Introduction to Titrations – A Powerful Analysis Tool

Objectives

In this experiment, you will learn how to read and manipulate a burette in a technique called titration and determine the unknown concentration of a reactant.

Skills to Master

1. By yourself, be able to set up and run an accurate and precise titration. This includes completing the calculations to find the concentration of an unknown solution.

2. Understand what is meant by equivalence point and how it may differ from endpoint.

Introduction

Titration is a technique where we use a very favorable (fast) reaction between one reactant with a known concentration and a reactant with an unknown concentration. If we have the reaction stoichiometry and the exact volume needed for reaction with one reactant, we can mathematically solve for the other reactant's concentration (finding an unknown concentration). We will work to become familiar with this calculation called simply a '*titration calculation'*.

The Burette

A burette is a piece of glassware used to dispense various amounts of a reactant in an accurate manner; because of the numerous calibrated markings on this item, it is expensive (see Figure 1). The burette is suspended by a ring stand with a burette clamp. A burette stored upside down with the stopcock open is a clean burette. We must be careful not to bump the tip of the burette as it is rather fragile. If the tip gets cracked or broken, then it is no longer usable.

The first step in getting familiar with a burette is the filling step. The solution that we place in the burette is always a good reactant, such as hydrochloric acid or sodium hydroxide. Thus, we need to be cautious with the filling process. This filling solution is called the 'titrant'. It is an important solution for our work; thus, keeping the solution from getting diluted is important. Therefore, we would rinse the burette first with the titrant, before actually filling it for use.

Figure 1 The Burette held via a burette clamp on a ring stand above a beaker. This is a typical titration set-up.

Here is a simple stepwise process that you will use **every time** you use a burette:

Titration Set Up

1. Place the burette into the burette clamp with the tip down and the stopcock closed.
2. Place a funnel into the top of the burette.
3. Pour about 5 to 10 mL of the titrant solution into the burette.
4. Set the funnel aside in a place where it will stay clean.
5. Take the burette out of the clamp.
6. Roll the solution around inside to rinse the inside of the burette completely and let some out of the tip into a waste beaker.
7. Pour the rest of this rinse solution into the waste beaker.
8. Place the burette back into the clamp and replace the funnel.
9. Place the waste beaker under the burette.
10. Pour titrant into the burette almost to the top. DO NOT overfill. Overfilling will mean the titrant is spilling over and will need clean-up.
11. Open the stopcock to dispense/flush out the air in the tip. If a bubble remains in the tip, open the stopcock wide open to flush it out, and then close it as soon as the bubble is gone.
12. Next open the stopcock to drain the level down below the first mark.
13. Now read the volume level as the initial reading. It is ready to go.

Learning to read the burette is now the challenge. As you recall, to determine the volume of liquid in a burette we read the bottom of the meniscus —the curve you see resulting from the surface tension of liquid touching the sides of the glass. We always take an initial reading before starting. Then after dispensing enough of the reactant to do the job, we take a final reading. Look at the diagrams, how are they read?

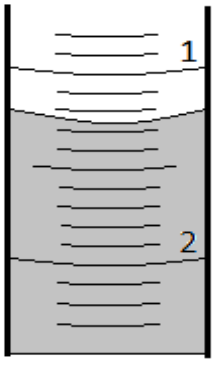

Figure 2 Reading the burette: Initial Burette (left) – The Meniscus is below 1.2, but above 1.3. Read: 1.26 mL.

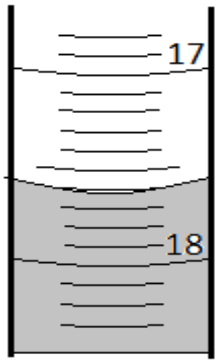

Figure 3 Reading the burette: Final Burette (right) – The Meniscus is below 17.6 but above 17.7. Read: 17.61 mL.

We are interested in what was used, not what is missing. Do not subtract from 25.00 mL (or 50.00 mL), simply write down the correct value for the initial and final volumes. Remember to count from the top of the burette when reading the volume.

