Experiment 7

Acid–Base Titration

Lecture and Lab Skills Emphasized

- Understanding the concept of titration.
- Explaining the difference between analyte and standard solutions.
- Know 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

- HCl 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 **http://genchemlab.wordpress.com/7-titration/**.

In Experiment 6, you were faced with one of two of the most common problems chemists face, that is, 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 an environmental agency. The agency recently found as part of their yearly inspections several containers that did not comply with standards of correctly labeling chemical supplies. These containers were marked with the identity of the solutions, but not the quantity of the analyte in the container. The environmental agency wants your team to determine the quantity of analyte in the container so they can dispose of the substances properly. You will need to submit your report of your findings and your reasoning behind your determination to Chem21.

Titration

Titration is a volumetric analysis procedure for determining the concentration of an unknown solution (the *analyte*) by measuring the quantity of a reagent solution of known concentration (the *standard solution*) required to completely react with it. The standard solution will react with the analyte, based on the stoichiometry of the balanced chemical equation. There are a variety of types of titrations, including acid–base and oxidation–reduction titrations. The focus of this experiment will be on acid–base, or *neutralization*, titrations. They are called neutralization titrations because the acid reacts with the base to produce salt and water. The pH (a measure of the solution's acidity or basicity) of the resulting solution would be neutral.

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.

$$HCl(aq) + NaOH(aq) \rightarrow NaCl(aq) + H_2O(l)$$

$$H_2SO_4(aq) + 2NaOH(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$$

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.

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

$$pH = -\log [H^+]$$

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.

In this experiment, you will titrate a hydrochloric acid solution, HCl, with a basic sodium hydroxide solution, NaOH. The concentration of the NaOH solution is given and you will determine the unknown concentration of the HCl. Hydrogen ions from the HCl react with hydroxide ions from the NaOH in a one-to-one ratio to produce water in the overall reaction:

$$HCl(aq) + NaOH(aq) \rightarrow H_2O(l) + NaCl(aq)$$

When HCl solution is titrated with NaOH solution, the pH value of the acidic solution is initially low. As base is added, the change in pH is quite gradual until close to the equivalence point, when equimolar amounts of H^+ and OH^- have been mixed. Near the equivalence point, the pH increases very rapidly. The change in pH then becomes more gradual again, before leveling off with the addition of excess base.

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 7.1 is 20.72 mL.

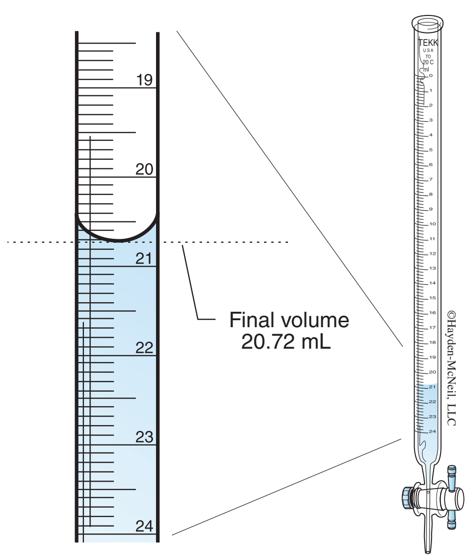

Figure 7.1. Reading buret volume.

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 7.1, 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.

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 7.2 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 collect 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.

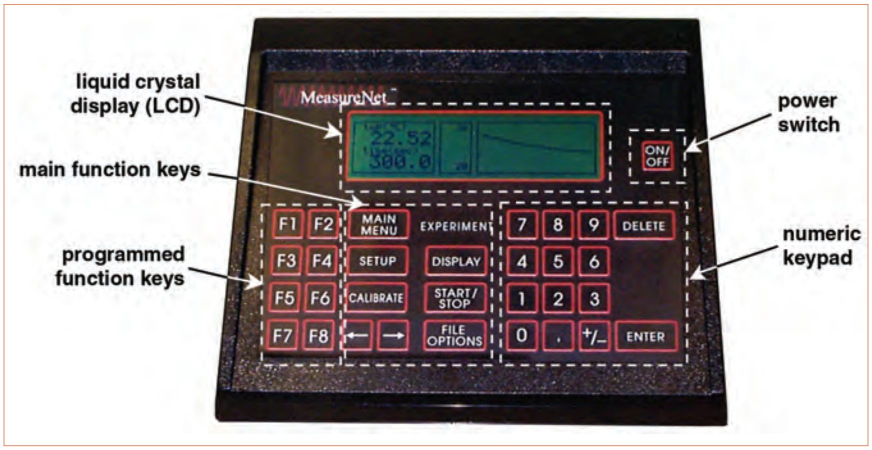

Figure 7.2. Front view of MeasureNet workstation.

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.

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

0.20 M NaOH solution	buret
unknown HCl solution	funnel
ring stand	2 50 mL plastic beakers
pH sensor	buret clamp
drop counter	beakers, assorted
wash bottle	50 mL graduated cylinder
clamp for drop counter	wash bottle
distilled water	
buffer solutions (pH = 4 and 10)	

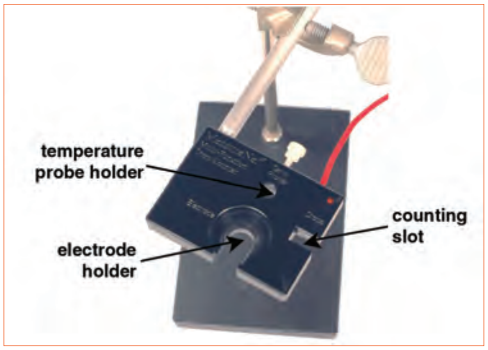

Figure 7.3. Drop counter.

