Experiment 13
Titrations

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Name:

Date:

Key Objectives

1. Nomenclature - Titration, End point, Indicator, Standard solution, Meniscus.
2. Solving titration problems.
3. Use of molecular weight and mol-to-mol ratio's.
4. Determine the concentration of an unknown solution.

Discussion

Titrations are a standard technique used to determine the concentration of a solution by chemically reacting it with a known solution. There are a variety of techniques for doing this, we will focus on the most basic technique realizing that we can apply it to more complicated problems if required.

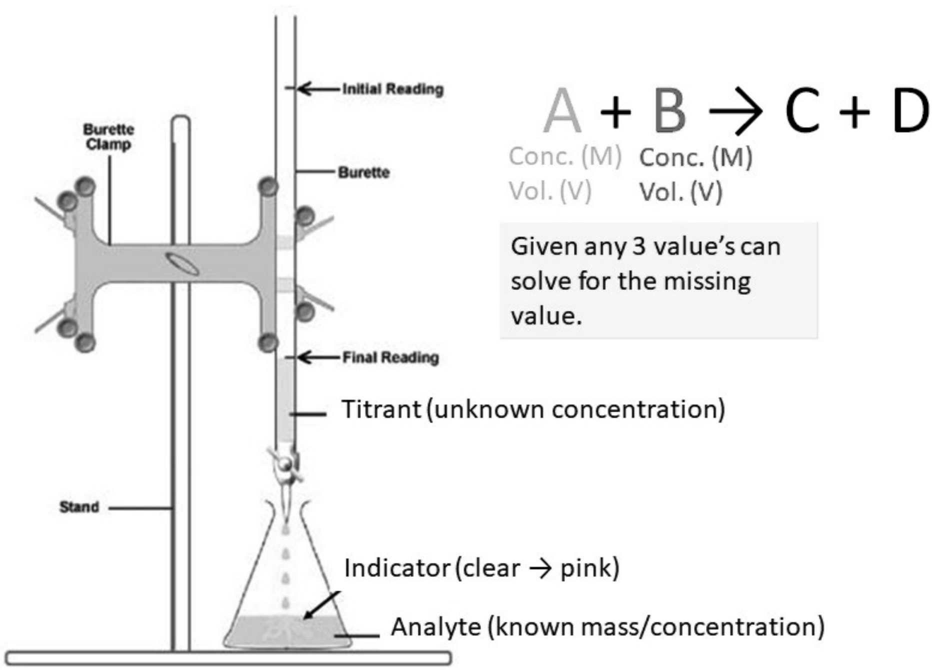

Figure 13.1: Typical setup for performing a titration. credit: author

A discussion of titrations can be found in Hein Ch. 15.10 or in McMurry Ch 3.10. **Titrations** are a method for determining the amount or concentration of an unknown in a solution. Titrations react a solution of known concentration (the **standard**) with a solution of unknown concentration (the unknown). If the reaction goes to completion, and the yield is 100%, then the concentration of the unknown solution can be determined through the stoichiometry of the chemical reaction. The completion of the reaction is determined by a variety of methods including color changes, pH, and electrical conductivity.

Concentration is generally measured in terms of **Molarity** (M), which has units of (moles of solute/L of solution), or (mol/L).

A typical reaction which goes to completion with 100% yield is the reaction of an acid with a base. Such reactions are also commonly referred to as neutralization reactions. Generally we can write:

$$\text{Acid} + \text{Base} \longrightarrow \text{Salt} + \text{Water} + \text{Heat} \tag{38}$$

In this lab we will use the reaction of potassium hydrogen phthalate, $KHC_8H_4O_4$, commonly known as "KHP" with Magnesium Hydroxide ($Mg(OH)_2$). The reaction is balanced as shown below.

$$\underline{\ 2\ }\,KHC_8H_4O_4(aq) + \underline{\ 1\ }\,Mg(OH)_2(aq) \longrightarrow \underline{\ 1\ }\,Mg(KC_8H_4O_4)_2(aq) + \underline{\ 2\ }\,H_2O(l) \tag{39}$$

The choice of reaction to study is made because KHP is a solid at room temperatures, we can measure its concentration by mass, which is much more accurate and precise then measuring liquid volumes. We will use this to titrate an unknown concentration of $Mg(OH)_2$.