Yes, reading that 2nd decimal place is important, and yes, it is our best guess. Determining the amount of titrant used is a simple subtraction problem:

$$Total\ Volume\ Dispensed\ =\ Final\ Reading\ -\ Initial\ Reading \quad (1)$$

Therefore: Total Volume = 17.61 mL – 1.26 mL = 16.35 mL Titrant Used for this Reaction

Given that you should be familiar with the idea of limiting reagents, moving on to the concept of titrations should not be all that confusing. Look at the following reaction:

$$A\ +B\ \rightarrow C\ +D \quad (2)$$

Suppose we have 0.10 L of A at 2.00 M. Let's add 1.00 M B to react all of A that is present. How many Liters of B are needed? You might be able to figure this out with logic, but we can set up a simple calculation to do this.

$$0.10\ L\ Soln.A \times \frac{2.00\ mol\ A}{L} \times \frac{1\ mol\ B}{1\ mol\ A} \times \frac{1\ L}{1.00\ mol\ B} = 0.20\ L\ Soln.B\ needed \quad (3)$$

This idea of limiting reagents helps us move on to titration reactions and calculations. Be aware that the mole ratio in the calculation comes directly from the balanced reaction equation.

The Titration Reaction

All titration reactions must be good reactions that go to essentially 100% completion. This means that titrants need to be quite reactive. As an example, hydrochloric acid is a strong acid and reacts very well with any kind of base. We aren't ready to really get into acid/base chemistry, but at least we know that HCl will easily give its one H^+ (proton) ion to any chemical that will take it. A common reaction is shown below.

$$HCl_{(aq)}\ +\ NaOH_{\ (aq)}\ \rightarrow\ \ H_2O_{(l)}\ +\ NaCl_{(aq)} \quad (4)$$

We would simply call this reaction a 1 to 1 reaction when looking at the number of moles of HCl needed to react with the NaOH. This stoichiometry relationship must be known and is critical to successfully calculating a concentration from a titration run.

So far, we know that we need a good reaction with a known stoichiometry. We also probably realize that we need to know the actual volumes that are used in the reaction. But to run the reaction, we keep adding titrant. How do we know when to stop?

The Color Change and the Indicator

All good titrations run to 100% completion, but they also must have a mechanism that tells us the exact moment when both reagents are limiting and are all used up, meaning our reaction is all done. This is the point in the titration where we MUST stop adding titrant. We use a chemical dye called an *'indicator'* for this job. It is always a dye molecule that changes color depending on which reactant is present in just a slim excess. Here is an example reaction:

Shown is an Erlenmeyer flask. (Erlenmeyer is capitalized due to it being a person's name.) The flask has exactly 20.00 mL of sodium hydroxide in it. We've added an indicator to the solution, and in NaOH, it has color (Figure 4). We don't yet know the concentration of the NaOH so it must be an unknown, but we know its volume. We were careful and used a volumetric pipet to transfer that volume into the flask.

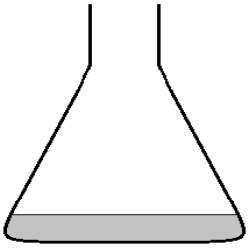

Figure 4 Erlenmeyer flask with 20.00 mL NaOH and Indicator

Now, we start to titrate with HCl from the burette. (Figure 5) Realize that in the flask is excess NaOH; thus, the solution stays colored. The H^+ from the HCl have not yet equaled the OH^- from the NaOH. HCl is the limiting reagent still at this stage.

Jump to the **equivalence point**, where the **moles OH^- = moles H^+**. This is a unique situation. If we had one micro droplet of Excess HCl, the moles of HCl are in excess. At the end point that *dye will change color* in that situation. (Figure 6) We've reached the end of the reaction. The color change will be dramatic and quick. We MUST STOP adding titrant. We call this the '*end-point'* of the titration. Now, we read the final volume value on the burette. The volume of titrant used is the value we need to calculate the moles of titrant used.

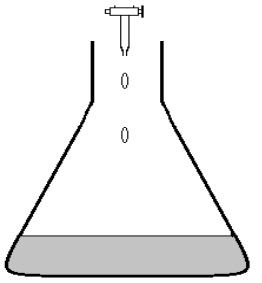

Figure 5 Titrating with HCl.

