

## Experiment 6 Amount of Active Ingredient in Aspirin (Two Lab Periods)

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Version 5

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A sodium hydroxide solution will be prepared using serial dilution, and standardized by titration with hydrochloric acid. The sodium hydroxide will then be used to determine the amount of active ingredient in a commercial aspirin.

### Objectives

- Perform a titration.
- Perform serial dilutions to obtain desired solution concentrations.
- Employ stoichiometric relationships and titration to determine an unknown quantity.

### Learning Outcomes

Students will be able to:

- Apply proper rounding rules and rules of significant figures in calculations.
- Employ dimensional analysis (factor-label method) for unit conversions.
- Evaluate chemical quantities related to compounds (mass, molar mass, moles, and molecules/formula units).
- Identify and apply the concept of solution concentration.
- Apply the concept of serial dilutions.
- Prepare and complete labs to apply practical chemical concepts.
- Master essential laboratory techniques critical for laboratory science (titration and serial dilutions).
- Use scientific reasoning skills (such as observing, measuring, inferring, and predicting) to solve a solution to a simulated real-world situation.

### Definitions

- **Acid-base reaction** – see neutralization reaction
- **Acids** – molecular compounds that form hydrogen ions,  $H^+$ , in water
- **Analyte** – the component of the sample to be studied or measured
- **Bases** – substances that form hydroxide ions,  $OH^-$ , in water
- **Concentration** – a measure of the amount of solute compared to solvent, often reported in molarity, M (mol/L), moles of solute in liters of solution

- **Endpoint** – when the indicator changes color during a titration; it is an estimate of the equivalence point, but often a little different (In this reaction, the indicator, phenolphthalein, turns pink in the presence of base so the equivalence point must be passed to have excess NaOH lead to color change.)
- **Equivalence point** – the stoichiometric point of the reaction, where the reagents have reacted completely
- **Indicator** – a substance that gives a visible change to show the presence or concentration of a chemical species
- **Neutralization or acid-base reaction** – when acid reacts with a base to form water and a salt
- **Standard solution** – a solution where the concentration is known to a great precision
- **Stock solution** – a solution that has a higher concentration than needed, used to prepare working solution(s) by serial dilution(s)
- **Titrant** – reagent used to carry out a titration, often the solution of known concentration
- **Titration** – process in which a solution of known concentration is reacted completely with a chemical of unknown amount
- **Working solution** – a solution prepared from the stock solution that we want to use in the experiment

## Introduction

How do we know if medicines actually have the active ingredients that they are supposed to? Often times, we cannot detect if a medicine is present or working - certainly not by looking at it. The U.S. Food and Drug Administration (FDA) has Current Good Manufacturing Practices that create standards for ensuring pharmaceutical quality. By following the standards, the identity, strength, quality, and purity of drug products is assured. However, it is up to each manufacturer to choose how the standards are implemented. In addition, it is up to each manufacturer to report their compliance with the standards. The FDA does very little testing itself. It only reviews the results provided by pharmaceutical companies.

You have just been hired as a quality control expert for Pain Be Gone, a pharmaceutical company that makes aspirin. You need to check the latest lot of your company's aspirin to verify the amount of active ingredient. How would you measure active ingredients in a commercial aspirin?

## Aspirin

Aspirin, also known as acetylsalicylic acid, is a medication taken to temporarily relieve pain and fever. It has been promoted as pain reliever for a wide range of maladies as can be seen in the label depicted in Figure 1. Acetylsalicylic acid (see Figure 2) is a weak acid. Recall that **acids** are molecular compounds that produce hydrogen ions,  $H^+$ , in water (pH is a measurement of the concentration of hydrogen ion).



Figure 1. Old Newspaper Advertisement of Bayer Aspirin.

Photo credit: Ads in History: 1926 Bayer Aspirin<sup>1</sup>, used under the Creative Commons license.

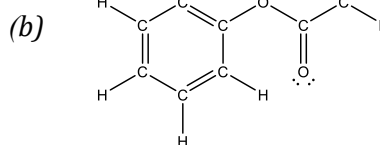
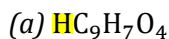


Figure 2. Acetylsalicylic acid.

(a) Chemical formula. (b) Structural formula. (The acidic hydrogen has been highlighted in yellow.)

