Experiment 9: Titration of Acids
(This experiment was adapted from Titration of a Weak Acid, by CC-BY Torres & González-Urbina, CUNY)

Purpose

Weak acids are acids that do not dissociate completely when dissolved in water, releasing only some of its hydrogen atoms into the solution. Acetic acid (CH_3COOH) is an important weak acid, produced from the fermentation of ethanol in wine. Commercial vinegar is just an aqueous solution of acetic acid. The goal of this experiment is to calculate the molar concentration and %wt of a sample of acetic acid in vinegar by means of a standard chemical procedure known as titration. You will use your results as a quality control check for the advertised %wt concentration on the bottle of vinegar. In order to do that you will react the weak acid with a basic solution of sodium hydroxide (NaOH), which has a known concentration. You will use phenolphthalein as indicator. A strong acid, HCl, ionizes completely when dissolved in water. The HCl solution will be used to first practice the technique of titration, before analyzing the vinegar sample.

Background

A titration is a technique where a solution of known concentration (often times a base) is used to determine the unknown concentration of another solution (often times an acid) [1]. Both substances react with each other in an acid-base reaction. The solution of known concentration is delivered carefully from a buret until an indicator (a third substance added to indicate the end of the titration) changes color. This experiment is a classic practice, previously implemented in numerous college chemistry laboratory manuals [2-5].

Volumetric Analysis

The determination of concentration by measuring volumes is called volumetric analysis. Titrations are volumetric analyses where a buret is used to add and measure the volume of one of the reactants. Acid-base titrations are an extensively used chemical technique employed to determine a solute concentration in a solution. In an acid-base titration, an acid reacts with a base by gradually adding one solution to the other. The volume of the acid solution (in the Erlenmeyer flask) is known from the pipet, and the volume of the NaOH solution required for the complete reaction is measured (from the buret).

An indicator is used to indicate the exact end of the reaction. The indicator chosen will have one color before the reaction is complete and a different color when the acid-base reaction finishes. For example, in the reaction between acetic acid (CH_3COOH) and sodium hydroxide (NaOH):

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$$
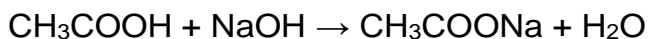
using phenolphthalein as the indicator, the solution will be colorless before completion of this reaction but pink after completion. At a specific point during the titration, a single drop of the NaOH from the buret will cause the solution being titrated to turn from colorless to a barely discernible pink color. This point is called the endpoint. The endpoint signals the exact point when the moles H^{1+} equals the moles of OH^{1-}, which is referred to as the equivalence point.

Figure 13: (Left panel) A buret employed in a titration. (Center panel) Illustration showing the correct way to control the buret valve. (Right panel) The titration set up

References

(1) Crouch, D. S.D.W.F.L.H. S., Fundamentals of analytical chemistry; Nelson Education: 2013.
(2) Beran, J. A., Laboratory manual for principles of general chemistry; John Wiley and Sons: 2010.
(3) Ebbing, R. W.D. D., Experiments in General Chemistry; Houghton Mifflin Harcourt Publishing Company: 2004.
(4) Russo, T., Merrill Chemistry Lab Manual; McGraw-Hill: 1998.
(5) Holman, G. H. J., Chemistry in Context; McGraw-Hill Education: 2014.

Chemicals
0.075 M HCl solution
0.10 M NaOH solution
Commercial vinegar (acetic acid solution)
Phenolphthalein 1% solution

Equipment

Buret, 50 mL	Pipet, 10 mL, and pipetor
Erlenmeyer flask, 150 mL	Funnel
Plastic droppers	Clean, dry beakers (various sizes)
Volumetric flask, 100 mL	

