Experiment 8

Introduction to Acid-Base Titration

Key Concepts

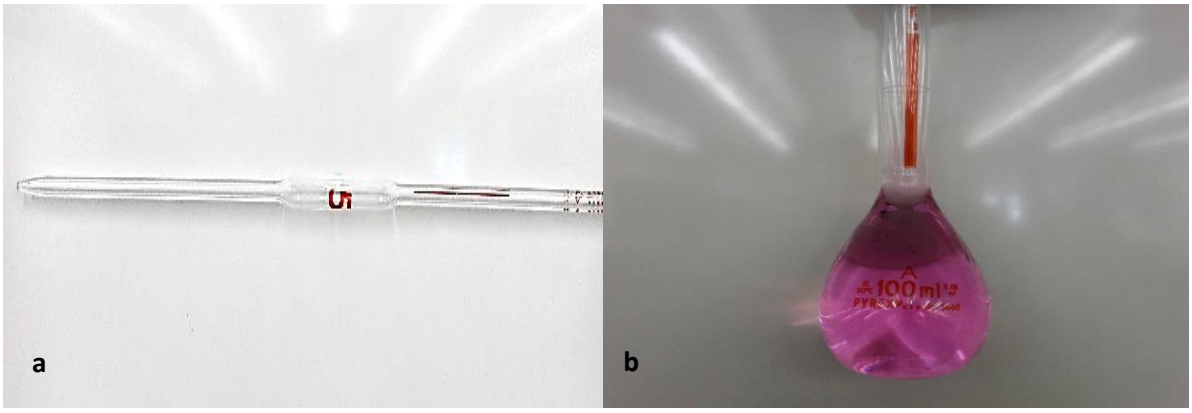
After completing this experiment, you are responsible for understanding: the use of volumetric glassware, indicators, endpoints, acid-base reactions and titrations.

Introduction

Volumetric Glassware

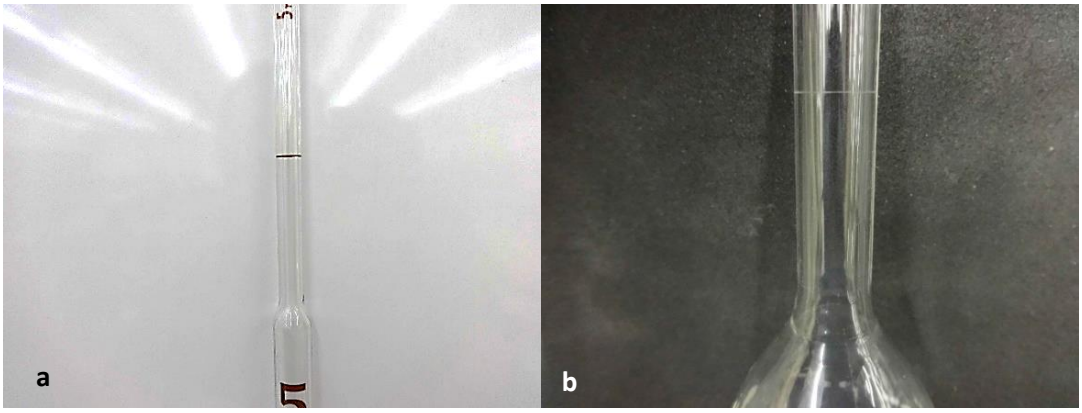
Before discussing acid-base titrations, we will look at a few types of glassware you have not used previously in this course. Titrations require the use volumetric glassware and burettes. **Volumetric glassware** is calibrated to contain an accurate and precise volume at the specified temperature. Typically glassware is calibrated at 20 °C which is around room temperature. Most volumetric glassware is labeled with its calibration temperature. Unlike the graduated glassware you have used in the past, volumetric glassware is designed to measure a single volume. There are two types of volumetric glassware used in this experiment, a 5.00 mL volumetric pipette as shown in Picture 1a and a 100.0 mL volumetric flask shown in Picture 1b.

Picture 1. (a) 5.00 mL volumetric pipette. (b) 100.0 mL volumetric flask.



