Acid–Base Titration: ***pH Titration Curve***

OVERVIEW

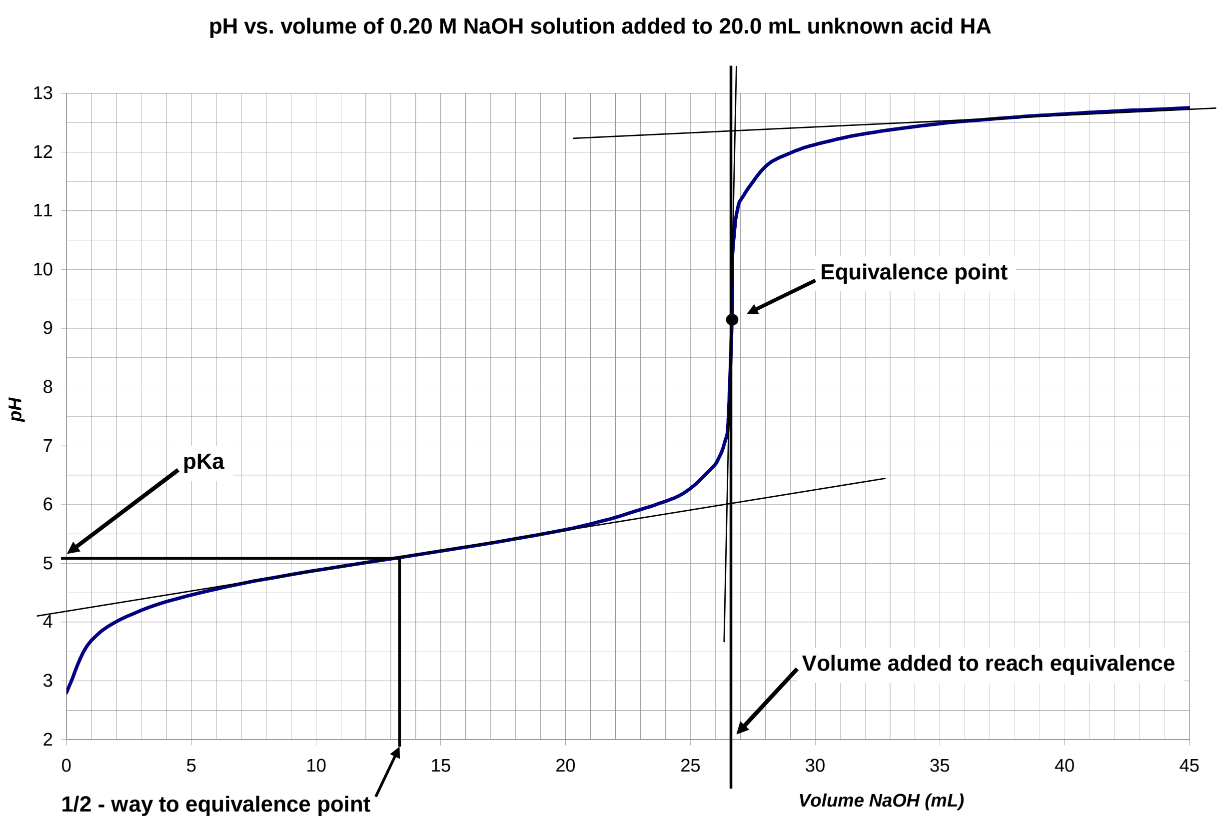
In this experiment, you will perform a pH-monitored titration of acetic acid and of an unknown acid. From the pH titration of the acetic acid, you will determine the equivalence point graphically (see figure 1 below). Based on the volume and concentration of NaOH added to reach equivalence, you will 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, 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.

HONORS section: Diprotic or triprotic acid.

You will 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 CO_2-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. Add approximately 2.00 mL of the NaOH solution. Record the precise volume added.
12. Turn off the stirrer. Record the pH.
13. Repeat the above two steps above, 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.
14. 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. Continue this procedure until the pH reaches ~ 10.2.
15. 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.
16. 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.
17. Calculate the K_a of acetic acid and the concentration of acetic acid in the original vinegar sample.
18. Calculate the percent acetic acid in the vinegar.

B. Titration of an unknown acid sample.

19. Obtain an unknown sample of a monoprotic acid and record the unknown number. You will be given 100-mL of the acid. This is all you will get – so be careful with it!
20. 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.
    B. Add 2.00 mL of standardized NaOH solution. Record the pH.
    C. Continue to add in 2.00 mL aliquots, recording the precise total volume and the pH after each addition.
    D. When the pH in the solution is ~ 2 pH units above what it was in part B, begin adding the NaOH solution in 0.10 mL increments (about 2 drops).
    E. When the pH ~ 10, begin adding the NaOH solution in 2-mL aliquots. Add six 2-mL aliquots, recording the pH after each.
21. Plot the pH versus volume of NaOH solution for this pH-monitored titration. Graphically determine the equivalence point and the pK_a.
22. Calculate the concentration of the unknown acid solution and the K_a.
23. 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).

<u>DATA ANALYSIS (Summary)</u>

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.

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

24. 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.
25. Titrate the sample in the same way you carried out the titration of the unknown sample in part B.

<u>DATA ANALYSIS (Honors section)</u>

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

<u>DISCUSSION / ERROR ANALYSIS / CONCLUSIONS</u>

Include the appropriate information in these sections.

POSTLAB QUESTIONS

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

1. Based on your calculated concentration the unknown acid and its Ka, calculate what the pH of the solution should have been before any of the NaOH solution was added to the solution.

   Compare this to the value you measured.

2. Based on the information discussed in part A, and the volume of NaOH that had been added to reach equivalence (as determined graphically), calculate what the pH should have been for the titrated solution at equivalence.

   Compare this to the value you measured.