Chemical Technology Program
General Education & Sciences Division
Cape Fear Community College
Wilmington Campus

C-TEACH ACADEMY
BRIDGING HIGH SCHOOL, COLLEGE, AND INDUSTRY

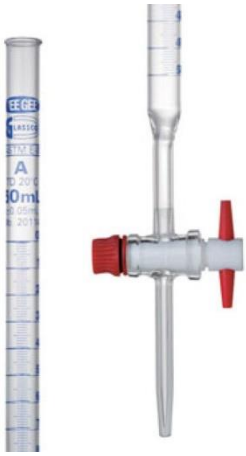
Lab Activity: Basics
USE OF BURETTES

Use of a Burette

Chemical Technology Program, Cape Fear Community College, Written by Tracy L. Holbrook

This laboratory experiment is designed to introduce you to a burette. Burettes are commonly found in analytical laboratories to measure delivery of liquids. Typically, traditional burettes hold 50.00 mLs and are used heavily for titrations (the process of adding two solutions together in order to determine concentration). You will use burettes in many different types of laboratories. While some of the procedures may seem trivial and the results obvious, this is a chance for you to develop good laboratory techniques.

"Modern" Burette



Stopcock of a Burette

"Original" Burettes

TERMINOLOGY:

Burette: *Devices commonly used in an analytical laboratory to dispense variable, measured amounts of a chemical solution. A volumetric burette delivers measured volumes of liquid. Most burettes will hold less than 50.00 mLs of solution. Burettes may come in many forms such as plastic or glass. Burettes may be ordered in different "classes", similar to volumetric pipettes. General Cost: $65.00 – $250.00 each.*

Stopcock: *A form of valve used to control the flow of a liquid or gas. Stopcocks for burettes are conical in shape and may come in Teflon or glass forms. They traditionally have a plastic retainer, a rubber washer, and a nut that tightens the stopcock to the burette. Glass stopcocks typically require grease to ensure a leak-free seal. Never overtighten the stopcock.*

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Titration: *One of the most common wet laboratory techniques. It is a method of quantitative analysis that is used to determine the unknown concentration of an identified analyte. Since volume measurements play a key role in titration, it is also known as volumetric analysis.*

Titrant: *A reagent, or liquid solution that is prepared by a laboratory analyst. The concentration of the solution is "known" and the solution is used to determine the concentration of an unknown sample.*

Standard Deviation: *A calculation that informs you how "close your numbers are". If your measurements are close to each other, your standard deviation will be LOW. If your measurements are NOT close to each other, your standard deviation will be HIGH.*

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(x_i - \mu)^2}$$

Good news! EXCEL will do this for you, so we will use it!

Types of Titrations:

Biodiesel Titration	Waste Vegetable Oil (WVO) must be neutralized before biodiesel may be processed and used.
Kjeldahl Method	Measures nitrogen in a sample; in food chemistry, the nitrogen content is related to protein.
Saponification Value	Measures the amount of fat present in a sample; may also be used in soap making
Winkler Test	Used to determine oxygen content in water.
Vitamin C	Provides vitamin C (ascorbic acid) content in a sample
Benedict's	Quantifies glucose in urine which may indicate diabetes
Karl-Fisher	Analyzes trace amounts of water in a substance.

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IN CLASS PROBLEM:
Suppose a technician is verifying the accuracy of a burette. The technician delivers 26.52 mLs of water, a total of six times. He weighs each sample (density of water at room temperature is a little less than 1.00 g per milliliter; therefore, the sample should weigh 25.5200 g).

Weights:	25.5441 g	25.3213 g	25.5101 g
	24.9906 g	25.4912 g	25.5997 g

a. What is the average mass?
b. What is the standard deviation?
c. The burette has a "specification sheet" that states it should be delivering a volume of water within +/- 0.02 mLs. Does the analyst "pass" or "fail" on their delivery?

PART A - USE OF THE BURETTE
Most burettes in the lab are "Class A" which are some of the most precise, accurate and reliable pieces of glassware to deliver quantities of volume. Although there are inherent limits in the accuracy and precision of these burettes most delivery errors are caused by incorrect handling of the sample or the burette itself. In this experiment you will practice using a burette and minimizing the errors associated with your technique.