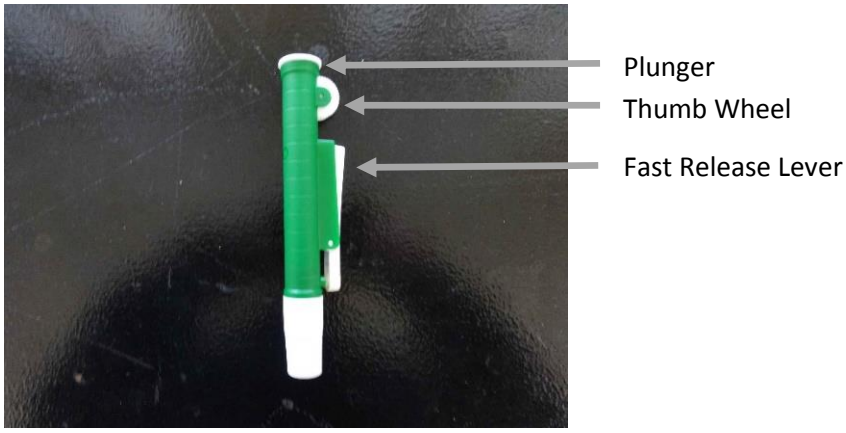
Volumetric glassware is filled with a liquid or solution so the bottom of the meniscus is even with the calibration line. The **calibration line** is a marking indicating the fill level of the volumetric glassware. Pictures 2a and 2b show the calibration lines for a 5.00 mL volumetric pipette and a 100.0 mL volumetric flask.

Picture 2. (a) Calibration line of a 5.00 mL volumetric pipette. (b) Calibration line of a 100.0 mL volumetric flask.



A volumetric flask is used by adding solution or solute to the flask and adding solvent to the calibration line. If solid is added, it is important to dissolve the solid in a small amount of solvent before filling the flask completely to the calibration line. So in the case of a 100.0 mL volumetric flask, one might dissolve the solid in 60 to 80 mL of solvent first and then fill the flask to the calibration line. In the titration section of this experiment you will dilute 2.00 mL of 5.00 M sodium hydroxide in a 100.0 mL volumetric flask.
A volumetric pipette is used to transfer a specific volume of liquid from one container to another. The tip of the pipette is submerged into the liquid to be transferred and a pipette pump, attached at the top, creates suction and draws the liquid into the volumetric pipette. A pipette pump employs a thumb wheel and fast release lever to draw and dispense liquids as shown in Picture 3.

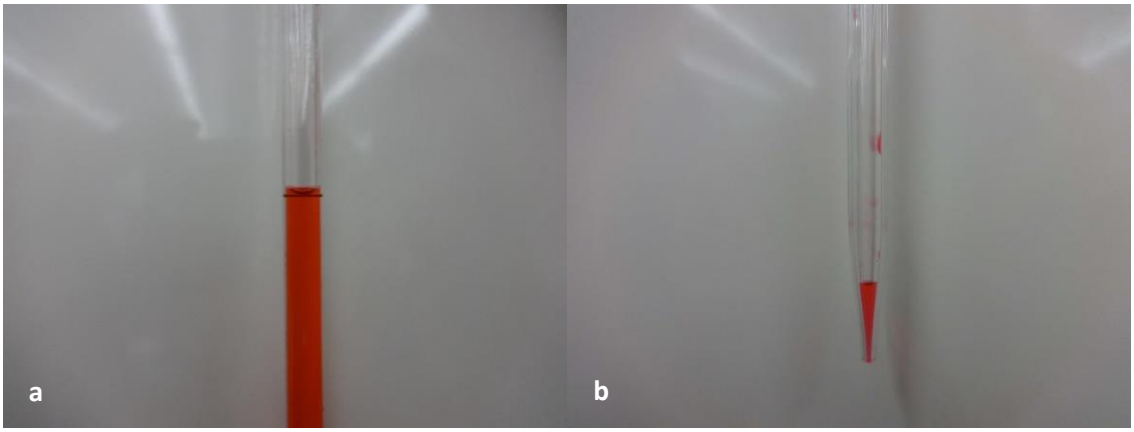
Picture 3. Pipette Pump.



The thumb wheel is used to draw liquid into the pipette and adjust the height of the liquid so the meniscus is at the calibration line as shown in Picture 4a. It is very important that the pipette remain submerged in the liquid while drawing in solution or air will be drawn into the pipette. The thumb wheel can be moved in either direction until the meniscus is even with the calibration line. Once the volumetric pipette is properly filled to the calibration line, the pipette is removed from the container of liquid and held over

another container. The fast release lever is pressed to release the suction and allow the liquid to flow into a new container. It is very important not to press the plunger to dispense the liquid into a new container as this will dispense all of the liquid in the pipette. Volumetric pipettes are designed to have a small hole in them and this small hole traps a small amount liquid in the tip as shown in Picture 4b. The liquid in the tip should not be dispensed.