When ingested, acetylsalicylic acid remains intact in the acidic stomach but in the basic medium of the upper intestinal tract, it forms the salicylate and acetate ions.<sup>2</sup> Aspirin selectively inhibits the production of cyclo-oxygenase 1 and cyclo-oxygenase 2, which are enzymes responsible for inflammation and pain<sup>3</sup>. (It is interesting to note that historical accounts related to treating illnesses by ingesting extracts of salicylate-containing plants date back thousands of years. For example, about 2400 years ago, Hippocrates recommended treating eye diseases and pain with juices of poplar tree and willow bark, respectively, both of which are salicylate-containing plants.<sup>4</sup>)

Different strengths of aspirin are based on the amount of acetylsalicylic acid present. In this experiment, you will measure this amount by using a quantitative technique known as titration. The process of titration allows determination of the concentration of an unknown acid or base by adding the exact amount of a base or acid to neutralize the other. A **neutralization** or **acid-base reaction** happens when an acid reacts with a base to form water and a salt. The balanced molecular equation for the reaction of acetylsalicylic acid and sodium hydroxide is shown in Equation 1 and in Figure 3.

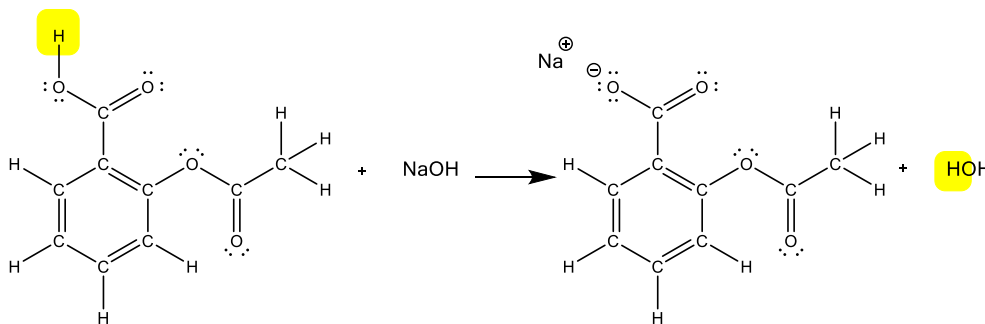
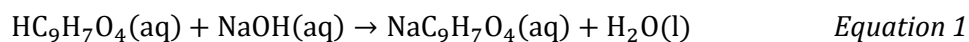


Figure 3. Structural representation of the reaction of acetylsalicylic acid with sodium hydroxide.

## Titration

To determine the amount of acetylsalicylic acid present in an aspirin tablet, the easiest process would be to dissolve a tablet and titrate it with a base. **Titration** uses a solution of known concentration to react completely with an analyte to determine its concentration or amount. Titrations are usually set up with the solution of known concentration in a buret as seen in Figure 4 (though in Part A of this experiment the solution of unknown concentration will be in the buret). The solution of unknown concentration, the analyte, is usually placed in an Erlenmeyer flask. Therefore, you might dissolve the aspirin tablet in water and slowly add 0.200 M NaOH to it until you reach the stoichiometric ratio, the **equivalence point** of the reaction. The **endpoint** of the titration, is shown with an indicator that changes color. From the known concentration and its volume used to reach the end point, the moles of the known solution are calculated, then the mole-to-mole ratio from the balanced reaction is used to determine the moles of the active ingredient. Finally, the mass of the active ingredient is found from its moles by use of its molar mass. Exercise 1 below, illustrates this process.



**Buret:** contains **titrant**, the solution of known concentration.

Graduations allow precise measurement of the volume added.

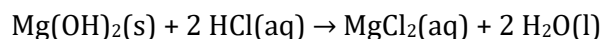
Erlenmeyer flask: contains **analyte**, the solution of unknown concentration.

**Indicator** is added to show the endpoint of the titration with a color change.

Figure 4. Components of a Titration

**Exercise 1:** A solution of magnesium hydroxide was titrated with a hydrochloric acid solution. What was the mass of  $\text{Mg}(\text{OH})_2$  in the solution if the titration required 71.10 mL of 0.150 M HCl?

