

NaOH Standardization and Titration of an Unknown Organic Acid

Overview: Methods for counting the number of molecules in a sample is a major emphasis of laboratory work. In this experiment we will use the method of titration to count the number of acid molecules in a solution.

Measuring mass is a relatively easy procedure to do in the lab (although a balance is expensive). Counting the number of particles requires more effort. Molecular counting can be done by setting an unknown amount of a substance equal to a known amount of substance. In the titration we will perform in lab this week, you will add OH^- ions to solution until they are equivalent to the number of H^+ ions in solution. The point at which this mole equivalency occurs is known as the equivalence point. An indicator in the solution will change color to signal that the equivalence has been reached (actually, the indicator responds to the slightest excess of OH^- ions). The color change in a titration is called the endpoint.

At the equivalence point of the titration, the moles of OH^- (base) are equivalent to the moles of H^+ (acid) in the sample. The moles of OH^- added to the solution from a buret are calculated from the concentration of the base (M_{OH}) and the volume of base (V_{OH}) added, as:

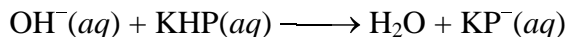
$$M_{\text{OH}^-} \left(\frac{\text{mol}}{\text{L}} \right) V_{\text{OH}^-} (\text{L}) = n_{\text{OH}^-} (\text{mol})$$

The lab goal is to determine the molar mass of an unknown monoprotic acid. The grams of acid are determined from weighing the acid and the moles are determined from the titration with NaOH. Because it is a monoprotic acid, the moles of acid are equal to the moles of OH^- at the endpoint of the titration.

$$n_{\text{OH}^-} (\text{mol}) = n_{\text{acid}} (\text{mol})$$

Standardization of NaOH:

Last week in lab you prepared a NaOH solution with an approximate concentration of 0.1 M. The first task is to determine the exact concentration of that solution by titrating the NaOH against a known amount of acid. The known amount of acid you will use comes from dissolving the solid acid potassium hydrogen phthalate (KHP, FW = 204.2212 g/mol) in 25 mL of H_2O . KHP is a large organic molecule, but can be viewed simply as a monoprotic acid. The titration of the KHP with NaOH can be expressed by the chemical reaction:



To standardize (determine the concentration of) your NaOH solution $\left(\frac{\text{mol}}{\text{L}} \right)$ you will measure volume of NaOH required to reach the titration endpoint. At the equivalence point the moles of NaOH are equivalent to the moles of KHP titrated.

$$n_{\text{NaOH}} (\text{mol}) = n_{\text{KHP}} (\text{mol}) .$$

Before you titrate a KHP sample, begin with a practice titration of 25 mL of a 0.1 M HCl solution with your NaOH. A practice titration is a good way to begin any titration. It lets you know the approximate concentration of your NaOH solution and the behavior of the indicator at the endpoint.

- Prepare a buret with your NaOH solution. Recall how you clean the buret first with DI water and then rinse the buret with about 10 mL of your NaOH solution. Drain the rinse NaOH into a large waste beaker at your lab bench. Finally, fill your buret with NaOH. Be sure to fill the tip of the buret before you use it and make sure there are no bubbles in the tip.
- With your graduated cylinder measure approximately 25 mL of 0.1M HCl. Record the exact volume you use.
- Add the HCl to an erlenmyer flask and add three drops of phenolphthalein indicator.
- Titrate the acid solution with the NaOH. Place the erlenmyer flask under the buret on a white sheet of paper (or towel). Remember to make an initial volume reading before you begin. You should need nearly the same volume of base as you have acid for this titration, because the molarities of each are similar.
- You can titrate quickly at first, but as you get near the endpoint slow your titrating. Small amounts of titrant can be added by quickly rotating the buret stopcock one revolution as you approach the end point. Even smaller quantities of a single drop can be added as you approach the end point. *Remember this is only practice. Don't spend a lot of time on this step.*
- After you have reached the endpoint, the lightest pink color that will persist for 20 seconds, show this solution to your TA. Anything past light pink indicates a solution with excess OH^- and you will be over counting the moles of acid in solution.
- After this practice titration, refill your buret, dump your titrated solution down the drain, and rinse your glassware with DI water.

Standardization of NaOH with KHP:

- Prepare a solution with a known number of moles of acid, by weighing exactly, approximately 0.5 grams of the solid acid KHP in a weigh boat. (Do not add KHP to the weigh boat while on the balance!) Add the weighed acid to a large erlenmyer flask. If any of the KHP remains on the weigh boat use your squirt bottle to rinse all of the KHP into the erlenmyer flask.
- Dissolve the acid in approximately 30 mL of water the exact amount does not matter. Add four drops of phenolphthalein indicator.
- Titrate the KHP acid solution with the NaOH. Place the erlenmyer flask under the buret on a white sheet of paper (or towel). Remember to make an initial volume reading before you begin. If not all of the KHP is dissolved, you can still begin the titration. Just make sure that all the KHP is dissolved before you reach the titration endpoint.
- The first titration is always the most difficult because you do not know how carefully you need to add the titrant. In this case, you should be able to add 15 mL of NaOH without passing the endpoint. As you are adding your initial amounts of NaOH,

notice the pink color of the indicator right as the NaOH enters the solution. As the pink color begins to persist, slow the titration down.