Picture 4. (a) Liquid to the calibration line in a volumetric pipette. (b) Residual liquid in the tip of a volumetric pipette.



Typically, solutions are drawn and transferred using volumetric pipettes. However, this can be dangerous because it is fairly easy to draw too much solution into the pipette and the excess solution then goes into the pipette pump. It is especially dangerous, and won't be done in this course, if concentrated acids and bases are pipetted. Even dilute acids and bases or other solutions can leave residues in the pipette pumps that can ruin them and pose chemical exposure dangers. To practice pipetting, without using solutions, we will start by pipetting water to determine the accuracy and precision of the 5.00 mL volumetric pipette. It is still important to avoid drawing water into the pipette pump, but if water gets in the pump try to remove as much as possible by pressing the plunger down.

To determine the accuracy and precision of the 5.00 mL pipette, the mass of 5.00 mL of water obtained from the 5.00 mL volumetric pipette will be determined. The mass can then be used to calculate the volume of this water sample. First, the mass of an empty 50 mL Erlenmeyer flask is recorded. Next 5.00 mL of water is transferred to the 50 mL Erlenmeyer flask and the mass of the water and the 50 mL Erlenmeyer flask is recorded. The mass of the water for the first trial can be found by difference. This is done over five trials to determine the accuracy and precision of the volumetric pipette. The MicroLab thermocouple can be used to measure the temperature of water and the volume of water can be calculated in Excel using the density of water from Table 1. Once five trials are completed, statistical calculations can be performed to determine the accuracy and precision. There is an example of a similar procedure and calculations in experiment 1. If you have questions please come see us at office hours before your lab.

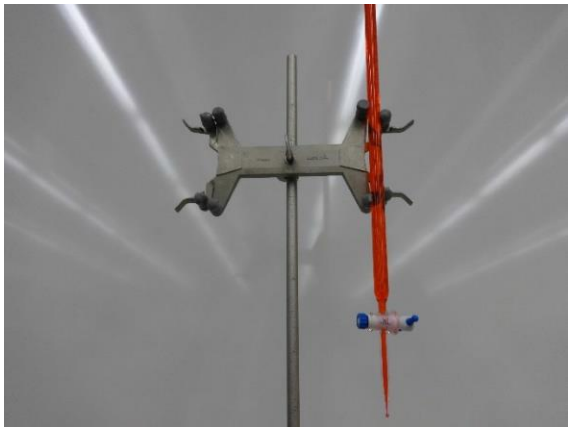
Table 1. Density of water at various temperatures and atmospheric pressure.

Temperature (°C)	Density (g/mL)	Temperature (°C)	Density (g/mL)
18.00	0.9986	23.00	0.9975
19.00	0.9984	24.00	0.9973
20.00	0.9982	25.00	0.9970
21.00	0.9980	26.00	0.9967
22.00	0.9977	27.00	0.9965

Burette

Unlike volumetric glassware discussed above, a burette is graduated. A **burette** is a device to aliquot measurable volumes of a liquid and is shown in Picture 5. Notice that the burette is clamped to the ring stand using a burette clamp.

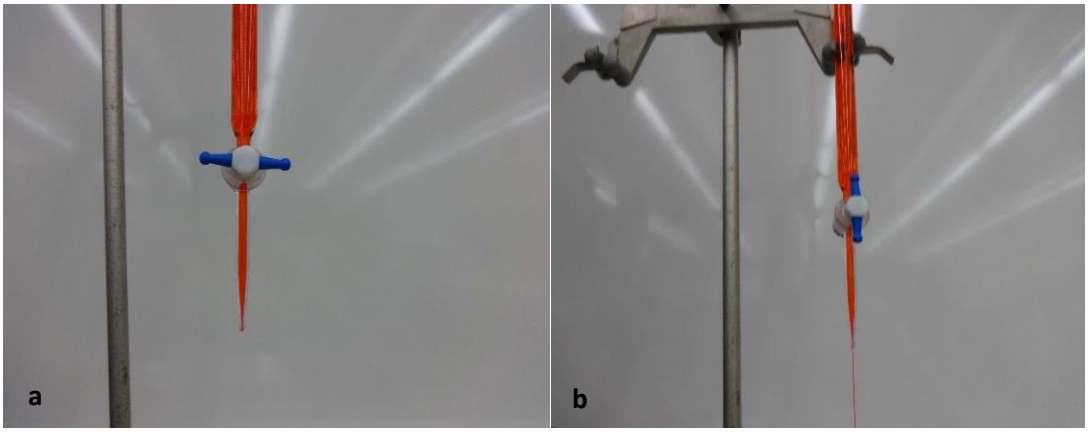
Picture 5. 50 mL burette clamped to a ring stand.