Titration Tips

- Be sure that there are no air bubbles in the tip of the burette.
- Be sure that you are reading 2 decimal places on the burette – to the hundredths place.
- Be sure to mix the reaction (swirling) as you add titrant.
- You can use deionized water to rinse the inside walls of the flask.
- By adding deionized water you will not change the number of moles of analyte in the flask. Therefore rinsing down the sides of the flask to make sure all of the reactants molecules are in the swirling liquid will produce a more accurate titration.
- Strive to set up titrations to use around 20 mL of titrant to get good precision for the 25.00 or use 40 to 45 mL for a 50.00 mL burette.

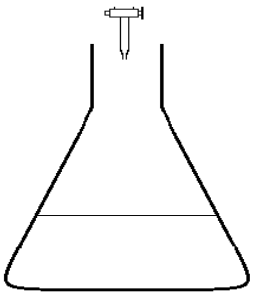

Figure 6 Color Change to clear at the End of the Titration.

The Titration Calculation

All titration calculations are performed in essentially the same format. We know the volume and concentration or moles of one reactant, which are used to determine how many moles of that reactant were needed in the reaction.

$$\frac{mol\ HCl}{L\ HCl} \times \frac{L\ HCl_{used}}{1} = mol\ HCl \quad (5)$$

With the moles of one reactant and the mole-to-mole ratio from the balanced equation, we can get the moles of the other species.

$$\frac{mol\ HCl}{1} \times \frac{mol\ NaOH}{mol\ HCL} = mol\ NaOH \quad (6)$$

Then we just need the volume where those moles of the sodium hydroxide were held. This volume is simply the sample size taken of the NaOH (typically transferred using a pipet).

$$\frac{mol\ NaOH}{L\ NaOH} = Sample\ NaOH\ Concentration\ (M) \quad (7)$$

Note: A titration calculation MUST always show the mole-to-mole ratio for the reaction. Many titrations are not a 1:1 stoichiometry. The mole-to-mole ratio also accounts for the correct units in the final answer and is from the balance reaction.

Experimental Preparation

In this experiment, you will be provided a standardized solution of NaOH (strong base) to use as your titrant. This solution has an exact, known concentration. You are also provided an unknown weak acid solution. We will just symbolize the acid as HA since it has just one acidic proton on the molecule. This acid will react in a 1:1 stoichiometry with the base. Your goal is to determine the concentration of this weak acid using titration with an indicator.

SAFETY

You are handling weak acid and strong base solution. The weak acid can be irritating. If you spill it on yourself, you should rinse it off. HOWEVER, strong base such as sodium hydroxide is very caustic. Rinse it off immediately with lots of water. If you spill the base, ask the instructor to help neutralize it so that it can be wiped up safely.

All solutions can go down the drain with water for this experiment.

EQUIPMENT AND MATERIALS AVAILABLE

Standard 0.0500 M NaOH	25.00 mL burette	Volumetric pipets
HA Unknowns – weak acids	250 mL Erlenmeyer flasks	Various beakers
Phenolphthalein indicator		

PROCEDURES

Part I: Preparation

1. Use a clean, dry beaker to obtain about 100 mL of weak acid sample. There are various unknowns. Your instructor will assign one of them to you.
2. Use a clean, dry beaker to obtain about 100 mL of the 0.0500 M sodium hydroxide solution. Be careful to mark the beakers so that their identities are clear.
3. Obtain a 25.00 mL burette. Rinse and fill the burette with the sodium hydroxide solution. Be sure to remove all air bubbles from the tip and valve before doing any titrations.

Part II: Unknown Weak Acid Titration

Rough Trial

Since the weak acid is a true unknown, it is unclear how much sample you should use. There are a variety of volumetric pipets available.

NOTE: Always use a volumetric pipet to aliquot out a specific volume of the sample. These pipets allow us to keep best available significant figures in our work.

1. Select a pipet (suggested that you start small) and then transfer that specific volume to a clean Erlenmeyer flask. This flask, rinsed with deionized water, does not need to be dry.
2. Add 2 drops of phenolphthalein indicator to the flask.
3. Add deionized water so that the overall volume is around 30 mL in the flask. Again, the actual total amount in the flask is not important. You can use a graduated cylinder to measure this pure water, deionized water, addition.
4. Once you have read the initial volume reading on the burette, begin titrating with base. Since this is a rough trial, you can let the burette run fairly fast, but keep swirling the flask while base is being added. Watch for signs of the color getting close to changing in the flask. You are doing a rough run, thus, it is not absolutely critical to stop precisely when the color changes, but you would like to be close to the volume needed to reach the end point, where the indicator changes color. All of these measurements are recorded in the 'Data' section.
5. Dispose of the contents of the flask down the large drain, clean and rinse the flask with deionized water.

