

experiment 7

Determining an Unknown Concentration

LECTURE AND LAB SKILLS EMPHASIZED

- Understanding the concept of titration.
- Explaining the difference between analyte and standard solutions.
- Knowing the definition of equivalence point.
- Converting between pH and the concentration of H^+ .
- Calculating molarity.
- Using stoichiometry with a balanced chemical equation.
- Learning how to use a buret.





IN THE LAB

- Students will work in pairs.
- Record your procedure and original data in your lab notebook along with your calculations.
- Report data collected and subsequent calculations to www.chem21labs.com.
- *All equipment should be returned to the correct location after use.*

WASTE

- All solutions should be disposed in the acid–base waste container.

SAFETY

- Citric acid and NaOH solutions should be handled with care.
- Gloves and safety goggles are mandatory when anyone is performing an experiment in the lab.
- Wear long pants, closed-toed shoes, and shirts with sleeves. Clothing is expected to reduce the exposure of bare skin to potential chemical splashes.
- Always wash your hands before leaving the laboratory.

Additional information can be found at <https://wp.as.uky.edu/genchem/home/che-111/7-titration/>

In Experiment 6, you were faced with one of two of the most common problems chemists face: determining what a substance is. In this experiment, you will address the other common question: how much of a substance is present in a solution. In this **quantitative analysis**, you will be using titration to determine how much of an analyte is present in a solution.

Your company has been hired to assist a beverage company in determining how much citric acid is in their beverages. You will need to submit your report of your findings and your reasoning behind your determination to Chem21.

Titration

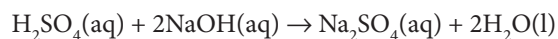
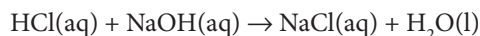
Acid–base titrations are used to determine the concentration of a solution. To do this, the concentration of another solution must be known very accurately. The **analyte**, or solution whose concentration is not known, is titrated using a **standard**

solution, or one whose concentration is known, until they react completely. The standard solution will react with the analyte, based on the stoichiometry of the balanced chemical equation. They are called neutralization titrations because the acid reacts with the base to produce a salt and water. The pH (a measure of the solution's acidity or basicity) of the resulting solution would be neutral. A typical titration curve for the strong acid hydrochloric acid with the strong base sodium hydroxide is shown in Figure 7.1.

At the **equivalence point** in an acid–base titration, the moles of H^+ will equal the moles of OH^- . For example, in the reaction between hydrochloric acid and sodium hydroxide shown below, there will be the same number of moles of H^+ as there are moles of OH^- at the equivalence point. Because there is a one-to-one ratio of HCl and NaOH in the balanced chemical equation, the moles of HCl will equal the moles of NaOH. For a diprotic acid (two hydrogens, such as H_2SO_4) and a base such



as NaOH, the moles of H^+ and OH^- will still be the same at the equivalence point, but the moles of acid and base will not be equal. There will be twice as many moles of NaOH because every mole of H_2SO_4 generates two protons so it takes two moles of NaOH to neutralize them.



The concentration of the acid will be determined by determining the moles of NaOH added at the equivalence point and using the stoichiometry of the balanced chemical equation. Solving a stoichiometry problem for an acid–base titration is similar to other stoichiometry problems you have done, only instead of looking at a reactant and a product, you are looking at two reactants. As long as you are starting with a balanced chemical equation, it is irrelevant whether you are looking at two reactants, two products, or a reactant and a product.

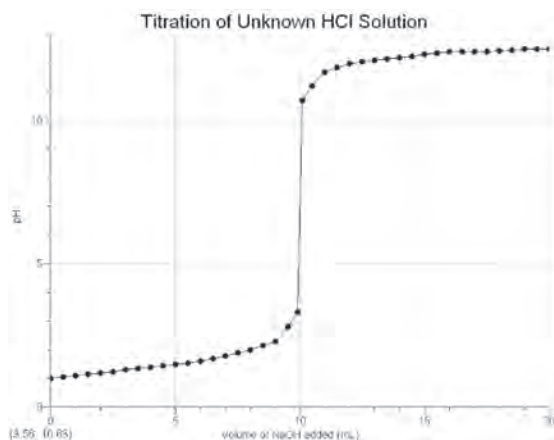


Figure 7.1. Typical titration curve.