In an acid-base titration, the base, in this case sodium hydroxide solution, is added to the burette. It is very important that the burette is cleaned with water before the base is added. After the burette is cleaned with water, several (3-4) small portions (1-2 mL) of the sodium hydroxide solution should be used to rinse the burette before the burette is filled. In fact, all glassware, including volumetric glassware, should be thoroughly cleaned before a titration. Ideally, any glassware used to measure volume should also be rinsed with the solution of interest or dried before use. However, to save time, sometimes it may be necessary to use wet glassware in this course.

There are a few practical things to consider when using a burette. When the stopcock is perpendicular to the burette, it is off, as shown in Picture 6a. When the stopcock is parallel to the burette, it is on, as shown in Picture 6b. The stopcock is not a simple on–or-off device. It can be used to slowly dispense solution, including a single drop at a time, by adjusting the stopcock position.

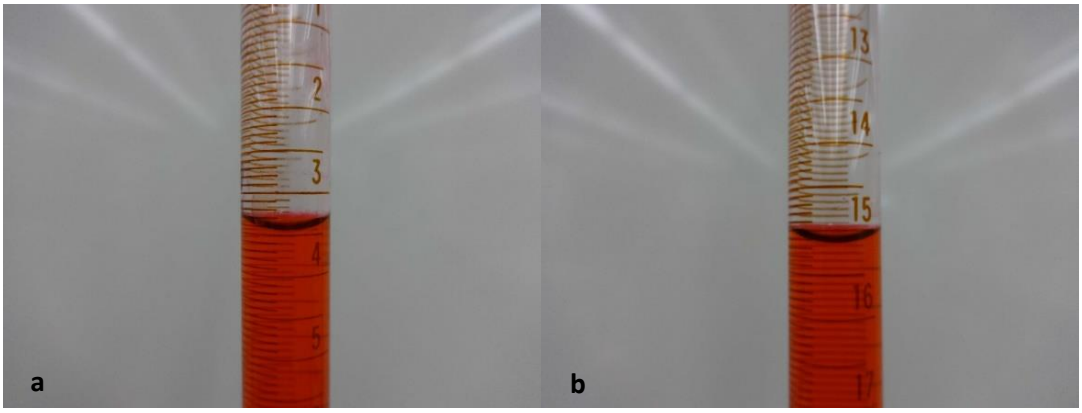
Picture 6. (a) Stopcock in the off position. (b) Stopcock in the on position.



A burette measures volume by difference. This means that there is no need to know the absolute volume of solution in a burette. Before reading the initial volume of the burette it is important to make sure there are no air bubbles in the tip. To remove the air bubbles, dispense a few milliliters of solution from the burette into a separate container.

The volume in a burette is read from the top down. For example, the burette in Picture 7a is read as 3.46 mL (top down) and not as 4.54 mL (bottom up). After some solution is let out, in Picture 7b, the burette is read as 15.26 mL and not 16.74 mL. By difference, the volume of solution delivered from the burette is 15.26 mL minus 3.46 mL which is 11.80 mL. Notice that all burette readings have two decimal places. One common mistake made with burettes is to not read them as accurately as possible. Be sure you read the burette to two decimal places, even if the last decimal place is a zero.

Picture 7. (a) Burette read as 3.46 mL. (b) Burette read as 15.26 mL.



It is a good idea to have a beaker labeled “waste” to catch any solution removed from the burette that is not part of the reaction. For example, when removing air bubbles, a different container is needed to catch the solution removed from the burette. It is also important to thoroughly rinse the burette with water after it is used. Sodium hydroxide is a non-volatile salt. If a sodium hydroxide solution is left in the burette, water will evaporate and a white solid will be left behind and clog the hole in the stopcock. In addition to

clogging the burette, solid sodium hydroxide is extremely caustic. Please rinse several portions of water through the burette into your waste beaker before returning it.

Acid- Base Titration

Now that we are familiar with the glassware used for titrations we can further discuss the theoretical and practical aspects of the titration itself. In a **titration**, a solution of known concentration, often called the **standard solution** (a titrant), is added to a solution with an unknown concentration to determine the unknown concentration or quantity of solute in the unknown. In an acid-base titration, one of the solutions is acidic and the other is basic. A neutralization reaction occurs when the solutions are added together.