The balanced equation for this reaction is:



*Solution:*

a) Find moles of HCl that reacted by using the molarity equation:

Concentration is usually reported in molarity, M:

$$M = \frac{\text{amount of solute (in mol)}}{\text{volume of solution (in L)}} \quad \text{Equation 2}$$

Rearranging equation 2 to solve for moles:

$$? \text{ moles HCl} = M_{\text{HCl}} \times \text{Volume HCl}_L = \frac{0.150 \text{ mol}}{\text{L}} \times 0.07110 \text{ L} = 0.010\bar{6}65 \text{ mol HCl}$$

b) Find mass of  $\text{Mg}(\text{OH})_2$ :

$$? \text{ g Mg}(\text{OH})_2 = 0.010\bar{6}65 \text{ mol HCl} \times \frac{1 \text{ mol Mg}(\text{OH})_2}{2 \text{ mol HCl}} \times \frac{58.326 \text{ g Mg}(\text{OH})_2}{1 \text{ mol Mg}(\text{OH})_2} = 0.311\bar{0}23 = 0.311 \text{ g}$$

## Titration of the Aspirin

There are a few steps that will lead to analysis of the aspirin. First, you need to prepare a known concentration of sodium hydroxide. To do this, you will be given a stock solution of approximately 1.6 M NaOH. You will dilute it to approximately 0.1 M NaOH and then standardize the diluted solution by titration with hydrochloric acid (HCl). A standard solution of HCl (approximately 0.15 M) will be provided; its exact concentration will be written on the carboy or bottle. (A standard solution is a solution whose concentration is known with a high precision.) Finally, you will dissolve the aspirin and titrate it with the NaOH solution you prepared to determine the amount of acetylsalicylic acid per tablet.

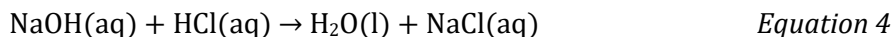
These steps lead to the following series of chemical and mathematical equations.

- 1) Dilute NaOH: for two solutions 1 and 2,

$$M_1 \times V_1 = M_2 \times V_2 \quad \text{Equation 3}$$

where:  $M$  is molarity  
 $V$  is volume, in this equation it can be in L or mL as long as both volumes are in the same unit

- 2) Standardize NaOH using standard HCl:



- 3) Titrate the acetylsalicylic acid in the aspirin with sodium hydroxide. See Equation 1.
- 4) Calculate the mass of acetylsalicylic acid present in the aspirin. See Equation 9.

The analyses will be performed in triplicate. Report the mean and standard deviation using correct units and correct significant figures (see [Appendix 8](#)). Average or mean ( $\bar{x}$ ) can be calculated by adding together values for all trials and dividing by the number of trials, as seen in Equation 5. Standard deviation will measure precision by showing the variation in a set of values around the mean. Equation 6 below has the formula for standard deviation.

$$\text{Mean: } \bar{x} = \frac{x_1 + x_2 + x_3 + \dots + x_n}{n} \quad \text{Equation 5}$$

$$\text{Standard deviation: } s = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n-1}} \quad \text{Equation 6}$$

where:  $n$  is the number of trials

$x_i$  is the individual result of each trial

Note: Standard deviation is usually reported to the same decimal places and unit of measure as the mean.

In addition, you will calculate the relative standard deviation (RSD, see Equation 7, [Appendix 8](#)), and the relative percent error (see Equation 8, [Appendix 8](#)).

$$RSD = \frac{s}{\bar{x}} \times 100 = \% \quad \text{Equation 7}$$

(If  $RSD < 5\%$ , you were precise in your measurements.)

$$\text{Relative \% error} = \left| \frac{\text{true value} - \text{experimental value}}{\text{true value}} \right| \times 100 = \% \quad \text{Equation 8}$$

where: *true value* is the expected or theoretical value  
*experimental value* is the calculated value based on data.

If relative % error  $\leq 5\%$ , the amount of active ingredient is acceptable.

