**EXPERIMENT 21**

Molarity of a Hydrochloric Acid Solution by Titration

**INTRODUCTION**

Volumetric analysis is a general term meaning any method in which a volume measurement is the critical operation; however, the term is most often used when referring to titration methods alone. A *titration* consists of the controlled and measured mixing of two solutions, one containing a known mass or volume of the substance being analyzed, the other a known concentration of a reagent which reacts with the component of interest in the substance. Usually, portions of the reagent are transferred from a *buret* to a flask containing the other solution. In order to serve as a satisfactory basis for an analysis, a titration operation must fulfill certain requirements. There must be only a single reaction during the titration, namely, the one involving the titrating agent and the component of interest in the substance being analyzed. This is usually either an acid-base reaction or an oxidation-reduction reaction.

A titration is continued until essentially stoichiometric amounts of the two reagents have been brought together, and this defines the *endpoint* of the titration. An endpoint is often marked by an abrupt change in the color of some constituent, known as the *indicator*. A very weak acid or base that will exhibit a different color in acidic or basic solutions can be added to be the indicator for acid-base titrations. As a neutralization reaction reaches completion, the acidity of the resulting solution changes drastically, and the color change of the weak acid or base indicator will signify the end of the reaction. When using a weak acid or base indicator, only a small amount of it should be used because it must react with some of the titrating reagent in order to change color. The following are common volumetric analysis techniques.

1. **SWIRLING.** During or following the delivery of a portion of reagent solution from a buret, the contents of the titration vessel should be swirled in order to insure adequate mixing. By placing the titration vessel on a stirring plate, a spinning magnetic stirring bar in the titration vessel can be used to insure that there is adequate mixing.

2. **ADDING REAGENT.** At first, the reagent solution can be added to the sample solution at a moderate rate. As the operation progresses, temporary changes in the color of the indicator at the point of mixing becomes more and more persistent, indicating that the reagent solution must be introduced more slowly. During the final approach to the end point, the solution is added one drop at a time until there is a permanent change in the color of the indicator.

3. **WASHING.** Just prior to reaching the end point, the vessel walls are washed down with a stream of liquid solvent to insure that all the reagent delivered from the buret actually reached the solution. If necessary, the walls can be washed during the earlier stages of the titration since the total volume of the solvent present in the reaction system is relatively unimportant.

A volumetric analysis for the molarity of a hydrochloric acid solution features the use of the base sodium hydroxide to neutralize completely the hydrochloric acid in an aqueous solution of known volume. The acid-base reaction is

\[
\text{NaOH (aq)} + \text{HCl (aq)} \rightarrow \text{NaCl (aq)} + \text{H}_2\text{O (l)}
\]

The end point is signaled by the change in color of the indicator phenolphthalein. The volume of sodium hydroxide used is noted, and knowing this and the volume of the hydrochloric acid solution, the molarity of the hydrochloric acid can be calculated.
ACCURACY AND PRECISION

Experimental determination of any quantity is subject to error because it is impossible to carry out any measurement with absolute certainty. The amount of error in any determination depends on the quality of the instrument or the measuring device, and the skill and experience of the experimenter. Thus, discussion of errors is an essential part of experimental work in any quantitative science. The types of errors encountered in making measurements are classified into three groups:

1. **GROSS ERRORS.** Gross, careless errors are those due to mistakes by the experimenter that are not likely to be repeated in similar determinations. These include the spilling of a sample, reading the mass incorrectly, reading a buret volume incorrectly, etc.

2. **RANDOM ERRORS.** Random errors are due to the inherent limitations of the equipment or types of observations being made. Generally these can be minimized by using high-grade equipment and by careful work with this equipment, but can never be completely eliminated. It is customary to perform measurements in replicate in order to reduce the effect of random errors on the determination.

3. **SYSTEMATIC ERRORS.** Systematic errors are those that affect each individual result of replicate determinations in exactly the same way. They may be errors of the measuring instrument, of the experimenter, or of the method itself. Examples of systematic errors in chemical analyses include such things as the use of impure materials for standardization of solutions, or improperly calibrated volumetric glassware such as pipets, burets, and volumetric flasks.