To determine the equivalence point experimentally, we look at the point on the graph in which there is an inflection point. While we can eyeball it using the x vs. y graph produced on Chem21, we can determine a better estimate of the inflection point using the second derivative curve. To determine the equivalence point on the second derivative curve, draw a straight line from the maximum value to the minimum value (the topmost point

and the bottommost point) on the graph. The point at which they cross the x-axis is the equivalence point.

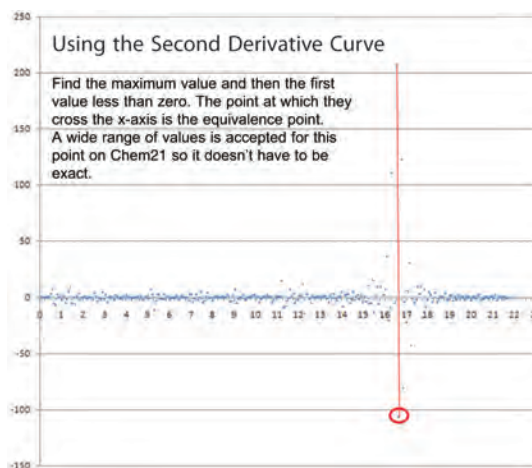
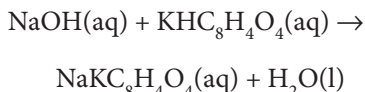


Figure 7.2. Finding the equivalence point with the second derivative graph.

Standardization of NaOH

In this experiment, you will be titrating a weak acid with sodium hydroxide to determine the concentration of the weak acid. While measuring out solid sodium hydroxide and determining its mass using an analytical balance is easy, it may not be accurate. Remember, our job is to be as accurate as possible. Sodium hydroxide is hygroscopic, meaning that it absorbs water. Unless the sodium hydroxide is stored in a completely dry atmosphere, it will absorb water from the air. The mass of sodium hydroxide will include the mass of water along with NaOH. Additionally, CO_2 from the air will dissolve in the solution and react to form carbonic acid. NaOH will react with the carbonic acid in a neutralization reaction which will lower the actual concentration of NaOH in the solution.

In order to determine the concentration of sodium hydroxide, it needs to be titrated against a **primary standard**. A primary standard is a pure compound that will not decompose at room temperature, can be easily dried, and will not readily absorb water. In this case, we will use potassium hydrogen phthalate, $\text{KHC}_8\text{H}_4\text{O}_4$ (204.22 g/mol), which is a monoprotic acid also known as KHP. Sodium hydroxide and KHP will react in a 1:1 relationship.



KHP can be dried before use and the exact mass being used can be determined using an analytical balance. The known mass of KHP is dissolved in water and then titrated with NaOH. The results of this titration will determine the exact concentration of the NaOH solution. Using this standardized solution of NaOH, an unknown concentration of an acid solution can be determined.

pH

pH is a value that can be determined experimentally to quantify the acidity of a solution. The pH scale is from 0 to 14. The pH is calculated according to the following equation

$$\text{pH} = -\log [\text{H}^+]$$

or the negative log of the H^+ concentration. The stronger the acid (i.e., more H^+ present in the solution) the lower the pH will be. The stronger the base (i.e., less H^+ present in the solution), the higher the pH will be. A neutral solution, such as distilled water, will have a pH of 7. For most aqueous solutions, we are typically talking about low concentrations of H^+ (such as 1.0×10^{-3}) so it is easier to say it has a pH of 3 than to say the H^+ concentration. Using a pH sensor to monitor the pH of the reaction will allow us to determine the equivalence point of the reaction. Based on the data from the equivalence point, we will be able to determine the concentration of the unknown solution.