The **end point** of a reaction is the point at which the reaction is neutralized (the concentration of the reactants are equal) or 100% complete and is determined by using an **indicator**. Most indicators signal the end point of a reaction by changing colors, though other changes are possible. The indicator chosen for this reaction is phenophthalein, an organic dye, that is colorless in acid solutions (the KHP solution) and turns pink in basic solution (when enough $Mg(OH)_2$ is added).

The standard instrument used in titrations is the **Buret** which is a piece of calibrated glassware used to accurately measure volumes of liquids. Figure 13.1 shows a typical example. The buret is filled with the solution ($Mg(OH)_2$) to be titrated and is often referred to as the titrant.

Volume measurements are made by reading the point on the graduated scale that coincides with the bottom of the curved surface called the **meniscus** of the liquid as shown in Figure 13.2. A discussion of why a meniscus is formed can be found in Hein Ch. 13.4, Chang Ch. 11.3, or McMurry Ch. 10.4.

Do not waste your time trying to fill the buret to the zero line or some other round number, because The exact volume to which the buret is filled is unimportant because we are measuring the difference in volume (Δv).

The titrant is now added to a solution made from the KHP, water, and indicator. Titrant is added until just one drop changes the solution from colorless to a faint pink color. The exact process will be demonstrated in class by your instructor. The final volume of liquid is then measured.

Using the mass of the KHP, the volume of $Mg(OH)_2$ used and the stoichiometry of the reaction it is then possible to determine the molarity of the $Mg(OH)_2$ solution.

For example, if you start with 1.025 g of KHP and add 22.50 mL of an unknown Molarity of $Mg(OH)_2$ calculate the Molarity of the solution.

$$\frac{1.025\,\text{g KHP}}{22.50\,\text{mL}} \times \frac{1\,\text{mol KHP}}{204.2\,\text{g KHP}} \times \frac{1\,\text{mol}\,Mg(OH)_2}{2\,\text{mol KHP}} \times \frac{1\,\text{mL}}{0.001\,\text{L}} = 0.1116\,\text{M}\,Mg(OH)_2$$

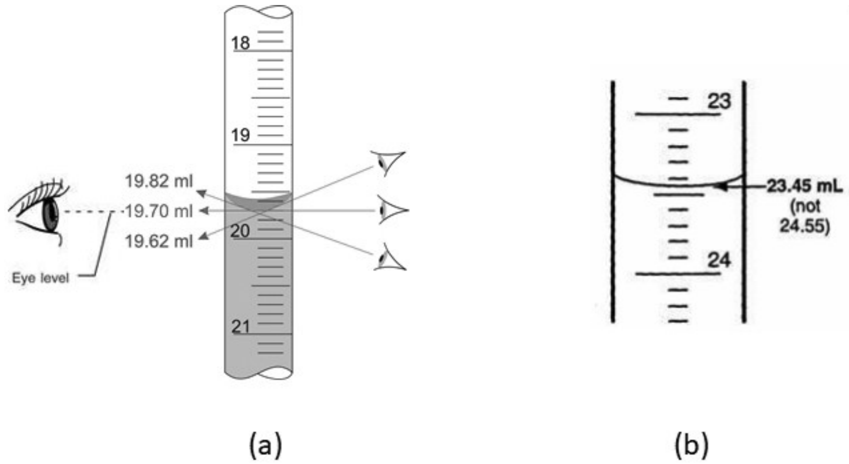

Figure 13.2: (a) Always read a buret with your eyes level with the bottom of the meniscus. (b) Notice the numbering on the scale, it goes from top to bottom (opposite of a beaker). credit: (a) E. Generalic, https://glossary.periodni.com/glossary.php?en=meniscus (b) https://effectiveness.lahc.edu/academic_affairs/sfcs/chemistry/Shared