GENERAL PROCEDURE FOR USING A BURETTE

1. To fill a burette, close the stopcock at the bottom and use a funnel. Lift up on the funnel slightly to allow the solution to flow in freely.
2. Condition the burette with the titrant solution. Place a small amount of titrant in the burette. Place your finger over the top and rock back and forth. Allow the titrant to drain. Repeat the process two to three more times.
3. As the liquid drains out of the burette tip, check to make sure that no air bubbles are present.
4. Ensure that the stopcock is not leaking. A replacement could be necessary if the leak cannot be fixed.
5. Make sure that you read the bottom of the meniscus when reporting volume.
6. When you are ready to deliver the liquid, turn the stopcock slowly. The solution should be delivered quickly until the endpoint is reached.
7. As you approach the endpoint, slow down the delivery of liquid. Begin to add the liquid drop by drop until the indicator changes.

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In this procedure you will deliver various amounts of water into a container using a burette. The sample will be weighed. You will then report the weights to the laboratory instructor to ensure that you are properly delivering the correct amount of liquid. Unless instructed otherwise, you should handle the liquid sample with care! Measurements on the balance should be made to the nearest 0.1 mg (0.0001 g). **If your balance does not read to FOUR decimal places, please see a laboratory instructor.**

PROCEDURE: Using a Burette to Deliver 5.00 mLs

1. TARE the analytical balance. The mass should read "0.0000 g". Place a weighing bottle on the balance and record the mass.
2. Fill the burette with water to the "0.00 mL" mark. Ensure that you are reading the bottom of the meniscus.
3. Place the weighing bottle under the prepared burette. Slowly open the stopcock and deliver 5.00 mLs of water into the container.
4. Weigh the container and water sample. Record weight on your data sheet. Calculate the mass of water delivered into the container by subtracting the mass of the empty weighing bottle.
5. Return to your burette. Add another 5.00 mL of liquid into the container (do not empty it from the previous addition).
6. Once again, TARE the analytical balance. Place your container on the balance and obtain a mass of this new addition. It should be greater than the last mass you obtained. Calculate the mass of water delivered into the container by subtracting the mass of the weighing bottle from the previous measurement from the current mass.
7. Repeat this process three more times.
8. Make sure that you record your values in the data sheet.

PROCEDURE: Using a Burette to Deliver 10.60 mLs

9. Repeat the process from above, using 10.60 mLs instead of 5.00 mL.

PROCEDURE: Using a Burette to Deliver 12.85 mLs

10. Repeat the process from above, using 12.85 mLs instead of 5.00 mL.

ONCE YOU COMPLETE ALL OF THE VOLUME DELIVERIES ABOVE, PROVIDE YOUR DATA TO A LABORATORY INSTRUCTOR. IF YOUR DATA "PASSES" YOU HAVE COMPLETED PART A. THE LABORATORY INSTRUCTOR WILL THEN PROVIDE YOU A BEER SAMPLE FOR TITRATION. IF YOUR DATA "FAILS", YOU WILL NEED TO ATTEMPT THE DELIVERY AGAIN.

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PART B – AN INTRODUCTION TO TITRATION

In this procedure you will titrate beer samples for their acidity values. Acidity is a major "driving force" in the taste and "degree of sharpness" of beer. Many organic acids and carbonic acid (carbon dioxide gas), when dissolved in beer, determine the acidity level. When beer is analyzed in the laboratory, the acidity (percentage of acid in a sample) is expressed as if all the acidity were present as lactic acid, but this is done for convenience; the acidity figure includes any acid present following degassing to eliminate carbon dioxide.

Almost all drinks that are considered refreshing and "drinkable" contain some notable acidity as part of a balance against sweet elements. Measurements of 0.1% to 0.3% acidity (expressed as lactic) are typical in beer, which would calculate out to 1000 to 3000 ppm. All-malt worts produce higher amounts of acidity in beer than do malt-adjunct worts. It is a generally assumed rule of thumb that most typical malt-adjunct beers will show about 0.1% acidity and all-malt beers closer to 0.2%. Light beers can be as low as about 0.07% acidity (or 700 ppm).