## Techniques

- [Technique 1](#): Cleaning glassware
- [Technique 2](#): Using a balance
- [Technique 4](#): Using a graduated cylinder
- [Technique 3](#): Transferring liquids
- [Technique 5](#), [Video Tech. 5](#): Using a volumetric pipet
- [Technique 6](#): Using a volumetric flask
- [Technique 11](#): Disposing chemical waste
- [Technique 16](#), [Video Tech. 16](#): Filtration by gravity
- [Technique 22](#), [Video Tech. 22](#): Titration
- [Technique 23](#): Fan-Folding Flat Filter Paper



## List of Chemicals

- 1.6 M NaOH
- 0.10 M NaOH
- 0.15 M HCl
- Phenolphthalein indicator
- 95% Ethyl alcohol
- Commercial Aspirin, several brands

## List of Equipment and Glassware

### Week 1:

- two 50-mL beakers, one 150-mL beaker
- one 25-mL volumetric pipet
- one 15-mL volumetric pipet
- pipet bulb
- one 50-mL volumetric flask
- one 200-mL volumetric flask
- one watch glass
- 3 Parafilm squares, or 3 watch glasses
- one magnetic stir bar
- one magnetic stirrer
- one buret
- one buret stand
- one buret clamp
- one small funnel
- one clean, dry plastic bottle
- three 125- or 250-mL Erlenmeyer flasks

### Week 2:

- one mortar and pestle
- six 125-mL or 250-mL Erlenmeyer flasks
- three medium-sized funnels
- three fluted filter paper (or three flat filter papers)
- one 10-mL or 25-mL graduated cylinder
- 3 Parafilm squares, or 3 watch glasses
- one buret
- one buret stand
- one buret clamp
- one small funnel
- one magnetic stir bar
- one magnetic stirrer

## Experimental Procedure

This experiment will take two weeks for completion. Part A will be done the first week. Report section for Part A is due at the beginning of week 2. Part B will be done the second week.

### *Part A Determination of NaOH(aq) concentration using standardized HCl(aq)*

**Procedure summary:** You will prepare a working solution of NaOH (that you will label as NaOH Solution B) by performing serial dilutions. You will then determine the concentration of the NaOH Solution B by titration using as titrant the standardized HCl solution provided (see Equation 4).

Since the stock solution of NaOH is too concentrated, you will need to perform two serial dilutions. Use the NaOH provided to prepare a dilute NaOH Solution A, then use NaOH Solution A to prepare an even more dilute NaOH Solution B. NaOH Solution B is the working solution. Samples of NaOH Solution B will be titrated using the standardized HCl solution to determine its concentration. This procedure also prepares sufficient NaOH Solution B to perform the aspirin analysis in Week 2.

Record the data of Part A Data Table 1.

#### **1. Prepare the NaOH Solution A and NaOH Solution B (serial dilutions).**

Obtain about 45 mL of the stock solution of NaOH in a clean, dry 50mL beaker. Record its concentration, which is written on the carboy or bottle.

- a) NaOH Solution A: Using a 25-mL volumetric pipet (condition it first), deliver 25.00 mL of NaOH stock solution into a 50-mL volumetric flask. Fill the flask to the line with deionized water, cap, and invert to mix. Label the flask as NaOH Solution A.
  - i. Calculate the concentration of this solution and record this theoretical value.
- b) NaOH Solution B:
  - i. Re-condition the above 25-mL pipet:
    1. Rinse the pipet twice with deionized water.
    2. Pour about 15 mL NaOH solution A into a dry 50-mL beaker and use it to rinse the pipet twice with a few milliliters of NaOH solution A.
  - ii. Place the pipet directly in the volumetric flask containing NaOH Solution A, fill it and deliver 25.00 mL of NaOH Solution A into a 200-mL volumetric flask.
  - iii. Fill the 200-mL volumetric flask to the line with deionized water, cap, and invert to mix.
  - iv. Calculate the concentration of this solution and record this theoretical value.
  - v. Transfer the NaOH Solution B into a clean, dry plastic bottle. If the bottle is not dry, rinse it first with three small portions of NaOH Solution B. Close with cap. Using labeling tape, label the bottle as "NaOH Solution B", the theoretical concentration, your names, and the date. Set aside for now.



[Technique 3](#)  
[Technique 5](#)  
[Video Tech. 5](#)  
[Technique 6](#)

#### **2. Prepare the HCl Standard samples that will be titrated.**

- a) Pour approximately 80 mL of the HCl Standard solution into a 150-mL beaker. Label and cover it with a watch glass. Record the concentration of the standardized HCl solution.