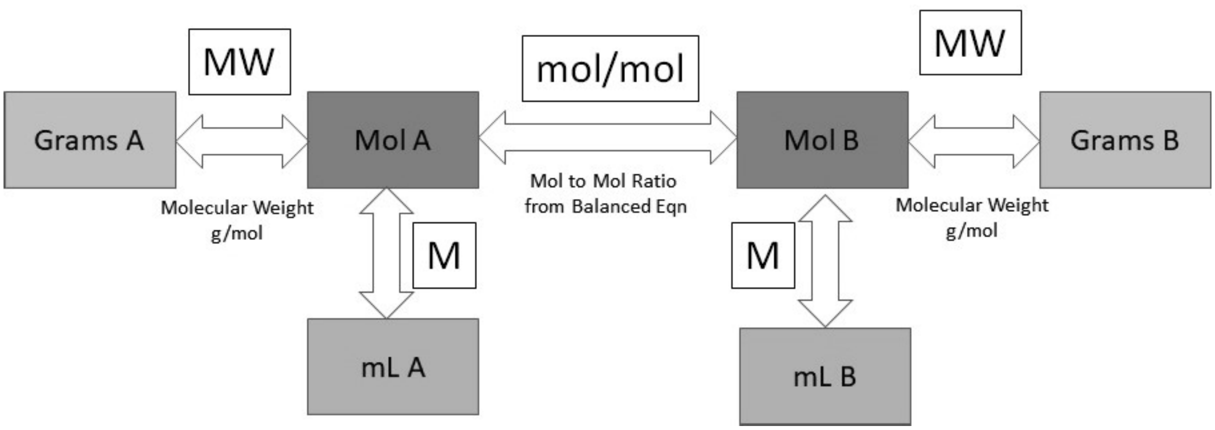

Figure 13.3: Using conversion factors to solve a titration problem. credit: author

Procedure

In this experiment you will be titrating a known mass of an acid (KHP) with an unknown concentration of base ($Mg(OH)_2$). After determining the concentration of the base solution you will use it to determine the concentration of acetic acid in vinegar and compare it to the manufactures claimed concentration.

Read the entire procedure before beginning the experiment.

Part 1 - Practice using the buret

1. When reading the meniscus level in the buret, you may find it helpful to hold a white card (Figure **??**) marked with a dark strip (or a dark colored object) behind and slightly below the meniscus. Remember, your line of sight should be level with the meniscus to obtain the most accurate reading.

2. To what uncertainty can you measure volume using a buret?
3. Add between 40 and 45 mL of distilled water to the buret using a funnel placed on the top of the buret. Be sure to check for air bubbles in the tip of the buret, if found you need to dispense your liquid until they are removed. Record your reading on your data sheet and have your instructor verify the reading.
4. Weigh an empty beaker.
5. Place an empty beaker below your buret and dispense between 10 and 12 mL of distilled water into it. Record your final reading, and have your instructor verify it.
6. Record the mass of water dispensed.
7. Calculate the volume of water dispensed into your beaker. (Show work)
8. Calculate the difference between the volume of water dispensed and the mass of water dispensed. Does the calculated volume of water dispensed into your beaker agree with the mass of the water dispensed into the beaker within 0.1g of the expected value? If not consult your instructor.
9. If you have any troubles with this portion of the lab, or your mass and volume measurements disagree too much, consult your instructor, and do a second trial if needed.
10. Continue practicing dispensing varying amounts of water, at varying rates from the buret into a beaker until you feel comfortable controlling the water flow using the stopcock.
11. Finish by draining all of the water from the buret into the beaker.
12. Any water used in this part of the experiment may be disposed of down the sink.

Part 2 - Obtaining a $Mg(OH)_2$ sample, and filling the buret.

You will need the $Mg(OH)_2$ solution for titrating both the KHP standard Each titration should take between 10-20 mL of the $Mg(OH)_2$ solution.