In this experiment, you will perform an acid-base titration by reacting a sodium hydroxide solution of known concentration (titrant) with a hydrochloric acid solution in order to determine the concentration of the hydrochloric acid solution as shown in Reaction 1. An acid-base reaction, such as that of hydrochloric acid and sodium hydroxide, is a double-displacement reaction, where the hydrogen ion from the acid replaces the sodium ion in sodium hydroxide, and the sodium ion replaces the hydrogen in the acid. The end result is water (from the hydrogen and the hydroxide ions) and a salt (from the sodium and the chloride ions).

To begin the experiment, a 0.100 M sodium hydroxide solution is prepared. You should start by calculating what volume of a 5M sodium hydroxide solution (stock solution) is needed to make a 100 mL of 0.100 M sodium hydroxide solution. This volume is added into a 100.0 mL volumetric flask and then water is added to the calibration line. Next, the hydrochloric acid solution is made by adding approximately 3.5 mL of 3M hydrochloric acid into a 125 mL Erlenmeyer flask containing about 25 mL of water and filling to the 50 mL line. Remember; never add water to concentrated acid solutions! It is important to add the acid to an Erlenmeyer flask already containing a substantial amount of water (in this case about 25 mL). It is also important to note that the glassware used to make or dilute the HCl is not very accurate. Since the volumes are not accurate, they will not be used to find the concentration of the acid. Instead, the titration data will be used to find the concentration of the acid.

Reaction 1. Hydrochloric acid reacting with sodium hydroxide.

$$HCl\ (aq) + NaOH\ (aq) \longrightarrow NaCl\ (aq) + H_2O\ (l)$$

A volumetric pipette is used to transfer 5.00 mL of the diluted hydrochloric acid to a clean, but not necessarily dry, 125 mL Erlenmeyer flask. Use water in a squirt bottle to rinse the side of the flask to make sure all the acid solution is at the bottom of the flask and nothing left on the walls. Add an indicator, phenolphthalein, to the flask and then begin to titrate with sodium hydroxide. Sodium hydroxide in the burette should be added slowly into the flask until the indicator changes color.

In a colorimetric titration experiment like the one you are doing right now, an **indicator** is used to monitor the endpoint of a titration**.** The **endpoint** is defined as the point at which the indicator changes color. This tells us the titration is complete. In this experiment, and the next, phenolphthalein will be used as the indicator. In an acid solution, phenolphthalein retains its clear color (colorless), however, when the

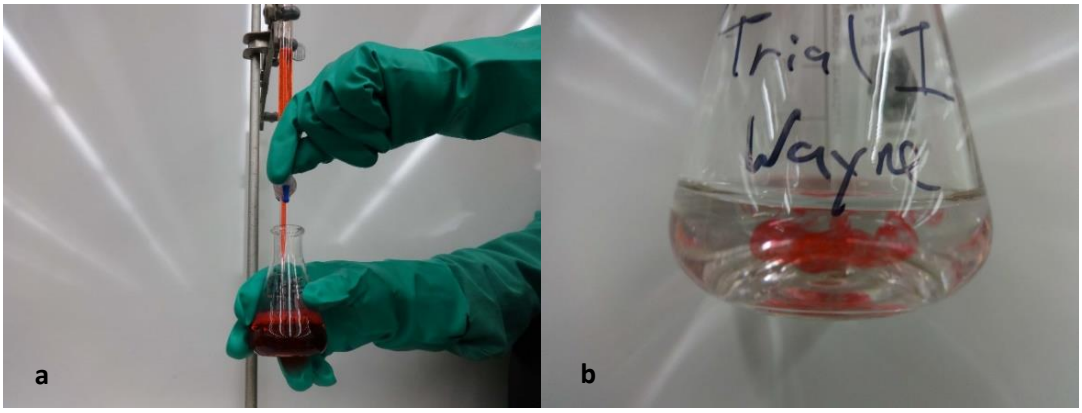
solution becomes basic phenolphthalein changes to a pink color. The endpoint is reached when phenolphthalein turns pink and the pink color persists.