- b) Obtain three 125-mL or 250-mL Erlenmeyer flasks (if there are no more Erlenmeyer flasks available, use beakers), and a 15-mL volumetric pipet. Label the flasks as HCl Trial 1, HCl Trial 2, and HCl Trial 3.
- c) Using the 15-mL volumetric pipet (condition it first), deliver 15.00 mL of the HCl standard into each of these three Erlenmeyer flasks. Add 2-3 drops of phenolphthalein indicator to each flask. Cover them with Parafilm or watch glasses. Set aside for now.

3. **Titration set-up.**

- a) Obtain a buret, a buret stand, buret clamp, a small funnel, a magnetic stirrer (if using magnetic stirrer/hot plate, make sure the heating element is off), and one magnetic stir bar.
- b) Place the buret clamp on the stand, and position the magnetic stirrer so that the buret may be suspended as close as possible over the center of the magnetic stirrer. See Figure 5.
- c) Condition the buret with the NaOH Solution B (use a funnel to pour the NaOH into the buret). Then add NaOH Solution B to the buret until the bottom of the meniscus is close to the 45-mL gradation mark. Suspend the buret with the clamp, and place the NaOH Solution B underneath it. Open the buret valve so the tip fills up with the solution. Do not allow it to completely empty out. Look closely just below the valve. If there is an air bubble, tap the buret while the valve is open, to see if it comes out. If not, ask your professor for help.
- d) Add NaOH Solution B into the buret until it is close to the 0 mL mark. Remove the funnel.



[Technique 22](#)  
[Video Tech. 22](#)

*Golden Lab Rule:*  
Place funnel in a beaker or on a watch glass after use.

4. **Titrate the samples of HCl Standard using NaOH Solution B.**

- a) Add a magnetic stir bar into the HCl Standard - Trial 1 (Part A-2), place it on the magnetic stirrer, and turn the stirrer on so it mixes the solution quickly without splattering (or mix manually after each addition if not using the stirrer). Lower the buret until its tip is barely below the rim of the Erlenmeyer flask.
- b) Make the initial NaOH volume reading. Read the buret to a precision of  $\pm 0.01$  mL and record.

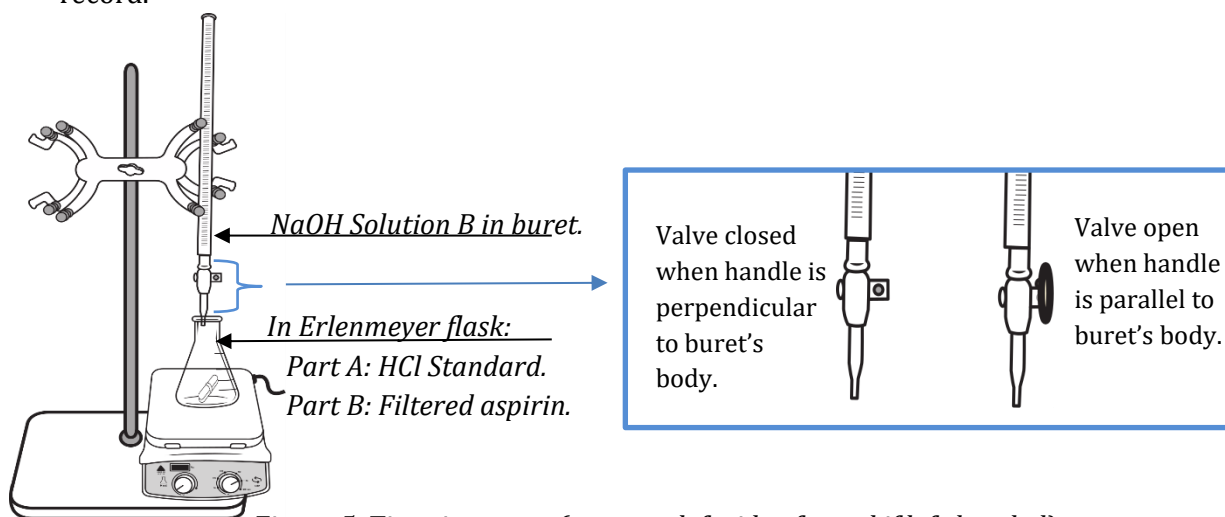


Figure 5. Titration setup (setup on left side of stand if left-handed).



