**Volumetric Analysis:**
**Analysis of antacid tablets**
**Analysis of Cl\(^{-}\) concentrations in IV solutions**

**OBJECTIVE:** The goals of this experiment are to learn titration concepts and techniques.

**SKILLS:** Titration, manipulation of stoichiometric relationships involving concentration

**EQUIPMENT:** Burets, Pipets, Erlenmeyer flasks

**INTRODUCTION:** It is frequently the case that the concentration of a compound or ion in a solution is unknown and in need of determination. In the iron lab, you became familiar with one method to quantitatively determine the concentration of substance – spectrophotometry. Spectrophotometry is an excellent choice for quantitative analysis if the species of interest either absorbs light in the visible spectrum or can be converted into a complex that does so fairly readily. For substances for which spectrophotometry is impractical, other quantitative methods must be employed.

One common method is titration, which involves reacting the sample to be tested with a reagent solution of known concentration. When the number of moles of the chemical of interest is equivalent to the number of moles of the added reagent, a change, usually in color, occurs in the system. This is the titration endpoint. Knowing the reaction that is occurring between our sample and the added reagent makes it possible to calculate the concentration of our sample using their stoichiometric relationship.

In this experiment, we will use the technique of titration to determine concentrations for two different systems. In the first part of the experiment, an acid-base titration will permit the determination of the concentration of base in an antacid tablet. In the second part of the experiment, a precipitation reaction will be used to determine the concentration of chloride ions in IV solutions before and after dialysis.

**Part I: Determination of the amount of base in antacid tablets**

Digestion of food occurs in the mouth, stomach and small intestines. No matter where digestion occurs, it is facilitated by catalysts that speed up the rate at which large molecules are broken down into smaller compounds. In the stomach, the rate of this reaction is further enhanced by the strongly acidic environment. The mixture of food and gastric juices, or chyme, is normally prevented from backing up into the esophagus by the muscles of the esophagus. If this muscle fails to fully completely close off the esophagus from the stomach the acidic chyme will irritate the esophageal walls, causing the burning sensation associated with heartburn and acid indigestion. For individuals susceptible to heartburn, one solution is to decrease the acidity of the stomach through the use of antacids, bases that will react quickly to neutralize some of the HCl in the stomach, bringing the pH up above pH 3. Different tablets may have different strengths (and use different bases, each of which comes with its own advantages and disadvantages).
In order to determine the amount of base available in an antacid tablet, two steps are needed. The first step involves preparing and standardizing (accurately determining the concentration of) a basic solution of NaOH using a known quantity of a chemically pure and dry standard acid. Once the concentration of this standardizes NaOH solution is known exactly, it can be used to determine the amount of base in a commercial antacid tablet through the use of a back titration. In a back titration, an excess amount of standardized acid is allowed to react with the antacid tablet. When the reaction is complete, the amount of acid unreacted is determined via a titration with the standardized NaOH. The difference between the known amount of acid that was originally placed in the flask and the amount of acid that reacts with the NaOH is the amount of acid that reacted with the antacid.

**Part II: Analysis of Cl− concentrations in IV solutions before and after dialysis**

In dialysis, solvent and small molecules pass through the dialyzing membrane in order to equalize the concentration of the solution on either side of the membrane. This was observed qualitatively in a previous lab. The same reaction can be done in a quantitative manner to determine the change in chloride concentration in the solution as a result of dialysis. The concentration of chloride ions before and after dialysis can be determined by taking advantage of the following reaction:

\[
\text{AgNO}_3(aq) + \text{Cl}^-(aq) \rightarrow \text{AgCl}(s) + \text{NO}_3^-(aq)
\]

AgCl is an insoluble salt. Silver ions added during titration will react with any chloride present in solution until all of the chloride ions have reacted. If the volume and concentration of the added silver is known, the concentration of chloride present in the original solution can be determined using stoichiometric calculations.

In order to tell when enough AgNO₃ has been added to fully react with all of the chloride, we need an indicator. Indicators are organic compounds that change color when the end point of the titration has been reached. Indicators are chosen based on the titration being performed. Dichlorofluoroscein can be used for the AgCl titration. In the presence of excess chloride ions, the indicator is yellow. In the presence of excess silver, the indicator turns the surface of the AgCl(s) pink. Therefore, we can tell that all of the chloride has been precipitated when we observe the color change from yellow to pink. Because the color change takes place on the surface of the precipitate, it is sharper if the surface area is large. Certain compounds, such as dextrin, keep precipitates of silver halides from coagulating and provide the needed surface area but are not involved in the calculations.

**PROCEDURE:**

Prepare about 500 mL of approximately 0.1 M NaOH solution by calculating the volume of 1 M NaOH needed for dilution with water to a total volume of 500 mL. Take that
amount of 1 M NaOH, place it in a 600 mL beaker, and add water until the total volume is about 500 mL. This solution will be standardized and then used for titration of an antacid tablet. If the solution is not properly prepared, or it is all used up before all of the necessary titration are performed, then a new solution must be made and standardized.