Buret

During this experiment, you will use a buret. A buret is a piece of glassware that is used to measure the volume of liquid that is dispensed. Unlike most other glassware, the volume markings on a buret go from top to bottom. It is important to remember this when reading the buret volume. For example, the volume in the buret pictured in Figure 7.3 is 20.72 mL.

Whenever using glassware such as burets or graduated cylinders to measure the volume, you also need to estimate one decimal place beyond the markings. For example, if the buret has markings for 0.1 mL increments, then you need to estimate the next decimal place based on the location of the meniscus between two markings. It is assumed that the last decimal place is an estimate, so for the buret pictured in Figure 7.3, the 2 in the second decimal place is estimated because the meniscus is slightly below the 20.7 mL mark. Without the second decimal place, it would be assumed that the first decimal place is an estimate when in fact it is known. If the meniscus appears to be exactly on the marking, then you estimate the next decimal place to be zero and include that in your value (i.e., 3.20 mL). Without the second decimal place, the value could be anywhere from 3.20 to 3.29 mL. By estimating the one additional decimal place, you have one additional significant figure to carry through your calculations which results in more precise results.

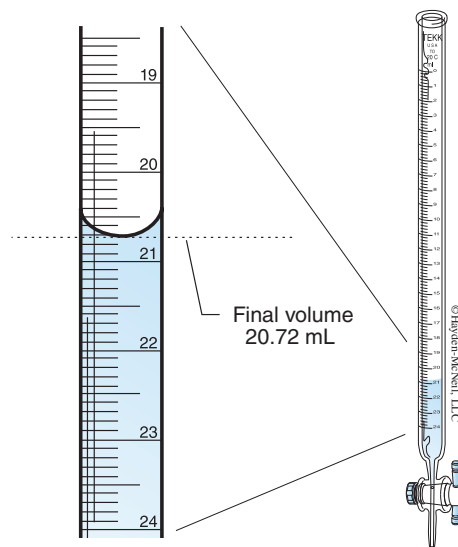


Figure 7.3. Reading buret volume.



MeasureNet

In this experiment, we will use the MeasureNet system to collect data electronically. Using an electronic data collection system allows students to collect more data than if all values had to be recorded on paper. Additionally, data saved at the student workstation will be imported into your Chem21 account so that you can complete your data analysis without having to enter each data point.

Please be aware that components of electronic data collection systems are expensive and must be handled with care. Follow the directions carefully to both collect good data and to protect the equipment.

Figure 7.4 shows a picture of a student workstation with the buttons divided into groups based on their function. Not shown in the figure are the various connector ports where students will connect probes used in collecting data in lab.

Before you start collecting data, your TA will log you into the workstation so that your data is transferred correctly. Do NOT begin work until your TA has confirmed that you are logged in to the correct workstation.

pH Electrode and Calibration

For this titration experiment, we will use a pH electrode to measure the change in pH as we add the standard solution to our analyte solution. In order to obtain accurate data, the pH electrode must be calibrated against solutions of known pH. Because we will be measuring pH values over a wide range, we will calibrate the electrode against two solutions with known pH values of 4.00 and 10.00. It is only necessary to calibrate before the first titration. Changes in signal from the electrode do change over time but the change during one lab period is not significant.

The pH electrode is a delicate and expensive piece of equipment. Use caution to avoid breaking the tip and do **not** use it to stir a solution. The only moveable/removeable part on the electrode is the storage bottle. If you have questions about the correct use and/or storage of the pH electrode, ask your TA for help.

The pH electrode must be stored in an appropriate solution and kept wet when not in use. If your storage bottle is not half-full, obtain additional storage solution from your TA.