- c) Start titrating: Add the NaOH into the HCl sample while continually mixing. As the endpoint nears, drip the NaOH solution into the HCl sample until the endpoint, which is when the solution turns pale pink and persists for at least 30 seconds. Stop titrating. Make the final NaOH volume reading. Read the buret to a precision of  $\pm 0.01$  mL and record.
- d) Refill the buret with the NaOH Solution B. Repeat Step A4 with HCl Trials 2 and 3.
- 5. Save NaOH Solution B. The NaOH Solution B will be used for the aspirin analysis in the next lab period.**
- a) Place the NaOH Solution B in a tray on the professor's bench or other designated area. You will use it next week.
- 6. Calculation of the Experimental Concentration of NaOH Solution B and % Relative Error.**
- a. Calculate the concentration of NaOH Solution B (Experimental value) in each trial.
- Calculate the moles of HCl Standard used in each Trial using Equation 2. Remember that the volume of the sample of HCl titrated was 15.00 mL.
  - Use the balanced equation, Equation 4, to calculate the moles of NaOH Solution B that reacted with the moles of HCl in each Trial.
  - Calculate the volume of NaOH delivered (final – initial buret readings).
  - Calculate the molar concentration of NaOH Solution B from each Trial (see Equation 2).
- b. Calculate the mean molar concentration of NaOH Solution B and its standard deviation using Equation 5 and Equation 6, respectively. Also, calculate the relative standard deviation using Equation 7.
- c. Finally, calculate the relative percent error between the mean experimental value and theoretical value for the concentration of NaOH Solution B using Equation 8.

Remember: Data Table 1, along with these calculations, are due at the beginning of the next laboratory period.

## Part B Determination of the amount of acetylsalicylic acid in aspirin by titration with NaOH

Record the data of Part B in Data Table 2.

### 1. Prepare the aspirin sample Trial 1.

- Select an aspirin brand. Record brand and amount of active ingredient.
- Remove one tablet from the selected brand, weigh it and record value.
- Pulverize the weighed tablet using a mortar and pestle.
- Tare a boat (or weighing paper) on the balance. Carefully transfer as much powdered sample into the boat as possible. Record the mass (record all digits from the analytical balance). You will need to determine the actual mass titrated in order to correct for the mass loss.
- Transfer the weighed powder into a clean Erlenmeyer flask (125-mL or 250-mL). Rinse the boat with DI water over the flask to ensure the sample is completely transferred into the flask. Label the flask as Aspirin Trial 1.
- Pour about 30 mL of ethyl alcohol (also called ethanol) into a 50-mL beaker. Label the beaker and cover with a watch glass.
- Using a graduated cylinder, add 10.0 mL of ethyl alcohol to the Erlenmeyer flask and stir for about 30 seconds with a glass rod. Then add 25.0 mL of water to the flask and stir again for another 30 seconds. The tablet will not completely dissolve due to inert ingredients (fillers and binding agents) in the tablet; the solution will be turbid. It will be necessary to filter it. (Remember to properly label your sample.)
- Place a funnel in a clean Erlenmeyer flask (125-mL or 250-mL). See Figure 6a. (Note: If the funnel touches the bottom of the flask, suspend it above the flask using a support ring clamp and, if needed, a clay triangle (Figure 6b) held on a stand.)



[Technique 2](#)



[Technique 16](#)  
[Video Tech. 16](#)

