

Lab 5: Titration of an unknown acid

Objective: Determine the concentration of an unknown monoprotic acid by titration, the process that matches the number of moles of base with the number of moles of acid.

Skills:

- Set up a single-buret titration experiment
- Use an indicator to monitor the progress of the titration
- Determine the concentration of an acid to a high precision (standardization)

Background: From a previous lab, you learned that an acid-base neutralization is a type of double-replacement reaction, where binary compounds switch cations (or anions). In an acid-base neutralization, an acid (which contains an H^+ cation) and a base (which contains an OH^- anion) react to make water and a dissolved salt.

A titration is a neutralization reaction in which the **moles of acid in the solution are exactly matched by the moles of base in the solution**; in other words, the reaction has achieved a neutral pH, which is neither acidic nor basic. Numerically, the neutral pH is 7. You will use an indicator, phenolphthalein, to monitor the pH of the reaction. A small amount of this indicator will be added to the reaction flask. Due to the indicator, when the flask contents are acidic, the solution will not have a color (transparent). When the flask contents are basic, the solution will be a deep magenta color. Neutrality is achieved when you have the faintest pink color in the solution.

In this experiment, you will titrate a known quantity of an unknown concentration monoprotic acid with a measured amount of a known concentration base.

“Monoprotic” refers to the fact that the acid will ionize only one hydrogen ion per acid molecule; hydrochloric acid (HCl) is a good example of such an acid. A diprotic acid releases two hydrogen ions per acid molecule; sulfuric acid (H_2SO_4) is an example.

Ultimately, due to your measurement of the volume of base needed to neutralize the acid sample, you will be able to calculate the concentration of the acid to four significant figures. Such a high degree of precision is said to **standardize** the acid.

Pre-lab: Read pages 391-393 on “Acid-base titrations” in the textbook. Complete the attached pre-lab by Thursday, February 26.

Materials needed:

- 50 mL buret with valve (cart)
- Two 125 mL Erlenmeyer flasks
- buret clamp (side cabinet)
- SEVERAL small beakers
- unknown acid sample (either “A” or “B” – on cart)
- phenolphthalein indicator (cart)
- 10 mL volumetric pipet (cart)
- funnel (cart)
- standardized NaOH solution (cart)

Safety issues: There will be acids and bases in this lab; please wear goggles and tie back long hair. In addition, there will be plenty of glassware, so be aware of your surroundings while you are working and while you are transporting glassware.

Remember: Read the buret correctly: the graduations run from 0 mL at the top of the buret to 50 mL near the bottom (note this is backwards from how a graduated cylinder's scale runs). Read the buret to the nearest **hundredth** of a milliliter.

Procedure: Select one of the two available acids, record the **letter** for the acid you chose on the lab 5 data sheet, and assemble the titration setup.

Prepare **two** samples for titration by this procedure:

(1) Pour about 40 mL of the acid into a beaker and, using the 10 mL volumetric pipet, transfer 10.00 mL of acid to each of two Erlenmeyer flasks.

(2) Add 2 drops of phenolphthalein to each flask. Swirl the flask to mix the phenolphthalein in (you will do this to mix the base in as well, so get used to it). The solution should still be colorless.

Prepare the buret as follows:

(1) Pour about 60 mL of the base into another beaker and record the concentration of the standardized base solution. "Standardized" means that the solution concentration has been measured to a high degree of precision. In this case, **record four or five significant figures** for the base concentration.

(2) Designate one of the beakers as a "waste" beaker (label it with tape). Rinse the buret with a few mL of titrant (the NaOH solution) with the stopcock closed and pour the rinse into the waste beaker. Set up the buret on the buret clamp and ring stand.

(3) Still with the stopcock **closed**, place a funnel in the buret and add the base solution carefully until the buret is filled up to between the 0 and 10 mL marks.

(4) With the waste beaker below the buret, open the stopcock to fill the tip, closing it as soon as the tip is filled. It is important that there be no air bubbles in the tip, as such bubbles leaving during the titration will affect the volume of base, and thus the molarity of the acid. If you cannot get all the bubbles out, get help from me.

(5) Once the tip is free of air, add more base to the buret to fill it back up to between the 0 and 10 mL marks. Wait a few minutes to let all the liquid run down the sides of the buret and record the **initial buret volume** for the first titration. **All buret volume measurements should be to the hundredths place.**

Perform the titrations as follows:

(1) Place one of the flasks of acid and indicator below the tip of the buret.

(2) Add base relatively quickly, 0.5 or 1 mL at a time with swirling until a pink or magenta color persists for a few seconds after adding the base. This means you are getting close to the endpoint and should add base more slowly. You want to reduce the amount you add to a few drops at a time, swirling each time to mix. As the color persistence time lengthens you should reduce the amount of base you add each time until you are adding one drop at a time. Stop after the first drop where the color change persists for a couple of minutes and record the **final buret volume** for the titration. Subtract the initial buret volume from the final buret volume and record the volume of base added. If you believe you overshot the endpoint, make a note of that too.

(3) Now you have an idea of how much base is required. Looking at the buret, determine if you have enough base left in it to titrate the second sample, with a few

extra mL leeway. If so, record the initial buret volume for the second sample. If not, add more base to the buret until you are sure there is enough for the second titration, let the liquid settle, and record the initial buret volume for the second sample.

(4) The second titration can go faster, because you know about how much base to add. Figure out what the new level would be if you added two mL less than the volume of base added on the previous trial. That is, take two mL from the volume of base added in the first trial, add that to the initial buret volume for the second trial and the result is the level of the buret to which you may safely go in adding base to the acid without risking passing the endpoint. After this addition, let the liquid settle, because you've added a lot of base. Swirl to mix, then begin adding a drop at a time with swirling until the color change persists for a couple of minutes. Record the final buret volume for the second trial.

(5) Calculate the volume of base added for the second trial.

Checking for **reproducibility**:

(1) Compare the base volumes for the two trials by taking their difference and dividing that by their average. Convert the decimal to a percent. If your percent is 0.5% or less, the agreement is good enough and another trial is not necessary. If the deviation is **more than 0.5%**, you should do another trial and compare the two closer values. Continue until you have two trials in agreement to 0.5%. Record the average volume of base for these two trials and the percent difference.

Calculating the **concentration** of the acid:

The acid is monoprotic, so it will react one to one with NaOH; therefore, the number of moles of base equals the number of moles of acid. For each solution, the number of moles of the substance is equal to the molarity of the solution times the volume of solution.

$$M_a V_a = M_b V_b$$

Rearranged to solve for M_a :

$$M_a = M_b V_b / V_a$$

Use the averaged base volume in this calculation to get the best value of the acid molarity and record it. Be careful about units and sig figs.

Analysis and Questions (Please use your own paper for the answers of these questions, but number them the same way):

1. Suppose, in the example above, the acid were **diprotic**. Recalculate the concentration for the acid. Show your work. Hint: after you determine what "diprotic" means, this calculation should involve the inclusion of only one other factor.
2. How would the calculation of the unknown acid concentration be affected if the buret tip was **not** filled when reading the initial volume? Be specific: would the calculated concentration have been **lower**, **higher** or **the same**? Show your reasoning!

Lab 5 Data Sheet

Acid unknown letter _____

Acid volume 10.00 mL

NaOH concentration _____

Trial	1	2	3 (if needed)
Initial buret volume (mL)			
Final buret volume (mL)			
Volume of base added (mL)			

Average base volume between two acceptable trials (units?) _____**Percent difference** in volume between the two acceptable trials _____

Unknown acid concentration (units? sig figs?) _____