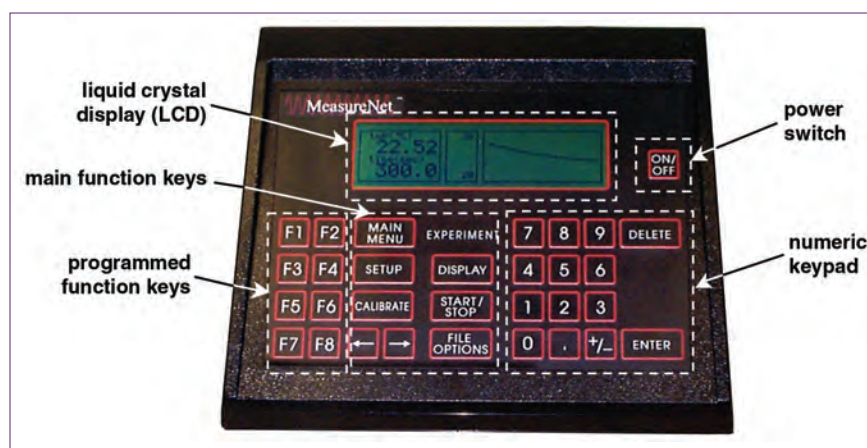


Figure 7.4. Front view of MeasureNet workstation.



Drop Counter

When using the pH electrode, we will also be using a device known as a drop counter. As each drop of liquid passes through the “counting slot” with its infrared beam, it is counted. By entering both the initial and final volumes on the buret, the average drop size can be determined which, along with the number of drops, will allow us to graph a pH vs. volume curve. This titration curve can then be used to determine the equivalence point which is then used to determine the concentration of the analyte solution.

When using a drop counter, it is imperative that you set the equipment up correctly and that the solution dispensed from the buret is done so in drops. If the buret is opened to allow a constant flow of liquid, the data will be unusable.

Materials and Procedures

KHP

0.2 M NaOH solution

unknown concentration of citric acid ($\text{H}_3\text{C}_6\text{H}_5\text{O}_7$) solution

MeasureNet pH probe and drop counter

buffer solutions (pH = 4 and 10)

buret

funnel

ring stand

clamp for drop counter

distilled water

2–50 mL plastic beakers

buret clamp

beakers, assorted

50 mL graduated cylinder

wash bottle

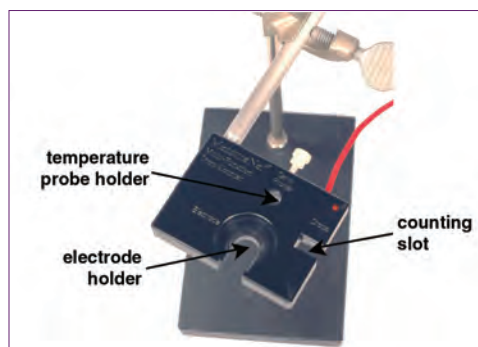


Figure 7.5. Drop counter.

Calibration

1. Obtain the equipment listed above and complete the setup of the drop counter and pH electrode as shown in Figures 7.5 and 7.6.
2. Obtain approximately 5 mL, just enough to cover the tip of the pH probe of each of the buffers (pH 4.00 and pH 10.00), in separate plastic buffer cups. These will be used to calibrate the pH probe.
3. Remove the storage bottle from the pH electrode, connect it to the workstation, and insert it into the opening on the drop counter so that the cap rests in the recess.
4. **Rinse the pH electrode with distilled water** and collect the waste into a 400 mL beaker. This will be used as your waste beaker throughout the experiment.
5. Record the room temperature in your lab notebook.
6. Place the pH 4.00 buffer solution underneath the pH electrode and lower the electrode into the solution.
7. Press “MAIN MENU” on the workstation and select “pH/mV” from the menu and then select “pH v Volume.”
8. Select “CALIBRATE” and the workstation will prompt you to enter the temperature and the correct pH of the solution (i.e., 4.00).



