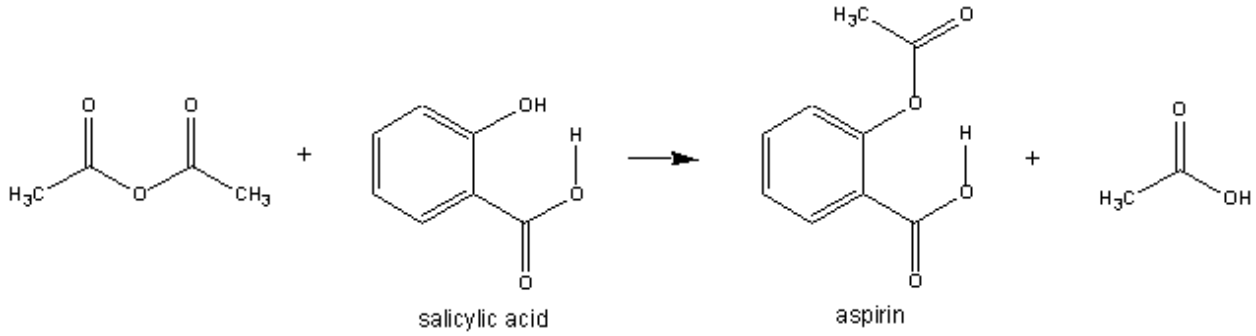
Experiment 9
Titration of Aspirin

Purpose
First, you will determine the precise concentration of a sodium hydroxide stock solution (by a process called "standardization"). The NaOH concentration will change with time due to reaction with atmospheric carbon dioxide as it sits on the shelf in the stockroom. The standardization procedure requires a titration, which is a technique that allows you to analyze your sample based on quantitative relationships that exist between the analyte (the NaOH) and a titrant (an acid) that reacts with the analyte with a known stoichiometry.

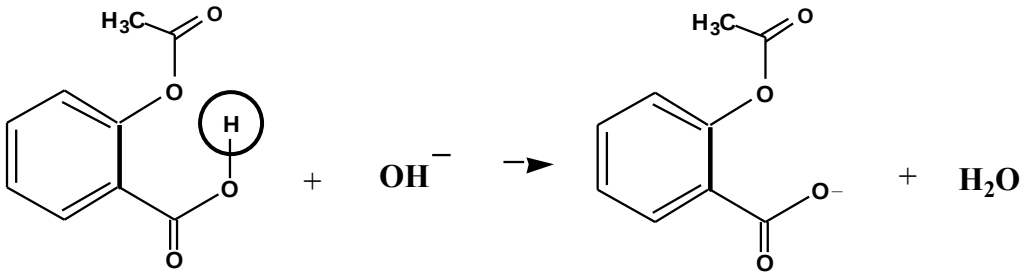
Second, you will titrate a sample of your aspirin (an acid) with the standardized NaOH to determine the moles of acid in a given weight of your product. This will allow you to assess its purity. Recall the equation for the synthesis of aspirin in the previous experiment:



Synthesis of Aspirin (Experiment 6)

Assuming the aspirin is not contaminated with other acids, the titration allows you to quantitatively determine the purity of your aspirin. Under the conditions of the experiment, the base will react with only with the acid proton on the aspirin and will not break off the acetyl group. (The reacting hydrogen is circled in the equation below.)

The **Net Ionic** Equation for the titration in this experiment:



aspirin (analyte) | hydroxide (titrant) *Standardized in Part 1 of this experiment*

Experiment 9 Turn-in Sheets

Titration of Aspirin

Experimental Procedure

Use this space to record your observations and/or data

1) Standardization of the NaOH solution.

Prepare a buret for titration by rinsing it with two small portions of distilled water, followed by two 10-ml portions of the sodium hydroxide solution. Fill the buret and follow the usual procedures for eliminating air bubbles and setting the initial level. Your buret is now ready for the rest of the lab (**NOTE: You should not need more than 50 ml of NaOH for all the titrations**)

Record the initial buret reading.

Place an accurately weighed 0.10 g sample of salicylic acid (SA) in a clean 125 ml Erlenmeyer flask. Dissolve the acid in 15 ml of 95% ethanol and add 2 drops of phenolphthalein indicator. ***Do you have to measure the amount of ethanol precisely? Does your glassware need to be dry?***

If the approximate concentration of the NaOH is known, estimate the approximate volume of this solution that will be required to react completely with the salicylic acid.

