Experiment 6: Using a pH Electrode for an Acid-Base Titration
CH2250: Techniques in Laboratory Chemistry, Plymouth State University

Adapted from "7. Using a pH Electrode for an Acid-Base Titration," *Experiments To Accompany Exploring Chemical Analysis, 4th Edition*, Daniel C. Harris (2008), available at http://www.whfreeman.com/exploringchem4e.

Suggested reading for background information: Section 10.1, 10.4, *Exploring Chemical Analysis, 5th Edition*, Daniel C. Harris (2013).

Introduction:

Although the reaction of an acid and a base occurs stoichiometrically, the pH of a solution during a titration does not always show an immediate and sharp jump at the exact point when equivalence is reached. Instead, the pH is controlled by equilibrium and changes in a manner characteristic of a "Titration Curve." In this experiment you will use a pH electrode to follow the course of an acid-base titration, observing how it changes slowly during most of the reaction and rapidly near the equivalence point. You will analyze the titration curve to locate the equivalence point, and use this to calculate the moles of titrant. From the mass of unknown base and the moles of titrant, you can calculate the molecular mass of the unknown base.

Equipment: Read through the procedures and make a list of the equipment you will need.

Safety Considerations: Read through the procedures and note any safety considerations.

Procedure:

1. Obtain Unknown Base from your instructor. You will be given a mass that equals 5-8 mmol. *Note* ***the number of your Unknown*** *in your notebook.*
2. Accurately weigh the Unknown and dissolve it in distilled water in a 250-mL volumetric flask. Dilute to the mark and mix well. This is your Stock Solution
3. Following instructions for your particular pH meter, calibrate a meter and glass electrode, using buffers with pH 4 and 7. Rinse the electrode well with distilled water and blot it dry with a tissue before immersing in any new solution.
4. Fill a 50 mL buret with your Standardized Acid (Lab 5) and record initial volume.
5. Perform a test titration to find the approximate equivalence point. Pipet 25.0 mL of Stock Solution into a 125-mL flask. Add 3 drops of bromocresol green indicator and titrate to the green endpoint. Add 0.5 mL of titrant at a time so that you can estimate the equivalence volume to within 0.5 mL.
6. Now comes the careful titration. Pipet 100.0 mL (*note: this is four times the volume used in Step 5! Keep this in mind as you consider the amount of acid titrant needed to reach the equivalence point*) of Unknown solution into a 250-mL beaker containing a magnetic stirring bar. Add 1 drop of indicator. Position the pH electrode in the liquid so the stirring bar will not strike the electrode. Allow the electrode to equilibrate for 1 min with stirring. Record the pH.
7. Refill your buret, record your starting volume of titrant, and perform the titration. Add ~1.5-mL aliquots of titrant and record the exact volume, the pH, and the color 30 s after each addition. When you are within 5 mL of the equivalence point, add titrant in 0.5 mL increments. When you are within 2 mL of the equivalence point, add titrant in 5-drop increments. When you are within 1 mL, add titrant in 1-drop increments. Continue with 1-drop increments until you are 0.5 mL past the equivalence point. The equivalence point has the most rapid change in pH. Add five more 1.5-mL aliquots of titrant and record the pH after each. Be sure to note when you see the indicator color change.

Analysis

1. In your lab notebook or in Excel, carefully construct a graph of pH (y-axis) versus volume of titrant (x-axis). If you use your lab notebook, use a full, separate sheet for the graph, so that you have a large enough scale to easily be able to record and read small increments. If you use Excel, print a copy of the plot and affix it in your notebook.
2. On the graph, identify the equivalence point and note the volume of titrant (x-axis) at this point.
3. Using the concentration of Standardized Acid you found in Experiment 5, calculate the mmoles of titrant needed to reach the equivalence point.
4. From the moles of titrant used, calculate the moles of the Unknown Base in the 100.0 mL sample of solution (step 6). (you may assume a stoichiometry of 1:1)
5. Calculate the total moles of Unknown base in the original Stock Solution (250 mL; step 2).
6. Based on the moles of Unknown in the 250 mL of Stock Solution and the mass of Unknown used to make that solution, calculate the molecular mass (g/mol) of the Unknown Base.
7. Review your final answers from Lab 5 and calculate (with a calculator or spreadsheet program) the standard deviation of the concentrations of HCl you used to determine the average concentration of your Standardized Acid. *This is the error of the concentration of titrant!*
8. Note that the volume of titrant (HCl) you dispensed from the buret (Procedure step 6 and Analysis question 2) was calculated by subtracting two volume readings on the buret. Determine the error in the volume of titrant.
9. Calculate the error in the moles of titrant (HCl) dispensed by the buret. *This involves a combination of the error of the concentration and the volume of titrant.*
10. Consider the inherent error in the 250 mL volumetric flask used to make the Unknown Stock Solution (step 2). Calculate the error in the concentration (mol/L) of the Unknown in the Stock Solution. *Because the moles of Unknown is found from the moles of titrant (HCl), the error in the moles is the same as your answer to question 9.*
11. Consider the inherent error in the balance used to mass out the Unknown. Calculate the error in the final molecular mass (g/mol) you calculated for the Unknown.

Conclusions

1. Your Instructor will provide you with a table of bases and their molecular weights. Use this to identify your Unknown Base.
2. Consider the molecular mass of the Unknown you calculated versus that of the Base you selected from the table. Calculate the percent error in your experimental answer.
3. Comment on the percent error (Conclusion #2) and the inherent error (Analysis #11) calculated for the molecular mass. How well do you trust your identification of Unknown Base?
4. Comment on the volume of titrant at the equivalence point versus the volume at the point when the indicator color changed. Which method of titration--using and indicator or using a pH meter--do you think is more accurate? Why?

Homework Problems

The following problems from your book must be completed in your lab notebook (see the Syllabus for other suggested problems): Ch 10: **4**, **6**, **21**; Ch 3: **6**, **11**