Two terms used to describe the reliability of experimental measurements are precision and accuracy. **Accuracy** refers to the agreement of an experimental measurement with the true value. **Precision** refers to the agreement among several experimental measurements of the same quantity, and reflects the reproducibility of a given measurement. The difference between these two terms is shown below by the results of four different dart throws.

The different types of errors are illustrated to the right. A **gross error** or **random error** will produce a measurement that has an equal probability of being high, low, left, or right, and large gross or random errors produce results that are not precise. The spread of the darts on the two top targets are a result of large gross or random errors, while the spread of the darts on the two bottom targets indicate small gross or random errors. The two top targets show throws that are not precise, while the two bottom targets show throws that are precise.

A **systematic error** will produce a measurement that is off consistently in the same direction, and large systematic errors produce results that are not accurate. The spread of the darts on the two left targets are a result of a large systematic error, while the spread of the darts on the two right targets indicate a small systematic error. The two left targets show throws that are not accurate, while the two right targets show throws that are accurate.

In this experiment, you will test both your precision and accuracy. You will perform multiple trials to determine the molarity of the hydrochloric acid solution, and the agreement between your results will demonstrate your precision. You will then check your experimentally determined molarity of the hydrochloric acid with its true value, and the agreement between the two will demonstrate your accuracy.
PROCEDURE

1. Students will work individually for this experiment. Except for the laboratory handout, remove all books, purses, and such items from the laboratory bench top, and placed them in the storage area by the front door. For laboratory experiments you should be wearing closed-toe shoes. Tie back long hair, and do not wear long, dangling jewelry or clothes with loose and baggy sleeves. Open your lab locker. Put on your safety goggles, your lab coat, and gloves.

2. Take out the stirring plate, plug it into the electrical outlet, and also make sure the cord is firmly plugged into the back of the stirring plate. Make sure the heating knob is turned to off. Screw in a small ring stand to the back of the stirring plate, and attach a double buret clamp to the ring stand. Obtain a 20.00-mL pipet and a 50.00-mL buret from the back counter. Always carry pipets and burets in a vertical position. Attach the buret to the double buret clamp. Obtain a pipet bulb from the back of the lab room.

3. Using a 100-mL beaker, obtain 50 mL of the hydrochloric acid solution from the carboy found on the lab bench in front of the instructor’s desk. Clean and dry a 125-mL Erlenmeyer flask. Condition the pipet with three small portions of the hydrochloric acid solution, disposing of the acid solution used for conditioning down the sink. Pipet a 20.00 mL sample of the hydrochloric acid solution into the 125-mL Erlenmeyer flask. Add 10 mL of deionized water and two drops of phenolphthalein to the flask.

4. Using a 250-mL beaker, obtain 75 mL of the standardized sodium hydroxide solution from the carboy found on the lab bench in front of the instructor’s desk. Record the molarity of the sodium hydroxide solution in your Data Table.

5. Condition your buret by rinsing it three times with 5 mL portions of the sodium hydroxide solution, holding the buret in a horizontal position with the stopcock closed and rolling it to make sure that the sodium hydroxide solution wets the entire inside surface. Drain the sodium hydroxide solution through the buret tip into a 150-mL waste beaker. When opening the Teflon stopcock, if it is difficult to turn, loosen its locking nut slightly. All chemicals can be washed down the sink with water. Using a funnel, and with the top of the funnel below eye level, fill the buret with the sodium hydroxide solution. Run some of the sodium hydroxide solution through the buret tip into the waste beaker until (1) you are sure that all the bubbles are removed from the buret tip, and (2) the sodium hydroxide meniscus is at or below the 0.00 mL mark on the buret. Remove the last drop from the tip of the buret, then remove the waste beaker.

