

Acid–Base Titration: *pH Titration Curve*

OVERVIEW

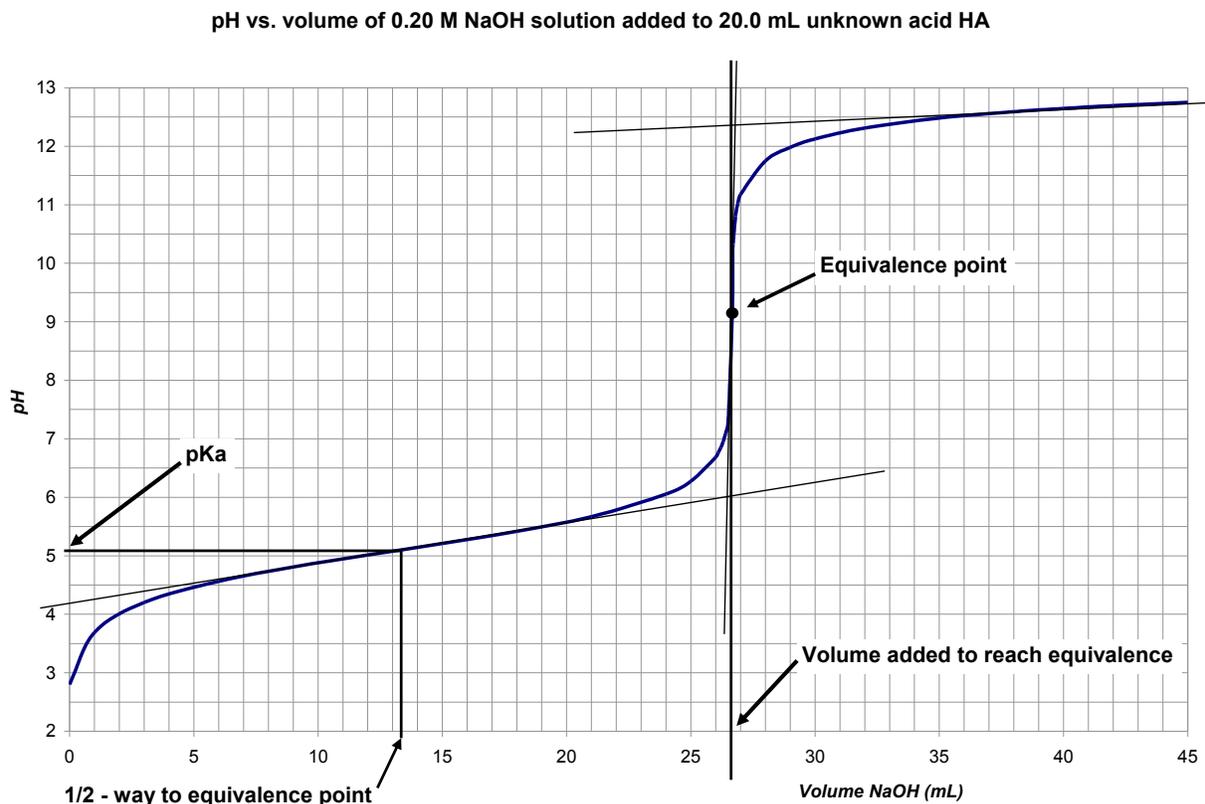
In this experiment, you will perform a pH-monitored titration of acetic acid and of an unknown acid. From the pH titration of the acetic acid, you will determine the equivalence point graphically (see figure 1 below). Based on the volume and concentration of NaOH added to reach equivalence, you will determine the concentration of acetic acid in the vinegar. You will also determine the pKa of acetic acid based on this titration and compare this experimental value to the accepted pKa of acetic acid.

From the pH titration of the unknown, monoprotic acid (“HA”), you will determine the concentration of the acid sample as well as the pKa of the unknown. Based on the pKa, you will identify your likely unknown acid from a list of possible unknowns provided by your instructor.

Optional: Diprotic or triprotic acid.