Concentrated NaOH solutions are hazardous. The dilute solutions used here are less so but can cause damage to skin, clothing and other valuables placed on the lab bench. Work neatly and wipe up any spills promptly.

I. Standardization of NaOH

Prepare a buret by rinsing it with two 10 mL portions of the NaOH solution. Then fill the buret with the NaOH solution. Be sure there are no air bubbles in the buret and that the tip of the buret is filled.

Into each of your clean 250 mL Erlenmeyer flasks, weigh out about 0.3 g of HKC₈H₄O₄ (potassium hydrogen phthalate, commonly abbreviated KHP, with MW = 204.2 g/mol), recording the exact mass of KHP used in each flask. The transfer of the solid KHP to your flask should be done quantitatively by rinsing the weigh boat with distilled water into your flask several times. Add 75 mL of distilled water to the flask and swirl it to dissolve the acid. After all of the KHP has dissolved, add 3 drops of phenolphthalein.

Rough titration

It is useful to perform a rough titration to get an idea of about how much of the titrant (the NaOH solution) will be needed to reach the end point. In subsequent trials, you can add titrant from the buret quickly at first and then slow down as you near the end point. Use your sample with the smallest mass of KHP for your rough titration. Record the starting reading of the buret. Then add NaOH from your buret in about 1 mL increments. After each addition, swirl your flask and observe the color of the solution. Continue addition NaOH in 1 mL increments until a pink color remains for 30 seconds. Note: you will most likely get a very dark pink/magenta color. This is acceptable for the rough titration ONLY since the dark color is an indication that you have surpassed the end point.

Record the final reading of the buret and determine the volume of titrant added. This will give you a rough idea of the amount of base needed in your subsequent standardization trials. For example, if your rough titration required 10 mL of base, then you can quickly add about 8-9 mL of NaOH from the buret quickly and then slowly add NaOH drop by drop to reach the end point.

The reaction taking place is:

\[
HKC₈H₄O₄ + NaOH \rightarrow H₂O + Na⁺(aq) + K⁺(aq) + C₈H₄O₄²⁻(aq)
\]
Quantitative trials

Perform a minimum of three quantitative trials to standardize your NaOH solution. In these trials, it is essential that you reach a quality end point, which is evidenced by a light pink color that remains for 30 seconds. A bright pink color similar to your rough titration indicates that you have surpassed the end point and that trial is not valid.

Begin your qualitative trials by adding a volume of NaOH as determined by your rough titration. As described above, use 1-2 mL less NaOH than was used in the rough titration. This will help decrease the chances of surpassing the endpoint during this addition. Continue to titrate the KHP by allowing the NaOH solution to drip slowly from the buret into the flask containing KHP. Be sure to swirl the solution carefully while titrating. As you get closer to the end point, the pink color will remain in the flask for longer periods of time as you swirl. At this point, you should add the NaOH drop by drop, taking care to have small drop sizes. Titrate until the pink color of the phenolphthalein indicator just appears and remains for 30 seconds.

Record the volume of NaOH used. Volumes in burets can read to the nearest 0.01 mL. Be sure to carefully read the buret. Repeat the procedure two more times. This information can be used to determine the concentration of the NaOH solution, since the number of moles of NaOH is equal to the number of moles of KHP at the endpoint of the titration, and since both the number of moles of KHP and the volume of NaOH used are known. Calculate the concentration of the NaOH solution in each of the three quality trials and average the results. Show your results to your instructor to see if you can proceed to the next analysis. Enter the concentration of your NaOH solution into the lab computer.

Ib. Antacid analysis

Accurately transfer 0.35 g of a pulverized commercial antacid tablet to a 250 mL Erlenmeyer flask, recording the exact amount used. Add about 20 mL of distilled water to the flask and then pipet 5.00 mL of standardized HCl to the flask swirl the mixture to dissolve the antacid. Be sure to record the concentration of the standardized HCl. Some solid may remain and is usually unreacted tablet fillers. You will be performing several trials of the antacid analysis so you will need several samples of the same antacid.

Slowly heat the solution to boiling and continue to heat at a gentle boil for at least one minute to expel the dissolved CO₂. Add 4-8 drops of phenolphthalein indicator to the flask. If the solution is pink, add an additional 5.00 mL of standardized HCl to the flask and boil again for one minute.

Titrate the excess HCl to the end point using the standardized NaOH you prepared above. It would be useful to perform a rough titration as you did previously. Record the initial and final volumes of NaOH in the buret and determine the total volume of NaOH used. Perform a total of three quantitative trials using the same antacid. Enter your data into the lab computer.
Analyze the class data in order to determine the cost per gram of the antacid tablet. In order to compare different commercial antacids, a useful comparison is the cost per equivalent mole of base in the antacid. This comparison will enable you to determine how much it costs when different antacids neutralize the equivalent amount of HCl as one mole of base would neutralize.

Class data for this part of the experiment can be downloaded from Blackboard. Tests were done on two different brands of antacids. In the class data part of the report, use Excel to find the equivalent moles of base per gram of tablet for each measurement made in the class, and then determine the average values for both types of antacid samples. Step by step instructions are provided below on how to use Excel effectively for these calculations.