9. Once the pH reading on the screen stabilizes, press “ENTER.” The value on the screen may not read 4.00; you are just waiting for the value to stop changing.
10. When prompted, enter the pH of the second buffer solution (i.e., 10.00). Remove the electrode from the first buffer solution and **rinse well with distilled water into your waste beaker.**
11. Place the beaker with the pH 10.00 buffer below the electrode and lower it into the solution.
12. Once the pH reading on the screen stabilizes, press “ENTER.” The value on the screen may not read 10.00; you are just waiting for the value to stop changing.
13. Press “DISPLAY” on the workstation. The pH value should be within ± 0.05 of the actual pH of the second buffer solution. If not, you need to redo the calibration.
14. Raise the pH electrode and remove the buffer solution. **Rinse the electrode well with distilled water.**
18. Attach a buret clamp to the ring stand and insert the buret so that the tip of the buret is directly over the “counting slot” on the drop counter. The solution from the buret must drop through the counting slot to collect data. You will see the red LED light blink when a drop goes through the slot correctly.
19. Place the waste beaker underneath the buret and close the stopcock.
20. With a funnel, fill the buret with the sodium hydroxide. Remember, do not pour solution above eye level.
21. Open the stopcock to drain enough sodium hydroxide to fill the tip. At this time, you also need to determine how far to open the buret so that the solution drips from the buret. The solution **must** drip in order to collect data.

Standardization of NaOH

15. Obtain about 150 mL of NaOH in a plastic bottle. Label the bottle as a “0.2 M NaOH solution.” As the concentration of the NaOH will vary among batches, you will use this NaOH solution for all of your titrations calling for NaOH.
16. Pour approximately 40–50 mL of the sodium hydroxide solution into a 100 mL beaker.
17. Rinse the buret with 3–5 mL of water by pouring it in the buret so that it covers all sides and then drain it into your waste beaker. Do this three times. Repeat this step with 3–5 mL of sodium hydroxide, draining it into your waste beaker.

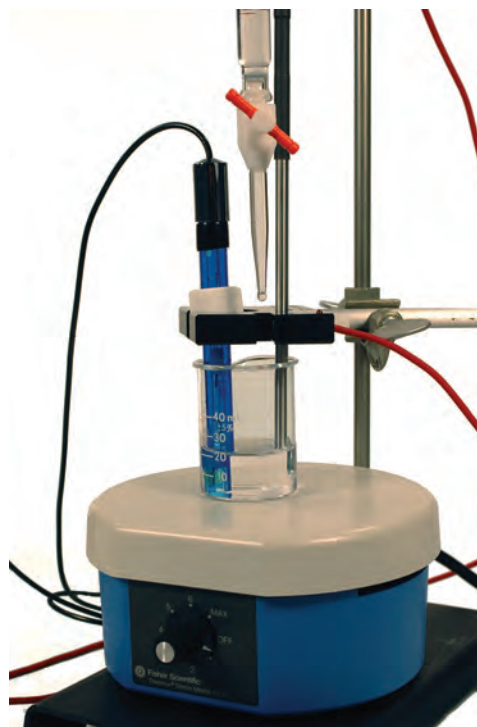


Figure 7.6. Titration setup.