You may be given a sample of a diprotic or triprotic acid of known concentration. You will carry out a pH-monitored titration to determine if the reaction is diprotic or triprotic, and to determine the pKa values for each successive deprotonation.

Figure 1: *Interpretation of a pH titration curve.*



PROCEDURE

A. Titration of Vinegar with pH monitoring.

1. Obtain approximately 80 mL of standardized 0.100 M NaOH solution.
2. Clean a burette and fill with the NaOH solution.
3. Calibrate the pH meter for the acid (4-7) range.
4. Record the brand and manufacturer's reporting of the percent acetic acid.
5. Pipette 5.00 mL (with a volumetric pipette) of vinegar into a clean 250-mL or 400-mL wide-mouth beaker. Add 20 mL of CO₂-free water. Add 2-3 drops of phenolphthalein.
6. Place a magnetic stirrer in the flask and place on a stir plate. Turn on and adjust the stirring rate to a moderate level.
7. Position the pH probe in the solution out of the way of the stir bar. Use the adjustable arm stand to hold the probe.
8. Position the burette with the NaOH solution inside the mouth of the beaker, but not touching its walls.
9. Record the initial volume of NaOH.
10. Record the initial pH of the acetic acid solution (before adding any NaOH). Turn off the stirrer to take this and all pH readings.
11. Turn on the stirrer.
12. Add approximately 0.50 mL of the NaOH solution. Record the precise volume added. Turn off the stirrer. Record the pH.
13. Repeat step 12 for three additional 0.50-mL aliquots.
14. Add approximately 2.00 mL of the NaOH solution. Record the precise volume added. Turn off the stirrer. Record the pH.
15. Repeat step 14, recording the precise total volume added and pH at each interval of base added. Continue adding 2.00-mL aliquots of base until the pH reaches ~ 5.8.
16. When the pH is ~ 5.8, start adding base at 0.10-mL increments (about 2 drops), recording the precise total volume added and pH with each addition. ***Be sure to mark the pH at which the persistence of pink color occurs.*** Continue this procedure until the pH reaches ~ 10.2.
17. When the pH ~ 10.2, begin adding base in 2.00 mL aliquots again, recording the precise total volume added and pH for each aliquot. Repeat for a total of six 2-mL aliquots.
18. Prepare a graph of the pH versus volume of NaOH added. Use the graph to determine the equivalence point and the pK_a of acetic acid.
19. Calculate the K_a of acetic acid and the concentration of acetic acid in the original vinegar sample.
20. Calculate the percent acetic acid in the vinegar.

B. Titration of an unknown acid sample (treat as “HA” for reactions).

21. Obtain an unknown sample of a monoprotic acid (HA) and record the unknown number. You will be given a sample of the acid that will allow for more than one titration if needed; however, this is all you will get – so be careful with it!
22. Perform a pH-monitored titration of a 25.00 mL sample of your acid.
 - A. Record the pH of the weak acid solution before the titration begins. Record the starting volume on the buret.
 - B. Add approximately 0.50 mL of the NaOH solution. Record the precise volume. Turn off the stirrer. Record the pH.
 - C. Repeat step 22.B. for three additional 0.50-mL aliquots.
 - D. Add approximately 2.00 mL of the NaOH solution. Record the precise volume. Turn off the stirrer. Record the pH.
 - E. Repeat step 22.D., recording the precise volume and pH at each interval of base added. Continue adding 2.00-mL aliquots of base until the pH in the solution is ~ 1.8 pH units above what it was at the end of part 22.C. Then, begin adding the NaOH solution in 1-drop increments (about 0.05 mL). Carefully record the precise volume and pH at each increment.
 - F. When the pH ~ 10, begin adding the NaOH solution in 2-mL aliquots. Add six 2-mL aliquots, recording the precise volume and pH after each.
23. Plot the pH versus volume of NaOH solution for this pH-monitored titration. Graphically determine the equivalence point and the pK_a .
24. Calculate the concentration of the unknown acid solution and the K_a .
25. Based on the K_a of the acid, determine its identity. Calculate a percent error based on this assignment (compare K_a 's for the percent error).