Since carbonation levels in beer lends to the acidity content, degassing the beer will help an analyst eliminate this source of acid and focus on other sources of acidity. Abnormally high acidity can be an indication of bacterial infection of wort and/or beer. Microbial contamination issues leading to abnormal acidity are usually perceived by the brewer or consumer before any testing would show the defect. Acidity in beer actually helps protect it because many pathogenic and food-spoilage microorganisms are unable to grow in high-acid (low pH) environments. To some extent the acidic nature of beer, along with the carbon dioxide (carbonic acid acidity), lack of oxygen, and the presence of significant amounts of alcohols, has helped make beer a safe, potable beverage throughout history.

One of the simplest and most effective ways to measure total acid content in beer is by the titration method. The brewer (or laboratory analyst) slowly adds a small amount of reagent (a base called sodium hydroxide, NaOH, whose concentration is known) to the beer sample until a change in color occurs due to the presence of an indicator.

To begin the test, a specific volume of beer is pipetted and diluted into a sample container. Next, a couple of drops of indicator and water are added to the solution and mixed well. The sodium hydroxide, via a burette, is slowly added into the beer sample. The color of the liquid will momentarily change upon the addition of reagent. If you are using a light-colored beer, the color change will be pink and if testing a dark beer, the color could be undetectable (without diluting the sample). As long as the color goes back to the original color, keep adding the titrant until the color change is permanent. When the color DOESN'T

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go away, stop and determine the amount of titrant used. The amount of titrant will correlate to the amount of acid in your sample. The more acid in the sample, more titrant will be required.

PROCEDURE: Titration of Beer for Total Acid Content
Method Adapted from AOAC: 950.07: Acidity (Total) of Beer. All abbreviations are kept in their original forms.

1. Bring 250 mL H_2O to bp and continue boiling 2 min.
2. From a pipet, add 25 mL beer previously decarbonated.
3. After emptying the pipet, continue heating 60 sec, regulating heat so that soln resumes boiling during final 30 sec.
4. Remove from heat, stir 5 sec, and cool rapidly to room temp.
5. Add 0.5 mL 0.5% phthln via micropipette or glass pipet.
6. Fill your burette with 0.10 N NaOH (standardized solution prepared by the laboratory instructor). Ensure that you rinse the burette with the titrant, dispense, and check for air bubbles in the tip!
   Hint: N = normality which is a concentration unit used for solutions in a laboratory environment. The actual concentration will be provided to you on the day of the lab. It is important that you use the ACTUAL concentration and not just "0.1" in your calculations.
7. Titrate with 0.1 N NaOH. Titrate to faint pink.
8. For beers with dark colors, a color change might not be detected. An alternative method should be used for these samples and will not be addressed here.
9. To calculate the % acidity (lactic acid), use the following equation:

$$\%\ acidty\ (ppm) = \frac{N * V * 9.01}{X}$$

N = EXACT concentration of NaOH titrant (on bottle or whiteboard)
V = volume (mL) of NaOH required to titrate
X = sample size of beer in mLs (25.00 mL)

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10. Repeat the entire procedure with a second beer sample. This second sample will be from a different supplier or different type.

11. Repeat the entire procedure again, this time with a quality control sample! The quality control sample will be a solution of known concentration (of lactic acid) that the lab has prepared for you (but you will not know the exact concentration). Can you obtain the correct value based on your laboratory skills? We must test your titration technique!

PROCEDURE: Titration of Beer for Total Acid Content
Method Adapted from AOAC: 950.07: Acidity (Total) of Beer

Automated Titrator Method: Some laboratories will be equipped with an automated titrator. If you have access to an automated titrator, please prepare a beer sample for the analysis. The laboratory instructor will provide you with details directions on how to use the instrument.

12. Under instructor guidance, use the automated titrator to analyze the sample of beer that has been provided to you. This instrument will automate titrations! A few clicks, and you are done!

