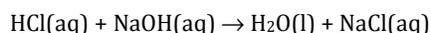


**Laboratory Activity III Introduction: Volumetric Analysis – Titration**

A special type of analytical procedure is called **volumetric analysis**. In this process, the volume of a known substance is used to determine the quantitative characteristics of another substance based upon the amounts of each required for the substances' complete reaction. When we use a solution for this process, we term it **titration**. The two most common titrations in a first-year course such as this are acid-base titrations and oxidation-reduction titrations (which are the topic of the next laboratory activity). In essence, we cause complete reaction between a solution whose molarity is accurately known – the **titrant** – and the substance about which we are determining some characteristic – the **analyte**. Then, we use the volume of the titrant and the stoichiometry to calculate what we want to know about the analyte. Most often, we will be determining the molar mass, percent composition or molarity of unknowns with titration.



Imagine that we have a 0.100 M solution of NaOH in a buret. We also have 25.00 mL HCl of unknown concentration in a beaker beneath the buret. We will add sodium hydroxide until the reaction reaches its **equivalence point**, which is the point at which the reaction has occurred to 100% according to the reaction. For many reactions, an indicator is used to indicate that the equivalence point has been reached – indicators change colors under certain conditions; here, we would choose an indicator that turns colors – or reaches its **end point** – as the reaction reaches completion, which tells us that all of the acid is consumed. (We will discuss the selection of indicators when we explore acid-base chemistry later.) Alternatively, one of the reactants may itself change colors under certain conditions – we will see this with oxidation-reduction titration.

We will demonstrate and perform calculations for a volumetric analysis together.

Using Volumetric Analysis: Determining Molarity

- Amount of NaOH required to reach the equivalence point: \_\_\_\_\_ mL NaOH
- Mol of NaOH required to reach the equivalence point: \_\_\_\_\_ mol NaOH
- Mol of HCl that reacted based upon the mol of NaOH used: \_\_\_\_\_ mol HCl
- Molarity of the HCl solution: \_\_\_\_\_ M HCl

We can also determine the molar mass of a compound using volumetric analysis. In this case, we determine the mol of the unknown in the sample using the amount of titrant used. Then, simply convert the sample mol to molar mass given the size of the sample used. We will demonstrate and perform calculations for a volumetric analysis to determine molar mass together.

We add a 0.500 g sample of an unknown solid monoprotic acid to distilled water. Then, we titrate the acid with 0.100-molar sodium hydroxide solution until the equivalence point is reached.

Chemical Reaction \_\_\_\_\_

*Using Volumetric Analysis: Determining Molar Mass*

- Mass of solid acid dissolved in water: \_\_\_\_\_ g unknown acid
- Amount of NaOH required to reach the equivalence point: \_\_\_\_\_ mL NaOH
- Mol of NaOH required to reach the equivalence point: \_\_\_\_\_ mol NaOH
- Mol of acid present in the solid sample: \_\_\_\_\_ mol acid
- Molar mass of the acid sample \_\_\_\_\_ g/mol

We can also determine the percent composition of a solid using volumetric analysis in a similar manner to that which we determined percent composition of solids using gravimetric analysis. We will demonstrate and perform calculations for a volumetric analysis to determine percent composition together.

We add 2.00 g of a mixture containing acetylsalicylic acid and salts to distilled water. We are going to determine the percent composition of the mixture that is acid. The NaOH is 0.100 M. We assume that the salts will not react with the hydroxide ion and that only one acid is present in the mixture.

*Using Volumetric Analysis: Determining Percent Composition*

- Mass of solid mixture dissolved in water: \_\_\_\_\_ g
- Amount of NaOH required to reach the equivalence point: \_\_\_\_\_ mL NaOH
- Mol of NaOH required to reach the equivalence point: \_\_\_\_\_ mol NaOH
- Mol of acid that reacted based upon the mol of NaOH used: \_\_\_\_\_ mol acid
- Mass of acid present \_\_\_\_\_ g acid
- Percent composition acid in the mixture: \_\_\_\_\_ % acid

You will titrate several acid samples – solid and aqueous solution – to both practice the process of titration and determine characteristics of some acids and bases. All data and discussion should be included in the lab notebook. First, however, complete the following items for practice.

**Practice Items: Volumetric Analysis/Titrations**

**Question 1.** A 14.57 mL sample of 0.300-molar HCl(aq) was required to neutralize the Ba(OH)<sub>2</sub>(aq) in a 20.00 mL sample.

- i) Write the balanced chemical equation for the reaction that occurred.
- ii) Determine the number of mol Ba(OH)<sub>2</sub>(aq) present in the Ba(OH)<sub>2</sub>(aq) sample.
- iii) Determine the molarity of the barium hydroxide solution.

**Question 2.** A 0.0250 g sample of an unknown acid with *two* ionizable hydrogens is titrated with 0.0050-molar NaOH(aq). A 40.98 mL sample of the NaOH(aq) solution was required to reach the equivalence point.

- i) Write the net ionic equation for the reaction that occurred.
- ii) Determine the molar mass of the acid that was titrated.

**Question 3.** This question combines gravimetric analysis with titration. A 1.093 g solid mixture containing NaCl(s) and a monoprotic organic acid HA(s) was treated to determine the percent composition of the mixture and the molar mass of the acid. After being dissolved in water, all of the chloride ion was precipitated as AgCl(s), and a 2.329 g sample of AgCl(s) was recovered. Then, the filtrate – now containing only the acid and spectator ions – was treated with 0.0500-molar NaOH(aq). A 12.91 mL volume of NaOH(aq) was required to reach the equivalence point.

- i) Determine the percent composition NaCl(s) in the original mixture.
- ii) Determine the molar mass of the acid.

**Question 4.** Answer the following.

- i) Distinguish between the *end point* in a titration and the *equivalence point*.
- ii) Burets are calibrated considering that the tip of the buret is filled with the solution to be used when the titration begins. Indicate how each the following will affect the *mol of acid* calculated (less than actual, equal to actual or greater than actual) during a titration in which an acid is titrated with a base: the buret is rinsed with water, the stopcock closed and then filled for use.
- iii) During a titration with phenolphthalein, you believe that the color of the analyte has changed to a persistent pink. However, you are not sure, so you add one more drop of base to confirm, and the analyte turns a dark pink. Can you think of a procedural step that makes this a usable trial? Explain your answer.