1. Obtain about 250 mL of the $Mg(OH)_2$ solution from your instructor. Put your name on the Erlenmeyer flask.
2. Close the stopcock on your buret and carefully add through a funnel 5 mL of your $Mg(OH)_2$ solution. Remove the funnel. Remove the buret from the support stand, and hold it almost horizontally. Carefully rotate so that the $Mg(OH)_2$ solution contacts the entire inner surface.
3. Drain the $Mg(OH)_2$ solution through the buret tip into a beaker labeled “Discarded Rinse Solutions” or dispose of it down the sink.
4. Clamp the buret into the support stand. Fill the buret to a point slightly below the 0 mL calibration mark.
5. Dispense the $Mg(OH)_2$ solution though the stopcock and remove any air bubbles present. If you

can not remove the air bubbles consult your instructor.

6. Once all air bubbles are removed, refill the buret to between the 0 and 10 mL mark.

Part 3 - Preparing the KHP sample

1. Obtain a 250 mL Erlenmeyer flask.
2. Calibrate your scale (hold button down until the calibration menu is reached. Then hit the on button, wait until it flashes 100 g. Place the 100 g calibration weight on the scale and push the on button again. Wait until the calibration is complete). Be sure to recalibrate the scale if you move it or spill anything on it.
3. Using weighing paper or boat, carefully weigh out about 1.0 grams (between 0.9 and 1.1 grams) of KHP onto the paper. (Be sure to tare the mass of the weigh boat or paper).
4. Carefully pour the KHP into the 250 mL Erlenmeyer flask. Add about 75 mL of distilled water (measured with graduated cylinder) into the Erlenmeyer flask and gently swirl the flask and its contents until the KHP is fully dissolved.
5. Add 3 drops of phenolphthalein indicator solution to the KHP solution and swirl gently to mix.

Part 4 - Titrating the KHP

1. Place the Erlenmeyer flask containing the KHP sample under the buret as shown in Figure 13.1.
2. Place a white sheet of paper under the solution. This will make it much easier to detect the color change.
3. Record the value of the starting volume on your data sheet.
4. Add 1-2 mL volumes of the $Mg(OH)_2$ solution from the buret to the KHP solution, while swirling gently.
5. A faint pink color will appear with each addition. When the pink persists for a second or two start adding the $Mg(OH)_2$ in smaller volumes. The goal of a titration is to have the solution be clear and after adding a single drop of KHP to the solution it will turn a faint pink color. You will be required to be this accurate in order to complete the lab.
6. You may wash down the sides of your container if you need to with distilled water from a wash bottle.
7. When a single drop of the $Mg(OH)_2$ solution causes a pink color to persist for 30 seconds you are done with the titration.
8. Have your instructor check your result to see if it is a good trial. This will be accomplished by adding several drops of an acid solution to your now pink KHP solution. If the color changes back to clear it is a successful trial. If it does not you will need to repeat the trial.
9. Record the final volume on your data sheet.

10. Repeat Parts 4 and 5 until you have a total of 3 good trials approved by your instructor. If you need another data sheet ask your instructor for one.

11. Finish your calculations for each trial and calculate the average molarity of the unknown $Mg(OH)_2$ solution. You will be graded on both the accuracy of your result and the precision.

Results

Be sure to measure as accurately and precisely as possible. Make sure all measurements have the proper uncertainty, significant figures and units. Show work for all calculations.

Practicing with a buret (Part 1)

	Trial 1	**Trial 2**	**Trial 3**
1. Uncertainty in volume measurements:	________	________	________
2. Mass of empty beaker:	________	________	________
3. Initial buret reading:	________	________	________
4. Verify with your instructor:	________	________	________
5. Final buret reading:	________	________	________
6. Verify with your instructor:	________	________	________
7. Volume of distilled water delivered (show calculation):	________	________	________
8. Mass of beaker + water dispensed:	________	________	________
9. Mass of water dispensed (show calculation):	________	________	________
10. Difference between Volume (7) and Mass (9) (show calculation):	________	________	________

1. Does the volume of water measured agree with the mass of the water measured? Explain.

2. List two sources of error that might exist when making these measurements? Explain.

Titration of KHP (Part 4)