In another type of acid base titration, the equivalence point is determined instead of the endpoint. An **equivalence point** is the point at which the number of moles of acid and the number of moles of base are the same (equal) in the solution. Note that the endpoint and the equivalence point are not the same thing. For phenolphthalein the endpoint occurs at a pH of around 8.2 but in the reaction of NaOH and HCl the equivalence point occurs at a pH of 7. Although pH won't be discussed in great detail in this experiment, a pH of 8.2 occurs close to the equivalence point of the acid and base used in this titration and we will consider the moles of acid and moles of base to be equal at the endpoint.

A titration is performed by making an initial reading of the burette before sodium hydroxide in the burette is added to the 125 mL Erlenmeyer flask containing the 5.00 mL of HCl solution as shown in Picture 8a. When an acid base titration is performed using phenolphthalein as an indicator, it is common to see some pink appear at the point where the base in the burette contacts the acid solution in the Erlenmeyer flask. This occurs because there is a high concentration of sodium hydroxide in that part of the solution that causes the phenolphthalein to turn pink as shown in Picture 8b. When the reaction mixture is swirled, the pink color disappears because the area of high concentration is diluted into the entire solution. As you get closer to the endpoint of the titration, the pink color persists longer and longer. A perfect titration is performed when a single drop of sodium hydroxide causes the entire solution to turn pink and remain that color.

It is very easy to over-titrate a solution. Once the endpoint is reached, the pink color does not change no matter how much more base is continue added. The color of a solution with 0.10 mL over titrated is the same as the color of a solution with 5 mL over titrated. Therefore, it is not possible to see a "correct" pink color. If more than the required amount of sodium hydroxide is added, the solution often looks very similar to when the correct amount of base is added. To avoid over-titration it is important to add the sodium hydroxide from the burette one drop at a time when the endpoint is approaching and stop the titration when the last drop turns the entire solution pink. When the titration is complete the burette is read again and the volume of NaOH solution added is found by difference.

Picture 8. (a) Performing an acid-base titration. (b) A locally high concentration of sodium hydroxide solution in the flask.



As an example, let's assume 10.47 mL of 0.100 M NaOH is used to titrate the 5.00 mL of hydrochloric acid. In Calculation 1, the concentration of the acid is calculated. Remember in a conversion using molarity, the correct unit of volume must be expressed in liters. Notice that the last step of the equation shown in Calculation 1 is not truly dimensional analysis. In this step the moles of HCl are divided by the volume of HCl solution in liters in order to find the molarity of the HCl solution.

Calculation 1. Concentration of hydrochloric acid solution.

$$0.01047\ L\ NaOH\ \times \frac{0.100\ mol\ NaOH}{1\ L\ NaOH}\ \times \frac{1\ mol\ HCl}{1\ mol\ NaOH}\ \times \frac{1}{0.00500\ L\ HCl} = 0.209\ M\ HCl$$

In this experiment, you will perform the titration of 5.00 mL of HCl solution four times to find the concentration of the HCl solution. Be sure to comment on the precision of your titrations. It will not be possible to comment on the accuracy as each group's solution will be somewhat different and there is no standard to compare the concentration to.

In an effort to be green, this lab was designed so salt water is the only product of the reaction. Please mix the solutions from all titrations as well as any leftover acid and base into a single 250 mL Erlenmeyer flask. After the solution is swirled to ensure it is well mixed, it can be poured down the drain.

Chemical Hazards and Waste

Gloves, goggles and a lab coat must be worn at all times. 5 M sodium hydroxide and 3 M HCl are hazardous, toxic and corrosive. Be sure to handle them with care. All glassware must be clean before it is used in a titration. Sodium hydroxide solutions will leave a corrosive residue behind, make sure all glassware is thoroughly rinsed after it is used. All waste should be mixed and then can be poured down the drain as described.

Experimental

Part 1: 5.00 mL Volumetric Pipette

Determine the accuracy and precision of a 5.00 mL volumetric pipette by adding five portions of water to a 50 mL Erlenmeyer flask and recording the mass of the flask and water for each trial. Use the density of water and the temperature to find the volume of water transferred for each trial. Calculate the percent error and RSD and comment on the accuracy and precision of the 5.00 mL volumetric pipette.

Part 2: Titration of HCl with NaOH

Make 0.100 M NaOH from 5.00 M solution using a 100.0 mL volumetric flask. Dilute 3.5 mL of 3 M HCl into a 125 mL Erlenmeyer flask containing about 25 mL of water and then fill to the 50 mL line. Never add water to concentrated acid solutions! Individually titrate four 5.00 mL samples of diluted acid with the 0.100 M NaOH solution. Calculate the RSD for the four titrations and comment on the precision of your experiment.