6. Obtain a clean and dry magnetic stirrer, and carefully slide it into the Erlenmeyer flask. Center the Erlenmeyer flask on the stirring plate, and adjust your buret so the tip is slightly inside the neck of the flask. Read the buret and record it as the Initial Buret Reading. Do this by holding a card with a thick black line behind the buret and below the meniscus. Make your reading at the bottom of the meniscus, which will appear as a thin, black arc. Have another student verify the reading. Turn on the stirring plate, slowly increasing the speed until the stirring bar creates a vortex in the liquid, but does not collide with the sides of the vessel.
7. Add the sodium hydroxide solution intermittently from the buret to the Erlenmeyer flask, noting the pink phenolphthalein color that appears and disappears as the drops hit the liquid and are mixed with it. When the pink color begins to persist, slow down the rate of the addition of sodium hydroxide until you are adding the sodium hydroxide drop by drop. In the final stages of the titration, rinse the inside wall of the flask with deionized water from your wash bottle, and add half drops until the entire solution just turns a pale pink color. If the hydrochloric acid sample turns from colorless to pale pink with the addition of only one drop of the sodium hydroxide solution, use this titration to determine the molarity of the hydrochloric acid and read the buret and record it as the \textit{final buret reading} for Trial 1. Have another student verify the reading. However, if the hydrochloric acid turns from colorless to dark pink because several drops were added, this would be a gross error, the trial should be discarded, and you should start again.

8. Calculate the molarity of the hydrochloric acid from the data in Trial 1 if it produced a pale pink endpoint. Read the actual molarity of the hydrochloric acid written on the large container in the back of the room, and compare it to your calculated molarity. The expected \text{\textit{accuracy}} for volumetric analysis is 1\%. This means that the molarity of the hydrochloric acid you calculated should agree within 1\% of the actual molarity. If your calculated molarity does not agree within 1\% of the actual molarity, try to do better on the next trial!

9. Clean and dry another 125-mL Erlenmeyer flask. Pipet a 20.00 mL sample of the hydrochloric acid solution into the 125-mL Erlenmeyer flask. Add 25 mL of deionized water and two drops of phenolphthalein. Refill the buret with the standardized sodium hydroxide solution. Read the buret and record it as the \textit{initial buret reading} for Trial 2. Titrate the hydrochloric acid solution again to a pale pink endpoint. Read the buret and record it as the \textit{final buret reading} for Trial 2.

10. The expected \text{\textit{precision}} for volumetric analysis is 1\%. This means that the volume of sodium hydroxide solution needed to neutralize each 20.00 mL sample of hydrochloric acid should agree within 1\% of each other. If your two trials do not agree within 1\% of each other, you should perform a third trial.

11. At the end of the experiment, rinse the buret thoroughly, including the tip, several times with tap water and three times with deionized water, and dry off the outside. Rinse the pipet thoroughly, several times with tap and three times with deionized water, and then dry off the outside. Return both items to the back counter, the buret with the stopcock open.

12. Once your working area has been checked your lab report can then be turned in to the instructor.
EXPERIMENT 21 LAB REPORT

Name: ___________________________________________ Student Lab Score: __________
Date/Lab Start Time: _____________________________ Lab Station Number: ______________

DATA TABLE

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<td>Volume of HCl Solution</td>
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<td>Molarity of NaOH Solution</td>
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<td>Initial Buret Reading</td>
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<td>Final Buret Reading</td>
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<td>1-3 Volume of NaOH Solution Used</td>
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<td>4-6 Molarity of HCl Solution</td>
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<td>7 Avg. Molarity of HCl Solution</td>
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CALCULATIONS

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POSTLAB QUESTIONS

1. Window cleaner contains ammonium hydroxide. A 10.00 mL sample of window cleaner is titrated with 28.50 mL of 0.311 M hydrochloric acid. What is the molarity of the ammonium hydroxide in the window cleaner? Box your answer.

2. A Rolaids tablet contains calcium carbonate to neutralize stomach acid. One Rolaids tablet requires 34.55 mL of 0.448 M hydrochloric acid to convert its calcium carbonate into calcium chloride, carbon dioxide and water. How many grams of calcium carbonate are there in one Rolaids tablet? Box your answer.