	Trial 1	Trial 2	Trial 3
1. Mass of KHP::	________	________	________
2. Uncertainty in volume measurements:	________	________	________
3. Initial buret reading:	________	________	________
4. Final buret reading:	________	________	________
5. Verify titration (drops of acid added):	________	________	________
6. Volume of $Mg(OH)_2$ delivered (show calculation):	________	________	________

	Trial 4	Trial 5	Trial 6
1. Mass of KHP::	________	________	________
2. Uncertainty in volume measurements:	________	________	________
3. Initial buret reading:	________	________	________
4. Final buret reading:	________	________	________
5. Verify titration (drops of acid added):	________	________	________
6. Volume of $Mg(OH)_2$ delivered (show calculation):	________	________	________

	Trial 7	**Trial 8**	**Trial 9**
1. Mass of KHP::	______	______	______
2. Uncertainty in volume measurements:	______	______	______
3. Initial buret reading:	______	______	______
4. Final buret reading:	______	______	______
5. Verify titration (drops of acid added):	______	______	______
6. Volume of $Mg(OH)_2$ delivered (show calculation):	______	______	______

	Trial 10	**Trial 11**	**Trial 12**
1. Mass of KHP::	______	______	______
2. Uncertainty in volume measurements:	______	______	______
3. Initial buret reading:	______	______	______
4. Final buret reading:	______	______	______
5. Verify titration (drops of acid added):	______	______	______
6. Volume of $Mg(OH)_2$ delivered (show calculation):	______	______	______

1. What is the molecular weight of KHP. (Remember KHP is an abbreviation for Potassium Hydrogen Phthalate).

2. Write the complete and balance reaction that occurs when KHP reacts with $Mg(OH)_2$.

3. In the space below show a sample calculation for determining the Molarity of your solution for your first "Good" trial.

4. List the Trial Number and Molarity of your 3 best trials. Calculate the Average Molarity (show calculation).

Trial Number: _____ __________

Trial Number: _____ __________

Trial Number: _____ __________

Average Molarity: __________

Post Lab Questions

5. In choosing an acid for the experiment, a solid (KHP) was chosen instead of a liquid (HCl for example). Why would a solid acid be chosen over a liquid acid. Explain?

6. Why is buret considered dirty (and must be cleaned) if water droplets adhere to the inner surface? (or why don't we want to use dirty burets?)

7. Why do we want to eliminate air bubbles in the buret, especially in the area below the stopcock?

8. Why are you allowed to wash down the sides of your Erlenmeyer flask during the titration? (Hint: Why is having no water in the buret important, but adding water to the acid in the Erlenmeyer unimportant.) Explain.

9. A student determined the percent KHP in an impure sample of KHP. A 2.750 g sample of impure KHP required 39.55 mL of 0.1215 M $Mg(OH)_2$ solution for titration. What is the percentage of KHP in the impure sample? (Hint: You can not start with the impure sample of KHP because you can't calculate the molecular weight of the mixture, since you don't know what it is contaminated with. Instead, determine the amount of pure KHP in the sample by using the titration information.) The percent of KHP in the impure sample is given by:

$$\text{Percent of KHP} = \frac{\text{mass pure KHP in sample}}{\text{mass of impure sample}} \times 100\%$$

10. What assumption must be made about the impurity in the previous question.

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Name: ____________________________ Class: ______________ Date: ______________

Prelab Questions

1. Define each of the following terms as they relate to the experiment:
   (a) Titration

   (b) End point

   (c) Indicator

   (d) Standard Solution

   (e) Meniscus

2. What is the molecular weight of KHP. (Remember KHP is an abbreviation for Potassium Hydrogen Phthalate).

3. Write the complete and balance reaction that occurs when KHP reacts with Magnesium Hydroxide.

4. A student standardized a $Mg(OH)_2$ solution, following the procedures outlined in this experiment. The student weighed a samples of KHP (1.350 g) and transferred it to a 250 mL Erlenmeyer flasks. She added distilled water, the indicator, and then titrated the sample with 18.75 mL an unknown concentration of $Mg(OH)_2$ solution. Calculate the Molarity of the $Mg(OH)_2$ Be sure to include the correct units and SF for all answers.