22. Once you have found where to turn the stopcock so that the solution will dispense dropwise, close the stopcock and remove the waste beaker.
23. Measure out 0.7 to 0.8 g of dry KHP, recording the exact value to 4 decimal places in your lab notebook. The KHP is located in the ovens near the balances. Be aware that the ovens are on to gently warm the KHP to keep it dry.
24. Dissolve the KHP in approximately 100 mL of water in a 250 mL beaker. Label the beaker as "KHP solution." Record the exact volume used in your lab notebook. You will use samples from this solution for the 2 KHP titrations.
25. Measure approximately 20 mL of your KHP solution with the small graduated cylinder; record the exact volume used in your lab notebook. Transfer the solution to your 150 mL beaker.
26. Lower the pH electrode into the KHP solution. The pH electrode should be just above the bottom of the beaker so that you can gently swirl the solution, but should also be covered by liquid while swirling.
27. Check that the buret tip is directly over the "counting slot" so the drops will go through the opening and into the beaker. Do **not** open the stopcock yet.
28. Begin the titration by pressing "START/STOP." The workstation will prompt you to enter the initial volume. Enter the actual volume reading from the buret. Remember to record the volume to the second decimal place when you estimate one decimal place beyond the markings on the buret. Also record the initial volume in your lab notebook.
29. Carefully, turn the stopcock on the buret so that the standard **drips** into the acid solution. You may need to make small adjustments to the stopcock so that the solution continues to drip at approximately 1 drop per second. The red LED light will flash each time it detects a drop.
30. Gently swirl the beaker while the NaOH is being added, being careful to keep the beaker under the buret at all times.
31. Allow the NaOH solution to drip into the acid solution until the titration is complete. When complete, the pH value will plateau at approximately 10–12 (see Figure 7.1).
32. When complete, simultaneously press "START/STOP" on the workstation and close the stopcock on the buret.
33. Record the final volume reading in your lab notebook and enter it into the workstation.
34. Press "FILE OPTIONS" on the workstation and select "SAVE DATA." When prompted for a file name use "001." You must use the correct file name so that the data is associated with your Chem21 account. If you save your file with the wrong name, repeat the save with the correct name.
35. Check with your TA to make sure the file was saved and uploaded correctly.
36. If needed, refill the buret with NaOH solution. You will need approximately the same volume of NaOH as used in the first titration.
37. Pour your reacted solution into the container labeled "Waste" in the fume hood.
38. Repeat steps 26 through 35 with another 20 mL of the KHP solution. Press "FILE OPTIONS" on the workstation and select "SAVE DATA." When prompted for a file name, save your file as "002." **You must use the correct file name so that the data is associated with your Chem21 account and ensure that you are not overwriting your previous file.**
39. Check with your TA to make sure the file was saved and uploaded correctly.
40. You should have two trials of KHP titrated with NaOH when you complete this step.



Titration of Citric Acid

41. Obtain approximately 10–12 mL of the unknown concentration of citric acid solution in a clean beaker. Label the beaker as “Citric Acid.” Using the 50 mL graduated cylinder, determine the exact volume. Don’t forget to estimate one decimal place beyond the markings on the glassware and record that in your lab notebook!
42. Add approximately 20 mL of distilled water to the 150 mL beaker and then add the known volume of the citric acid solution.
43. Refill the buret with sodium hydroxide from your beaker.
44. Follow steps 26–33, using the citric acid and sodium hydroxide solutions.
45. Press “FILE OPTIONS” on the workstation and select “SAVE DATA.” When prompted for a file name, save your file as “004.”
46. Repeat this process with fresh citric acid from your beaker labeled “Citric Acid.” Save your file as “005.”
47. Rinse all glassware with distilled water before returning to the correct storage location.
48. Rinse the pH electrode with distilled water, remove it from the drop counter, and replace the storage bottle. The electrode needs to be stored wet in the storage solution, not in distilled water and never stored dry. See your TA if you need additional storage solution.
49. Carefully wipe any liquid from the surfaces of the drop counter.
50. Pour all materials in the appropriate waste container in the fume hood.

You should have completed three titrations using KHP and NaOH and three titrations using citric acid and NaOH at the end of the lab.

Data Analysis

1. Write the balanced chemical reaction between potassium hydrogen phthalate (KHP) and sodium hydroxide.
2. For the first titration, use the graph to determine the pH at the equivalence point and the volume of NaOH needed to reach the equivalence point. Repeat this with the other KHP and NaOH titration.
3. Determine the molarity of the NaOH solutions.
4. What is your average molarity of NaOH?
5. What is your standard deviation?
6. Write the ionic equation for the reaction in this experiment between citric acid ($\text{HC}_6\text{H}_7\text{O}_7$), and sodium hydroxide.
7. Write the net ionic equation for the reaction with citric acid and sodium hydroxide.
8. Using your average NaOH molarity, determine the molarity of the citric acid used in the first titration. Repeat this for your other two citric acid titrations.
9. What is the average molarity of your citric acid solution?
10. What is the standard deviation amongst your two citric acid trials?
11. Obtain the accepted value of the unknown concentration of citric acid from Canvas (under the Experiment 7 Module) and determine the percent error in your results based on the average molarity calculated in the previous step.



EXPERIMENT 7:

DETERMINING AN UNKNOWN CONCENTRATION