- Do not let the buret go below 50 mL during the titration. If you get close to 50 mL, stop the titration, record the volume of the buret and then refill the buret and continue to titrate.
- The end point of the titration occurs when the solution is the lightest pink color that will persist for 20 seconds. It is very likely that you will overshoot the endpoint on your first try. Don't let this worry you.
- Record the final volume on the buret.

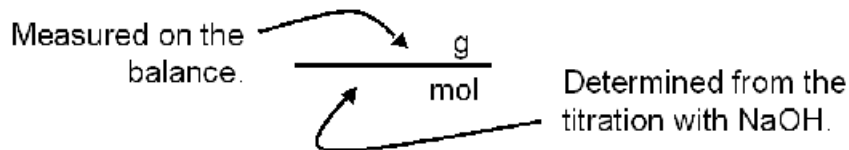
At the equivalence point, you have added the exact number of moles of OH^- as there were moles of acid initially. The moles of acid (monoprotic) you can determine from the mass of the acid and its molar mass (KHP = 204.2212 g/mol). Before you continue, calculate the approximate concentration of your NaOH solution.

Perform a second titration of KHP with NaOH.

- Weigh and dissolve another 0.5 g sample of KHP and dissolve it in a clean erlenmyer flask. The flask can be wet inside. Why can the Erlenmyer flask be wet when sample is added, but the buret must be carefully rinsed with the solution that will eventually be dispensed?
- Refill your buret with NaOH and titrate the new KHP sample to its pink endpoint.
- Do not forget to make an intial buret reading, add indicator and not go past 50 mL on the buret.
- After the titration, calculate the concentration (molarity) of your NaOH solution.
- Titrate a third KHP sample with the intent of obtaining a third measurement of your NaOH solution concentration.
- After titrating the third sample, calculate the NaOH concentration and compare all three calculations of the NaOH concentrations. If the three measurements of the concentration are the same to within ± 0.02 M, you may conclude that the concentration of your NaOH is the average of these three measurements. If you have accomplished this *precision* in your standardization of NaOH, continue to the titration on an unknown acid.
- If any of the measured NaOH concentrations are more than ± 0.02 M, you should perform a fourth standardization titration, using another 0.5 g sample of KHP.
- After examining the four concentration of NaOH, determine if any one concentration is suspect as either too large or too small. You can do this by inspection. However, there are statistical tests (Student T-tests) that will calculate if a data point is an outlier. If we ever team teach this course with Math140 we will use the student T-test at this point. For now, if three concentrations are within ± 0.02 M then average these three concentrations as the NaOH concentration.
- If you still don't have three measurements within ± 0.02 M of each other, perform a fifth standardization with a new sample of KHP.
- Obtain the NaOH concentration from the fifth titration and average all five measurements to obtain an average NaOH concentration.

Titration of an Unknown Acid to Determine Molar Mass:

The units of molar mass are g/mol. This intensive property is the ratio of two extensive properties, as is shown in the figure below.



To determine the molar mass of your unknown acid, you will perform the titration of the unknown acid in the same way you performed the titration of KHP.

- Obtain from your TA an unknown acid sample vial. Write your unknown number in your notebook. This sample vial contains two samples of your unknown acid.
- Weigh the vial and all its contents. Add half of the acid to a clean erlenmeyer flask. Reweigh the sample vial to determine how much acid you will be titrating in Trial # 1.

Note: There are only two samples of your unknown available. Exercise caution while titrating.

- Treating the unknown acid in the same way as the KHP sample, titrate to the endpoint and calculate the moles of NaOH require to reach the endpoint.
- From the mass and mole measurements of the unknown acid, determine the molar mass of the unknown acid.
- Clean your glassware and repeat the titration on the second sample of unknown acid.

To report the molar mass of the acid, take an average of the two molar mass measurements. Report the uncertainty as half of the difference between the two mass measurements (ex. if Trial # 1 gives a mass of 240 g/mol and Tiral # 2 gives 256 g/mol the average should be reported as 248 ± 8 g/mol).

Before you leave the lab, clean your buret with distilled water then place it upside down in the buret clamp with the stopcock open. All solutions can be placed down the sink. Return your unknown vial to your TA. Finally, turn in your blue notebook pages.