DATA ANALYSIS (Summary)

- A. Be sure to include each of the chemical reactions on which the titrations are based. For the unknown acid, treat it as “HA”.
- B. Plot the pH vs. volume of NaOH solution added for the pH-monitored titration of vinegar. Use the plot to determine the volume of NaOH required to reach equivalence, the pH at equivalence, and the pK_a of acetic acid.
- C. Calculate the K_a of the acetic acid.
- D. Calculate the $[CH_3CO_2H]$ in vinegar, the % by mass of CH_3CO_2H , and the % difference from the manufacturer’s claim.
- E. Compare your value for pK_a to the accepted value.
- F. Plot the pH vs. volume of NaOH solution added for the pH-monitored titration of the unknown acid. Use the plot to determine the volume of NaOH required to reach equivalence, the pH at equivalence, and the pK_a of the unknown acid.
- G. Calculate the K_a of the unknown acid. Identify the unknown (based on the list of knowns provided in lab).
- H. Calculate the $[HA]$.
- I. Compare your value for pK_a to the accepted value (based on your identification of the acid).

C. Optional: Titration of a sample of a diprotic or triprotic acid with pH monitoring.

26. Prepare a sample of the 0.10 M unknown acid using by mixing 10.00 mL of the acid solution with 20.0 mL of deionized water.
27. Titrate the sample in the same way you carried out the titration of the unknown sample in part B. Adjust step 22.E. as suggested by your instructor based on whether you have a diprotic or triprotic acid.

DATA ANALYSIS (Optional section)

28. Prepare a pH vs. volume NaOH curve.
29. Find the overall equivalence point.
30. Determine whether the acid is diprotic or triprotic.
31. Use the plot to determine the pH at the overall equivalence point, as well as at the equivalence point for each successive deprotonation.
32. Use the plot to determine the pK_a and K_a for each acid dissociation step.
33. Based on the number of protons on your acid, and the K_a values identify your acid.

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ASSIGNMENT & REPORT GUIDELINES

Prepare a standard prelab, including the purposes, the reactions studied, and the procedure summary / general strategy **FOR EACH PART** to be performed presented in the required manner for the course.

Answer the PRE-LAB Question and submit as instructed.

Prepare a standard formal laboratory report, including presentation of the original data, reorganized data tables (if appropriate), calculations and results (as outlined in the procedure and summarized in the DATA ANALYSIS (Summary) Section) and formal, typed discussion/error analysis and conclusions sections.

Answer the POST-LAB Questions on the printed page.

PRELAB QUESTION

Please show your complete calculations on a separate page. Please prepare your plot using appropriate graphing software. Submit as instructed.

pH Titration Curve

A 40.0 mL sample of a 0.30 M solution of NH_3 is titrated with a 0.50 M HCl solution.

Calculate the pH of the solution when the following percentages of the HCl solution required to reach the equivalence point have been added.

Plot the data you determined for the pH at various points in the titration.

- **What is the pH of the ammonia solution before the titration begins (0% of the required HCl added)?**
 - 2% of the total volume of HCl?
 - 5% of the total volume of HCl?
- **20% of the total volume of HCl?**
 - 35% of the total volume of HCl?
- **50% of the total volume of HCl?**
 - 65% of the total volume of HCl?
 - 90% of the total volume of HCl?
- **95% of the total volume of HCl?**
 - 98% of the total volume of HCl?
- **100% of the total volume of HCl?**
 - 101% of the total volume of HCl?
- **105% of the total volume of HCl?**
 - 120% of the total volume of HCl?

POSTLAB QUESTIONS

Consider your pH-monitored titration of the ***unknown acid in part B***.

1. Based on your calculated concentration of the unknown acid and its K_a , calculate what the pH of the solution should have been (in a perfect system at 25 °C) before any of the NaOH solution was added to the solution.

Compare this to the value you measured.

2. Based on your calculated concentration of the unknown acid, its K_a , its volume, and the volume of NaOH that had been added to reach equivalence (as determined graphically), calculate what the pH should have been (in a perfect system at 25 °C) for the titrated solution at equivalence.

Compare this to the value you measured.