13. Repeat with the Quality Control (QC) sample.

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DATA SHEET

Part 1: Using a Burette to Deliver 5.00 mL

Mass of Empty Container #1: ..... _____ g

Mass of Empty Container and 1st 5.00 mL..... _____ g

Difference for First Addition: ..... _____ g

Mass of Empty Container and 2nd 5.00 mL..... _____ g

Difference for Second Addition: ..... _____ g

Please subtract the mass of the second addition from the mass of the first addition.

Mass of Empty Container and 3rd 5.00 mL..... _____ g

Difference for Third Addition: ..... _____ g

Please subtract the mass of the third addition from the mass of the second addition.

Mass of Empty Container and 4th 5.00 mL..... _____ g

Difference for Fourth Addition: ..... _____ g

Please subtract the mass of the fourth addition from the mass of the third addition.

Mass of Empty Container and 5th 5.00 mL..... _____ g

Difference for Fifth Addition: ..... _____ g

Please subtract the mass of the fifth addition from the mass of the fourth addition.

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Part 2: Using a Burette to Deliver 10.60 mL

Mass of Empty Container #1: ..... _____ g

Mass of Empty Container and 1st 10.60 mL..... _____ g

Difference for First Addition: ..... _____ g

Mass of Empty Container and 2nd 10.60 mL..... _____ g

Difference for Second Addition: ..... _____ g
Please subtract the mass of the second addition from the mass of the first addition.

Mass of Empty Container and 3rd 10.60 mL..... _____ g

Difference for Third Addition: ..... _____ g
Please subtract the mass of the third addition from the mass of the second addition.

Mass of Empty Container and 4th 10.60 mL..... _____ g

Difference for Fourth Addition: ..... _____ g
Please subtract the mass of the fourth addition from the mass of the third addition.

Mass of Empty Container and 5th 10.60 mL..... _____ g

Difference for Fifth Addition: ..... _____ g
Please subtract the mass of the fifth addition from the mass of the fourth addition.

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Part 3: Using a Burette to Deliver 12.85 mL

Mass of Empty Container #1: ..... _____ g

Mass of Empty Container and 1st 12.85 mL..... _____ g

Difference for First Addition: ..... _____ g

Mass of Empty Container and 2nd 12.85 mL..... _____ g

Difference for Second Addition: ..... _____ g

Please subtract the mass of the second addition from the mass of the first addition.

Mass of Empty Container and 3rd 12.85 mL..... _____ g

Difference for Third Addition: ..... _____ g

Please subtract the mass of the third addition from the mass of the second addition.

Mass of Empty Container and 4th 12.85 mL..... _____ g

Difference for Fourth Addition: ..... _____ g

Please subtract the mass of the fourth addition from the mass of the third addition.

Mass of Empty Container and 5th 12.85 mL..... _____ g

Difference for Fifth Addition: ..... _____ g

Please subtract the mass of the fifth addition from the mass of the fourth addition.

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Part 4: Titrating a Beer Sample 1

Initial Volume of NaOH for Trial #1: ..... _____ g

Final Volume of NaOH for Trial #1: ..... _____ g

Difference for #1..... _____ g

Part 4: Titrating a Beer Sample 2

Initial Volume of NaOH for Trial #1: ..... _____ g

Final Volume of NaOH for Trial #1: ..... _____ g

Difference for #1..... _____ g

Part 5: Titrating a Quality Control Laboratory Sample

Initial Volume of NaOH for Trial #1: ..... _____ g

Final Volume of NaOH for Trial #1: ..... _____ g

Difference for #1..... _____ g

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CALCULATIONS & QUESTIONS:

1. For Part 4, calculate the % acidity for the beer samples based on your titration value (milliliters of titrant used). However, why not have everyone in the class to write down their volumes and use an average value? Equation is provided in the procedure.

   % Acidity, ppm for Sample 1: _____ %

   % Acidity, ppm for Sample 2: _____ %

2. Does your beer sample meet the thresholds (typical range) described in the background?

3. For Part 5, calculate the % acidity of the QC sample based on your value (eq (or classroom average).

4. Compare your results with those from the automated titrator. How do they compare?

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