

CH 2212

Practice titration exercise (From Dr. David Chesney)

Proper titration technique is essential for optimum accuracy in a volumetric determination. You should follow these steps each time you prepare for a titration.

How to recognize a dirty pipet/buret.

Fill your pipet or buret with distilled water. Allow to drain at the normal flow rate. If droplets of water are observed adhering to the inside glass walls, you will not get reproducible delivery of solutions. Clean by soaking in Micro reagent overnight.

Use of the buret.

To add solution to a buret, **always** remove the buret from the buret clamp and hold it so that the addition funnel is **no higher than your eyes**. This will prevent filling solution from the inevitable overflows from running down your arm or into your eyes. When full, the buret can be clamped back onto the support stand.

It is absolutely inconsequential whether or not the titration starts at 0.00 mL on your buret. It **is** imperative that you record the initial buret volume when beginning the titration. Do not assume that you always start at 0.00 mL. Sooner or later you won't.

Hints.

A) To save time, do the first titration in about 30 seconds. This will give you an idea ($\pm 10\%$) of the titrant volume required to reach the end point. For the next titration, the first 80-90 percent of the titrant can be added quickly. The last 10-20 percent (getting close to the end point) can then be added carefully to obtain an accurate determination of the end point.

NOTE: There are some qualifiers to the above statement:

1) Do you have enough sample to do this? Figure this out ahead of time. If you only have 100 mL of sample and need 25 mL of sample per titration, you're going to be in trouble. You can do an initial "rough" titration on a smaller volume of your sample, say, 10 mL. Remember to adjust your expected titration volume by a factor of 25/10 for your 25 mL sample.

2) If your samples have differing amounts of reagent in them (a good example is the standardization of NaOH with solid KHP) then you must adjust your expected titration volume according to the sample size. For example, if a 0.7000 g sample of KHP requires 30.00 mL of your NaOH titrant, then a 0.6000 g KHP sample can be expected to require $(0.6/0.7)*30.00$ mL of NaOH.

B) Be sure and rinse down the sides of the sample flask several times during the titration. If you accidentally get titrant on the flask walls, the titrant solution may not reach the sample.

C) **Always record both the initial and final volume of your titration to two decimal places with units (± 0.02 mL).** Record in your notebook such that the larger value (Final volume) is at the top and the smaller volume (Initial volume) is on the bottom. In this manner, you can easily subtract to find the difference (the volume actually delivered).

Example:

Final volume	34.56 mL
- <u>Initial volume</u>	<u>1.25 mL</u>
Difference	33.31 mL

D) Hold a piece of white paper with a colored mark on it as a background behind the buret to assist in accurate reading of the meniscus. Read the meniscus the same way each time you titrate to minimize systematic reading errors.

E) You can use a white sheet of paper under your sample flask to help determine the endpoint. A disk of filter paper works very well.

F) The "titration thief". This is a very old trick used by experienced chemists. The "thief" was actually an eyedropper. What they would do is take about 0.5 mL of the solution to be titrated out of the vessel with the eyedropper. They would then titrate the remaining solution rapidly to the endpoint, knowing that they might overtitrate a bit. The solution inside the "thief" would then be added to the vessel, which would change the indicator color back to that prior to the endpoint. Then they would carefully titrate the total vessel contents to the final endpoint.

Not much of a trick, really, except that you don't have eyedroppers available. However, the concept is the important thing. You can use a small beaker as the "thief". Put 1-2 mL of your solution to be titrated into the small beaker and proceed with the titration as above. Just don't forget to add the small volume back into the vessel before ending your titration!

G) Splitting drops. Often a volume smaller than a single drop is required at the endpoint of a titration. You "split" drops by carefully controlling the stopcock of your buret to allow a very small flow of titrant to collect on the buret tip (not enough to form a drop and fall). Then rinse the buret tip with distilled water from your wash bottle. This in effect adds less than a full drop of titrant to your vessel.

PRACTICE TITRATION: Standardization of NaOH and HCl solutions.

Weigh out triplicate 0.4 g portions of KHP into 250-mL Erlenmeyer flasks. Add about 30 mL of distilled water to dissolve.

Using a beaker, take about 100 mL of the NaOH stock solution labeled "0.05 F NaOH". Rinse and fill your buret.

Titrate each KHP sample to the phenolphthalein end point as shown by your instructor. **Pay attention to details and ask questions if necessary.** Do one rough and three "good" titrations. Calculate the molarity of the NaOH solution. Record your results in your notebook. Add your data to the class data tabulated on the blackboard.

Remove about 100 mL of HCl stock solution from the bottle labeled "0.05 F HCl". Pipet 25.00 mL of the solution into a 250 mL Erlenmeyer flask. Add 2 drops of phenolphthalein indicator.

Titrate the HCl sample to the phenolphthalein end point as shown by your instructor. **Pay attention to details and ask questions if necessary.** Do one rough and three "good" titrations. Calculate the molarity of the HCl solution. Record your results in your notebook. Add your data to the class data tabulated on the blackboard.