little effort.

Fig. 6.

Burets come in various volume capacities. Typically, you will want to use a buret that contains enough volume for the range of titrations you will be conducting. Large volume burets have gradations that allow volume measurements to 0.1 mL, while smaller volume burets often allow volume measurements to 0.01 or 0.02 mL. A good general purpose buret volume is 25 mL, with gradations down to 0.1 mL.

In addition to plain burets, there are self zeroing burets (fig. 7). These are more "delicate" and expensive, but eliminate the need to set the buret to zero with the meniscus. Another even more expensive buret is the electronic version, which allows you to simply turn a knob to deliver reagent, and which reads the titration volume digitally. There are also automatic titrations systems which eliminate the skill of the chemist altogether, but we will not cover these here. More next month. ***P&SF***

Fig. 7.