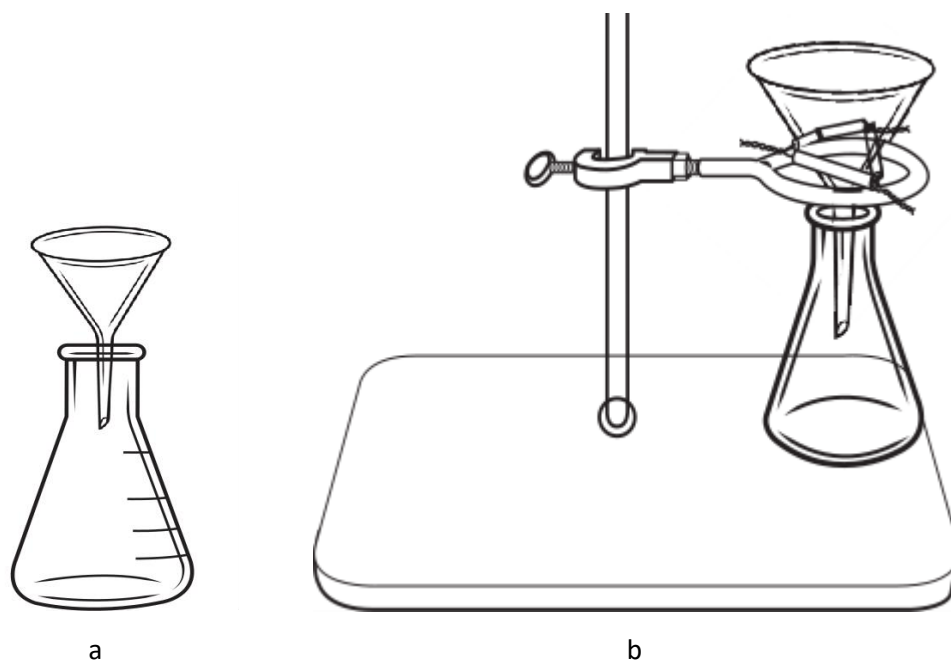


Figure 6. Funnel over Erlenmeyer flask

- i. Obtain a fluted filter paper (Fisherbrand Qualitative P8-Flute 09-790-14E) or fan fold a flat filter paper (Whatman #1 Qualitative). Open it (Figure 7).



[Technique 23](#)  
[video](#) starting  
at 1:04



Figure 7. Open fluted (or fan-folded) filter paper

- j. Place the filter paper inside the funnel and seal it by wetting it with a small amount DI water. (You can leave this water in the Erlenmeyer flask.) Filter the aspirin sample prepared above. Once all the solution has gone through, rinse the filter paper and precipitate with a few milliliters of deionized water. Allow all the water to drain into the receiving flask. Rinse two more times.
- k. Remove funnel and cover flask with Parafilm or a watch glass.
- 2. Prepare the aspirin sample Trials 2 and 3.**
- a. Wipe the mortar and pestle with a paper towel to remove residue left from first tablet.
- b. Prepare two more trials: obtain two additional tablets of the same aspirin brand and repeat steps B1b – B1k; wipe mortar and pestle after each use, use a new filter paper and new flask each time. Label as Aspirin Trial 2 and Aspirin Trial 3.
- 3. Titration set-up.**
- a. Retrieve the NaOH Solution B that you prepared last week (located on professor's bench or other designated area).
- b. Set up the titration following Part A steps 3a-d.
- 4. Titrate the aspirin samples.**
- a. Add 2-3 drops of phenolphthalein indicator and a magnetic stir bar to the filtered aspirin sample Trial 1. Lower the buret until its tip is barely below the rim of the Erlenmeyer flask.
- b. Make the initial NaOH volume reading. Read the buret to a precision of  $\pm 0.01$  mL and record.
- c. Start titrating: Add the NaOH into the aspirin sample while continually mixing. As the endpoint nears, drip the NaOH solution into the aspirin sample until the endpoint, which is when the solution turns pale pink and persists for at least 30 seconds (titrate slowly – it takes longer for the aspirin sample to react with the NaOH near the endpoint). Stop titrating. Make the final NaOH volume reading. Read and record the buret to a precision of  $\pm 0.01$  mL.
- d. Repeat Part B steps 4a-c with aspirin Trial 2.
- e. Repeat Part B steps 4a-c with aspirin Trial 3.
- 5. Calculation of active ingredient in the aspirin.**
- a. Calculate the volume of NaOH added and the moles of NaOH.
- b. Calculate the moles of acetylsalicylic acid that reacted with the NaOH in the titrated sample using Equation 1.



[Technique 3](#)  
[Technique 22](#)  
[Video Tech. 22](#)

- c. Calculate the grams of acetylsalicylic acid in the titrated sample using the molar mass acetylsalicylic acid.
- d. Determine milligrams of acetylsalicylic acid (asa) in the whole tablet by using Equation 9.

$$mg_{asa \text{ per tablet}} = mg_{asa \text{ in sample}} \times \left( \frac{\text{mass tablet}}{\text{mass pulverized sample}} \right) \quad \text{Equation 9}$$

- e. Calculate the mean  $mg_{asa \text{ per tablet}}$  and its standard deviation. Also, calculate the relative standard deviation.
- f. Calculate the relative percent error between the experimental mean and theoretical value (the value on label) for the  $mg_{acetylsalicylic \text{ acid per tablet}}$ .
- g. Finally, state if the brand analyzed contained an acceptable mass of acetylsalicylic acid per tablet. (Acceptable industry tolerance for this type of product ranges within 5% of the value stated on the label.)