Procedure
Set-Up
1) Obtain a 10 mL pipet and pipetor, a 50 mL buret, a buret stand, and buret clamp.
2) Obtain about 50 mL of hydrochloric acid solution in a clean, dry 100 mL beaker and about 80 mL of the NaOH solution in a clean, dry 150 mL beaker.
3) Rinse your buret with deionized water. Use a plastic funnel to rinse your buret three times with approximately 2 mL of NaOH solution per rinse, and then fill it with the NaOH solution.
4) Remember to drain the NaOH solution so the meniscus is within the calibration marks and the air in the tip is pushed out.
5) Record the initial volume in the buret. _Record all buret volumes with two digits after the decimal point._

Doing the titrations

Hydrochloric Acid (To prove you can do a titration.)
1) Pipet 20.00 mL of hydrochloric acid into a clean 125 mL Erlenmeyer flask. Add approximately 10 mL of distilled water and 2 drops of phenolphthalein.
2) Record the molarity of the NaOH solution indicated in the stock solution bottle.
3) Place the flask under the buret.
4) Add the NaOH solution from the buret to the Erlenmeyer flask, while swirling the solution in the flask. Add the NaOH fast in the beginning. As you approach the endpoint, slow the addition of NaOH. By the time you get close to the endpoint, you should be adding the NaOH dropwise.
5) The titration is completed when the addition of 1 drop of NaOH causes the color to change from colorless to a very light shade of pink.
6) Record the final buret volume. _This was your practice run._
7) Repeat the steps above three more times; these three titrations of hydrochloric acid will be used for the calculations.
8) Calculate the concentration of the original hydrochloric acid solution, in each of the three trials. Calculate the standard deviation of three trials. If the standard deviation is greater than 0.005 M, do two more titration trials. Repeat the standard deviation calculation using the five trials.

Vinegar
1) Vinegar is advertised as 5%wt. acetic acid. Assume the density of the acetic acid solution is 1.0 g/mL. Do a quick calculation to convert 5%wt to molarity, M, of acetic acid.

$$5\ \% = \frac{50\ g\ acetic\ acid}{1000\ g\ soln.} \times 100 \qquad 1000\ g\ solution = 1000\ mL\ solution = 1\ L\ solution$$

How many moles of acetic acid are in the 1 L of solution?
That will let you calculate molarity.

2) The vinegar is too concentrated for a titration using 0.10 M NaOH. You will need to dilute the vinegar, using either a 100 mL volumetric flask. Figure out the dilution you want, so that you will use approximately 15 mL of NaOH when titrating 20 mL of the diluted vinegar solution. (Hint, use the dilution equation to solve for Md.)
3) Pipet 20.00 mL of the diluted vinegar into a clean 125 mL Erlenmeyer flask. Add approximately 10 mL of distilled water and 2 drops of phenolphthalein.
4) Place the flask under the buret.

5) Add the NaOH solution from the buret to the Erlenmeyer flask, while swirling the solution in the flask. Add the NaOH fast in the beginning. As you approach the endpoint, slow the addition of NaOH. By the time you get close to the endpoint, you should be adding the NaOH dropwise.
6) The titration is completed when the addition of 1 drop of NaOH causes the color to change from colorless to a very light shade of pink that persists for at least one minute.
7) Record the final buret volume.
8) Repeat steps 3 – 7 above two more times; these three titrations of the diluted vinegar will be used for the calculations.
9) Calculate the acetic acid molarity concentration in the diluted vinegar solution, in each of the three trials, and then the average acetic acid molarity. Then calculate the molarity of acetic acid in the original vinegar solution, before the dilution.
10) Use the average acetic acid molarity to calculate the %wt acetic acid in the vinegar solution. Assume the density of the vinegar is 1.0 g/mL. *Is the vinegar solution really 5% acetic acid as advertised on the label?*

The equation for calculating the standard solution for three trials is:

$$\sqrt{\frac{((dev.\ 1)^2 + (dev.\ 2)^2 + (dev.\ 3)^2)}{(3-1)}}$$

deviation 1 = / trial 1 - average /

deviation 2 = / trial 2 - average /

deviation 3 = / trial 3 - average /