Procedure

Calibration

1. Obtain the equipment listed above and complete the setup of the drop counter and pH electrode as shown in Figures 7.3 and 7.4.

2. Obtain approximately 20–25 mL of each of the buffers (pH 4.00 and pH 10.00) in separate plastic 50 mL beakers. 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 250 mL beaker. This will be used as your waste beaker throughout the experiment.

5. Use a thermometer to measure the temperature of the pH 4.00 buffer solution and record the value.

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.**

Titration

15. Obtain approximately 40–50 mL of sodium hydroxide in a 100 mL beaker.

16. Rinse the buret with 3–5 mL of the sodium hydroxide solution by pouring it in the buret so that it covers all sides and then draining it into your waste beaker. Do three rinses.

17. 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.

18. Place the waste beaker underneath the buret and close the stopcock.

19. With a funnel, fill the buret with the sodium hydroxide solution.

Expt. 7

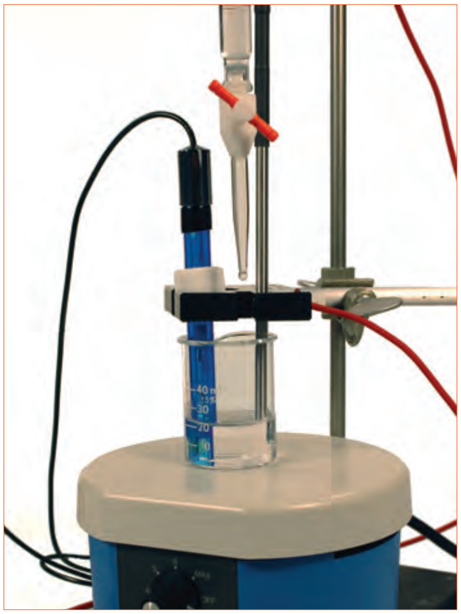

Figure 7.4. Titration setup.

20. 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.

21. Once you have found where to turn the stopcock so that the solution will dispense dropwise, close the stopcock and remove the waste beaker.

22. Obtain approximately 15–20 mL of the unknown HCl solution. Using the 50 mL graduated cylinder, determine the exact volume. Don't forget to estimate one decimal place beyond the markings on the glassware!

23. Add approximately 20 mL of distilled water to the 150 mL beaker and then add the known volume of the HCl solution.

24. Place the 150 mL beaker underneath the drop counter and lower the pH electrode into the solution. The pH electrode should be just above the bottom of the beaker so that you can gently swirl the solution.

25. 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.

26. 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.

27. 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.

28. Gently swirl the beaker while the NaOH is being added, being careful to keep the beaker under the buret at all times.

29. 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 7.5).

30. When complete, simultaneously press "START/STOP" on the workstation and close the stopcock on the buret.

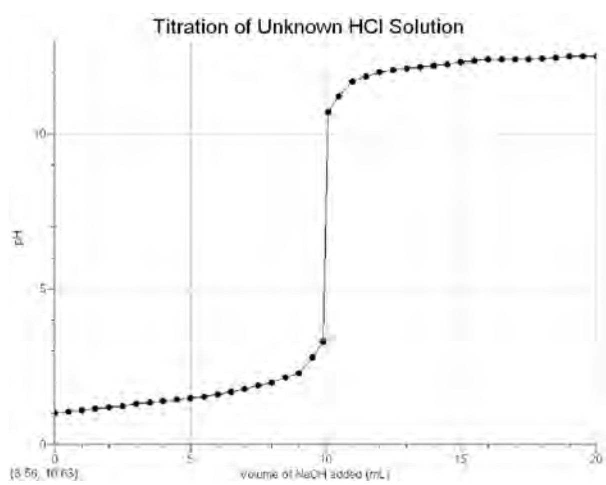

Figure 7.5. Typical titration curve.

31. Record the final volume reading in your lab notebook and enter it into the workstation.

32. 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.

33. Check with your TA to make sure the file was saved and uploaded correctly.

34. If needed, refill the buret with NaOH solution. You will need approximately the same volume of NaOH as used in the first titration.

35. Repeat steps 22–31 with a fresh HCl sample.

36. Press "FILE OPTIONS" on the workstation and select "SAVE DATA." When prompted for a file name use "002." **You must use the correct file name so that the data is associated with your Chem21 account and ensure that you do not overwrite your previous file.**

37. Check with your TA to make sure the file was saved and uploaded correctly.

38. With the water running, pour the waste and excess solutions down the drain.

39. Rinse all glassware with distilled water before returning to the correct storage location.

40. 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.

41. Carefully, wipe any liquid from the surfaces of the drop counter.

42. Pour all materials in the appropriate waste container in the fume hood.

Data Analysis

1. Write the ionic equation for the reaction in this experiment between HCl and NaOH.

2. Write the net ionic equation for the reaction.

3. Look at the shape of your titration curve and determine if the acid is mono-, di-, or triprotic. Explain.

4. 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.

5. Determine the molarity of the HCl used in the first titration.

6. For the second titration, use the graph to determine the pH at the equivalence point and the volume of NaOH needed to reach the equivalence point.

7. Determine the molarity of the HCl used in the second titration.

8. Determine the average molarity of the HCl from both titrations.

9. Obtain the accepted value of the unknown HCl concentration from Blackboard (under "Experiments") and determine the percent error in your results based on the average molarity calculated in the previous step.