### Clean up/Disposal

- Use a magnetic retriever (long plastic covered bar with a magnetic end) to remove magnetic stir bars. The retriever is usually hanging inside of the hood.
- Dispose all solutions into the waste container for liquids/aqueous solutions.
- Place pulverized solid tablet, and the filter paper with the precipitate in the waste container for solids.
- Rinse pipets and buret several times with small amounts of deionized water. Collect rinses into a waste beaker, transfer to waste container for liquids/aqueous solutions.
- Place pipets and buret, tip up, in the designated canisters.



[Technique 11](#)

### References:

1. Hanneman, J. *Adds in History: 1926 Bayer Aspirin*. Treasured Lives [Online] May 1, 2016. <https://treasuredlives.us/2016/05/01/ads-in-history-1926-bayer-aspirin/> (accessed July 3, 2018).
2. Beran, J. A. *Laboratory Manual for Principles of General Chemistry, 10th ed.*, John Wiley & Sons: Hoboken, NJ, 2014; p 237.
3. Rainsford, K. D. Pharmacology and Biochemistry of Salicylates and Related Drugs. In *Aspirin And Related Drugs*, Rainsford, K. D. Ed.; CRC Press, Sheffield, UK, 2004; p 266.
4. Rainsford, K. D. History and Development of the Saicylates. In *Aspirin And Related Drugs*, Rainsford, K. D. Ed.; CRC Press, Sheffield, UK, 2004; p 2.

## Pre-lab

1. What volume, in milliliters, of a stock solution of 2.55 M NaOH would you have to use to prepare 1.00 L of a 0.500 M NaOH?
2. The titration of a 25.00 mL sample of NaOH required 28.25 mL of a 0.200 M HCl solution to reach the end point. What was the molar concentration of the sodium hydroxide solution?
3. A group obtained the following results:

Trial #	1	2	3
Amount active ingredient/tablet (mg)	598	583	587

Report the average, standard deviation and relative standard deviation (RSD) using correct units and correct significant figures.

4. A tablet of Pain Be Gone Aspirin, which had a mass of 1.213 g, was pulverized and 1.159 g were dissolved in 10.0 mL of ethyl alcohol and 25.0 mL of DI water. The titration of this solution with 0.1052 M NaOH required 15.62 mL to reach the phenolphthalein endpoint.

Answer questions a – d below.

- a. Determine the moles of NaOH that reacted with the acetylsalicylic acid.
- b. Determine the mass, in grams, of acetylsalicylic acid in the sample analyzed.
- c. Determine the mass, in milligrams, of  $HC_9H_7O_4$  in the tablet.
- d. The manufacturer claims that each tablet contains  $325 \text{ mg} \pm 10 \text{ mg}$  of acetylsalicylic acid. Is the actual amount of acetylsalicylic acid in the tablet acceptable?

## Post-lab

In the data/calculations section:

1. Include the completed data tables.
2. In the calculations section show calculations requested.

## Experiment 6: Amount of Active Ingredient in Aspirin Experimental Data and Calculations

Name: \_\_\_\_\_ Date: \_\_\_\_\_

Lab Partner: \_\_\_\_\_ Section: \_\_\_\_\_

Table 1. Determination of NaOH Concentration. (Part A due next Lab.)

1) HCl concentration written on carboy			
2) NaOH concentration written on carboy			
3) NaOH Solution A concentration (Theoretical value)			
4) NaOH Solution B concentration (Theoretical value)			
	Trial 1*	Trial 2	Trial 3
5) Volume of HCl sample			
6) Moles HCl			
7) Moles of NaOH			
8) Initial buret reading			
9) Final buret reading			
10) Volume NaOH added			
11) Molar concentration of NaOH Solution B (Experimental Value)			
12) Mean molar concentration of NaOH Solution B			
13) Standard deviation			
14) RSD			
15) Relative percent error			

\* Show calculations for Trial 1, standard deviation, RSD and relative % error. (Continue on back of sheet if needed.)

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