Run slightly less than this amount in to the flask containing the acid, while swirling the flask. Use your wash bottle to clean the walls of the flask of drops of base that may have splattered out the titration mixture. Continue adding the NaOH solution. As you approach the volume estimated to be needed for complete reaction, add the NaOH more slowly, while continuing to swirl the flask and wash down the walls.

Stop the titration when the addition of a single drop of base changes the color of the solution to a light pink, indicating that you have reached the endpoint. The endpoint should persist for 30 seconds without fading.

Record the final buret reading.
Repeat the titration twice more.

2) Titration of the Aspirin Sample

Use this space to record your observations and/or data

Follow procedure (a) or (b) depending on your source of aspirin.

a) If you have a sample of aspirin you synthesized in Experiment 9:

Accurately weigh out 0.10-0.15 g of your aspirin sample into a 125 ml flask. Add 15 ml of 95% ethanol and a dropper-full of phenolphthalein indicator. Slowly titrate the aspirin with the standardized NaOH solution using the same procedure as above. Record the initial and final buret readings.

Repeat the titration twice more.

Waste Disposal: All solutions should be emptied into appropriate containers in the fume hood.

b) If you are using commercially available aspirin tablets:

Place two aspirin tablets into a 250-mL flask with 50 ml of a 1:1 mixture of 95% ethanol: water. Record the brand name. Allow the tablets to set for a few minutes and then tap the tablets using your stirring rods; they should disintegrate (the solution will remain cloudy due to the insoluble starch binder). Swirl the mixture to dissolve the aspirin. Aspirin itself does not dissolve well in water, hence the ethanol is used. However, the water is required for the titration (think about why?).

Add a dropper full of phenolphthalein indicator. Slowly titrate the aspirin with the standardized NaOH solution. Record the initial and final buret readings to the correct number of significant figures.

Repeat the titration once more. Do a third trial if necessary.

Waste Disposal: All solutions should be emptied into appropriate containers in the fume hood.

Data

Construct a data table that organizes all of your data for each trial and for each titration (Part 1 and 2). Make sure to include the precise volume of analyte (in the flask) used for each trial, the starting and ending buret readings, the volume of titrant added (from buret to flask), and an average value of this volume from your trials.

Don't forget to use units and significant figures. Record all data in permanent ink.

Calculations

Show your work for the first trial. Use the proper number of significant figures.

Standardization of the NaOH solution.

1) Calculate the moles of salicylic acid used in each trial.

2) Calculate the moles of NaOH required to neutralize the salicylic acid in each trial.

3) Calculate the molarity of the NaOH in each trial

Average NaOH molarity = _____ M

Calculations, continued

Titration of the Aspirin Sample

1) Assume that your aspirin sample is pure (not a good assumption) and calculate the moles of aspirin to be titrated in each trial. These moles are the **theoretical** moles of aspirin (in theory, your sample should be pure).

2) Calculate the moles of NaOH used in each trial.

3) Calculate the moles of aspirin that reacted with the NaOH in each trial These are the **actual** moles of aspirin in your sample (the moles of acid that actually reacted with the NaOH).

4) Calculate the percent purity of aspirin in each trial.

$$\%\ \text{purity} = \frac{\text{actual moles of aspirin}}{\text{theoretical moles of aspirin}} \times 100$$

Average % purity of aspirin based on three trials = _____%

If your percent purity is not 100%, explain what may have occurred to cause this deviation.

If your percent purity is 100%, explain why this shouldn't be the case.[1]

[1] There are several good explanations for these questions. Don't assume your masses or calculations are incorrect. Instead, look to flaws inherent in this experiment or incorrect assumptions.

Pre-Lab Assignment

Refer to Chapters 6 and 7 in your textbook.

1) Go to the Data section of this experiment and create a data table given the guidelines at the top of the page. Use permanent ink and leave space for data you will collect during lab. **This table may be checked for completion at the beginning of lab!**

2) What volume of 0.812 M HCl is required to titrate 1.33 g of NaOH to the endpoint?

3) A student measured out 0.132 g of her aspirin sample and added 15 ml of 95% ethanol and 2 drops of phenylphthalein indicator.

   a. What is the theoretical number of moles in her sample?

   b. The student found that it took 5.66 ml of 0.1002 M NaOH to reach the endpoint in her first trial and 5.98 ml of the NaOH in her second trial. What is the actual number of moles of aspirin in each trial?

   c. What is the percent purity of aspirin in each trial?

   d. Give a possible explanation why the student's percent purity is not 100%.

4) A student found that his titration had taken 10.00 ml of 0.1002 M NaOH to titrate 0.132 g of aspirin. Calculate his percent purity. Give a possible explanation of what might have affected his percent purity.