1. Find the initial number of moles of HCl added by multiplying the molarity of the HCl solution (cell H3) times the volume of HCl added initially (column C) in liters. For example, if you are working on the data in Row 11 (the first row with data) you can end the following formula in cell D11: =SH$3 * (C11/1000). The dollar signs ($) create a fixed cell reference (the molarity of the HCl is the same for each sample). You can then copy and paste this formula into the remaining cells in column H.

2. Next find the number of moles of base you added in the titration with the leftover HCl. To do this, multiply the molarity of the NaOH (column F) times the volume of NaOH titrated in liters. The formula for row 11 in column G would read: =F11 * (E11/1000). The NaOH you added from the buret during the titration reacted with the leftover HCl that did not react with the antacid. For each mole of NaOH added, one mole of leftover HCl is neutralized. Thus, the number of moles of NaOH added during the titration equals the number of moles of HCl leftover.

3. Now it is possible to find out how many moles of HCl reacted with the antacid: moles HCl reacted with antacid (H) = initial moles HCl added (D) – leftover moles of HCl (G) where the bold letter indicates the corresponding column in the Excel worksheet. For the data in row 11, the formula in cell H11 would read: = D11 – G11.

4. For every mole of HCl reacted, one mole of antacid (base) also reacted. Therefore, the moles of base provided by the antacid (column I) equals the moles of HCl reacted (column H).

5. We’re now interested in how much base is in the antacid per gram of tablet. This will enable us to compare the two brands. The mass of each antacid sample is recorded in column J. Therefore, to find the moles of base in the antacid per gram of tablet, divide the value in column I by the value in column J. This can be done in column K. For example, in K11 you might write: = I11 / J11.

6. Finally, we want to find out which brand is a better buy. To do this we must make use of the data for the cost of the antacid per gram. If we take the cost per gram of tablet and divide by the moles of base in the antacid per gram of tablet, we get the cost per mole of base in the antacid (a value we can use to compare the two brands):

\[
\frac{\text{cost (dollars $)}}{\text{gram antacid}} \times \frac{\text{gram antacid}}{\text{moles base}} = \frac{\text{cost (dollars $)}}{\text{moles base}}
\]
The cost per gram can be found in cells H4 for Rolaids and K4 for Tums. So, for example if row 11 was a Rolaids sample, then in cell L11 you would write: =H$4 / K11. Report the cost per gram of antacid ± standard deviation.

Look at the antacid data; are any of the values obviously wrong? If so, make a note of which ones and set those values to a blank. Once this is finished, the results can be averaged in the same way that we have done previously.

In doing complicated calculations like these, it is often useful to do one calculation by hand so that you can compare it to the Excel result for a particular case. You will have already done this for your individual data, so you can check to see if you told Excel how to do it correctly.

Relative error analysis: Compare the results for your antacid samples to the class values by calculating their relative errors. The relative error is defined as the error (the difference between your value and the average value) divided by the average value. It is often given as a % error. A positive (negative) relative error means that your value was higher (lower) than the class average.

\[
\text{Relative Error} = \frac{\text{Your Value} - \text{Average Value}}{\text{Average Value}} \times 100
\]

II. Determination of Cl\textsuperscript- concentration with AgNO\textsubscript{3}

AgNO\textsubscript{3} is expensive. Minimize waste. It is not particularly toxic but will stain skin black if spilled. Dispose of all silver containing solutions in the labeled waste jar.

Using a pipet, measure 5.00 mL of IV solution into a 250 mL Erlenmeyer flask. Add 40 mL of distilled water, 0.1 g dextrin and 3-4 drops of dichlorofluoroscein. Using the techniques of good titration developed above, titrate the solution using 0.050 M AgNO\textsubscript{3}. The end point of titration can be seen when the dextrin turns pale pink. Record the volume of AgNO\textsubscript{3} used and calculate the number of moles of AgNO\textsubscript{3} used and the Cl\textsuperscript- concentration. Repeat the titration with a second portion of the IV solution to determine an average concentration.

Titrates the dialyzed IV solutions using the same method described above. Report the concentration of Cl\textsuperscript- using an average from two titrations. Enter your data into the lab computer.
The ion concentrations in physiological solutions are often recorded in milliequivalents, or mEq, per liter. One equivalent is the amount of positive or negative ion that supplies one mole of electrical charge. For a singly charged ion, the concentration in Eq/L and mol/L will therefore be the same.

Calculate the percent error for the pre-dialyzed IV solution, using the chloride concentration listed on the IV bag for the theoretical value. Note that, while the calculation is the same as that used for determining the percent error in the antacid tablets, the ‘true’ value used in the manufacturer’s listed value rather than the class average. This is a reflection of the fact that the amount of base in the antacid tablets is likely due to several different bases and thus comparison of your experimental value to the ingredients of the tablet is slightly more complicated than is the comparison between your chloride concentration to that for an IV solution. Compare your data to the class average by reporting the relative error. Report class values as average ± standard deviation.