Adjustment of Sample Size

With the data for the rough trial, you need to adjust the sample size volume so that the subsequent runs will use about 20 mL of titrant. Use dimensional analysis to calculate the projected amount of analyte that will require about 20 mL of titrant. Be sure that your work is shown on the Experiment Worksheet. You should be able to determine whether this approximate amount should be delivered in a precise manner with a 5.00 mL, 10 mL, 15 mL, 20 mL, or 25 mL volumetric pipet.

Preparation for Analytical Trials

Now that you have a calculated volume that is needed, you need to find a volumetric pipet that will work. Find the pipet that is closest to your calculated **analyte –sample volume** for a titration that will use about 20 mL of titrant.

1. Choose the pipet that has a volume closest to the calculated amount to transfer this calculated volume of sample to a clean Erlenmeyer flask with a pipet. Be sure to use the correct precision for that volume in your calculations. For example, an eight-milliliter volumetric pipet dispenses 8.00 mL of liquid.
2. Add 2 drops of indicator and some deionized water to bring the volume to about 30 mL.
3. Refill your burette with known base. Be sure your sample is not under the buret while filling it! Be careful not to overfill it.
4. Adjust the volume so that the meniscus is below the 0.00 mL mark. Record your initial volume.

Analytical Trials

Titrate the sample with the NaOH until the indicator begins to change color. You should already have an idea where your final volume of titrant will be based on the rough trial and adjuated analyte volume.

1. Record the initial burette volume.
2. Add titrant; slow down near the expected volume for the color change. Then add the titrant drop by drop.
3. Watch for the color change. Droplets on the sides of the flask should be rinsed down with a small amount of distilled water to make sure that the reaction is complete.
4. Strive to STOP additions right when the color makes a permanent change. The color should change and stay for over 5 seconds to be considered at the end point. It is possible after some time that the color will fade away due to reactions with the air. If you overshoot the end-point, you may have a very intense color. We are seeking to hit the end-point as close to the color change without going over. Record the final burette volume.
5. Take a photo of the solution at the end point right when you reach the end point.
6. Your solution should be disposed of in the large sink with water. Clean and rinse the flask with deionized water.
7. Repeat the analytical titration at least 2 more times using the same sample size. Be sure each student performs at least one titration, including pipetting the acid sample.

NOTE: If you found your sample size to be too large in the first analytical run, re-evaluate the volume of the weak acid sample pipet size.

The volumes of base used for each trial should be within 0.40 mL of each other. If a trial does not meet this criterion, run another trial to ensure a good mean concentration value and omit the suspect data. You should have three trials that are within this criteria. Only enter those three trials into Labflow for analysis, but you should record all of your trials in your data table. The data table has two extra columns for analytical trial data.

Clean up

1. Take all flasks to the large sink and rinse contents down the drain with lots of water.
2. Wash the flasks with glassware hot soapy water and rinse with tap water.
3. When all the soap is rinsed out, rinse with deionized water.
4. Take the burette to the sink and empty it, rinsing the solution down with lots water.
5. Rinse the burette, including the tip, several times with deionized water.
6. Carefully hang the burette upside down in the clamp with the stopcock open.
7. Rinse all of the pipets you used with deionized water.
8. Fill the DI water bottle that is at your lab bench.
9. Return clean equipment to the side bench or bin/drawer depending on where you got it let it air dry.
10. Check the bin/drawer at your station and make sure all of the items were returned to it. If materials are missing let your instructor know so that they can be replaced before the next class.
11. Clean the common area assigned to you by your instructor.
12. Put your personal items away.
13. Clean your bench and items that couldn't be washed with the disinfectant spray. Let the disinfectant spray air dry, do not wipe.
14. Wash your hands before you leave the lab.

Acknowledgements

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