Determination of Calcium Carbonate in Eggshells – Background

The major component of eggshells is calcium carbonate, $CaCO_3(s)$. This analysis is done volumetrically by using a characteristic reaction of carbonate compounds, namely their reaction with acids. Calcium carbonate (limestone) is very insoluble in pure water but readily reacts in acid according to the reaction below.

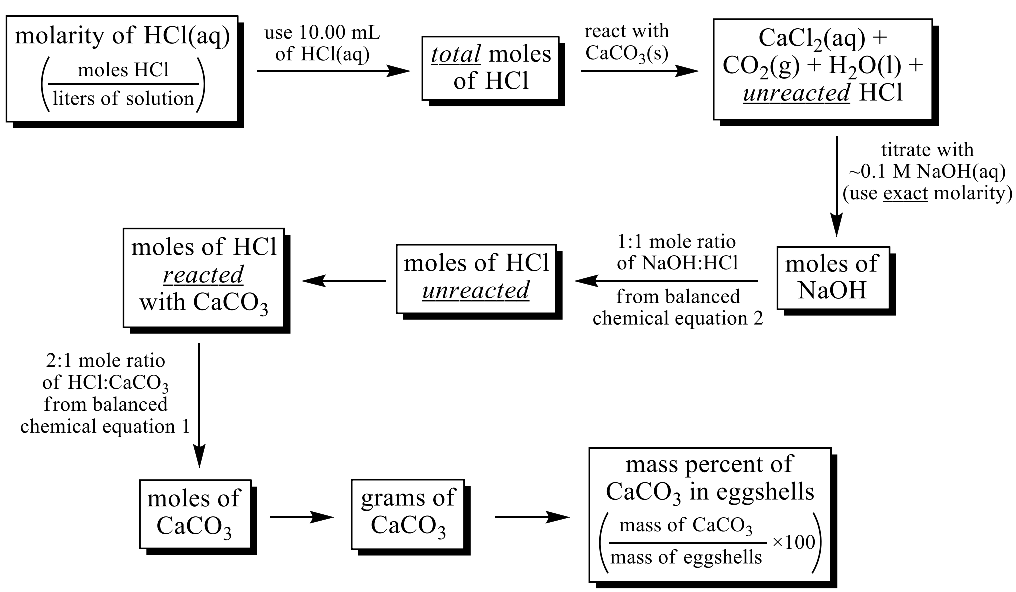
$$\underset{\text{(in excess)}}{2HCl(aq)} + \underset{\text{(limiting reagent)}}{CaCO_3(s)} \rightarrow CaCl_2(aq) + CO_2(g) + H_2O(l) + \underset{\text{(unreacted)}}{HCl(aq)} \quad (1)$$

This reaction cannot be used directly to titrate the $CaCO_3$ because it is very slow when the reaction is close to the endpoint. Instead the determination is achieved by adding an excess of hydrochloric acid to react with all of the $CaCO_3$ and then titrating the remaining unreacted HCl with NaOH solution to determine the amount of acid which did not react with the calcium carbonate. The difference between the moles of the acid (HCl) initially added and the moles of HCl left unreacted after the reaction, is equal to the moles of HCl that did react with $CaCO_3$. The reaction used to determine the amount of unreacted acid by titration is given below. This type of analysis is generally referred to as a back-titration.

$$\underset{\text{(unreacted)}}{HCl(aq)} + NaOH(aq) \rightarrow NaCl(aq) + H_2O(l) \quad (2)$$

Calculations:

The purpose and goal of today's experiment is to determine the mass percent of $CaCO_3$ in an eggshell. The following logic stream may be helpful to understand how you will reach that goal. The calculation steps are reproduced at the end of the written procedure in a more formal step-by-step manner. As with all calculations, make sure to show all units and pay close attention to significant figures.



Pre-Lab Homework

1. How many milliliters of 0.383 M HCl are needed to react with 16.2 g of $CaCO_3$?

$$2HCl(aq) + CaCO_3(s) \rightarrow CaCl_2(aq) + CO_2(g) + H_2O(l)$$

2. If a 45.0 g sample of $CaCO_3$ (s) is added to 1.25 L HCl (aq) that has a density of 1.13 g/mL and contains 25.7% HCl, by mass, what will be the molarity of HCl in the solution after the reaction is completed? (Assume that the solution volume remains constant.)

3. A piece of marble (assume it to be pure $CaCO_3$) reacts with 2.00 L of 2.52 M HCl. After dissolution of the marble, a 10.00 mL sample of the remaining HCl(aq) is withdrawn, added to some water, and titrated with 24.87 mL of 0.9987 M NaOH.
(a) Write net equation for the two reactions involved.
(b) What must have been the mass of the piece of marble?

<u>Determination of Calcium Carbonate in Eggshells – Procedure</u>

Purpose: To measure the mass percent of calcium carbonate in a chicken's eggshell.

Special Equipment: Things to use and return on the same day.

- You will be using a buret, buret brush, and a 10.00 mL volumetric pipet.
- Be sure to rinse all the glassware with deionized water and return it when you are done.

Procedure:

<u>Part 1. *Preparation of the Eggshell*</u>

This part of the experiment can be performed together by two teams of lab partners (four students total). For each group of four students (two pairs of lab partners), obtain one egg and any necessary glassware. Break the egg into a large beaker. Add water to the egg and stir before pouring down the drain to dispose of it. Wash the shell with deionized water and peel off <u>*all of the membranes*</u> from the inside of the shell. There are two membranes, one that is easy to see and one that you can find by rubbing your finger on the inside of the shell.

Dry the shell with a paper towel and put into a beaker labeled with your names. Place the beaker with the shell in the oven, and dry the shell for about 20 minutes. It is important that the shell be dry in order to get the best results. Be patient and keep the oven closed as much as possible! Remove the shell from the oven and grind it to a very fine powder using a mortar and pestle.

<u>Part 2. *Dissolution of the Eggshell in Excess HCl(aq)*</u>

The remainder of this experiment (Parts 2 and 3) should be done in teams of two, as usual. Accurately weigh between 0.450 g and 0.550 g of dried shell into each of three 125 mL or 250 mL Erlenmeyer flasks, labeled as "trial 1", "trial 2" and "trial 3". Be certain you record the mass of shell for each flask in your notebook. Add several drops of ethanol to each flask. This acts as a wetting agent and helps the hydrochloric acid dissolve the $CaCO_3$.

Pour ~35 mL of the 0.1 M HCl(aq) stock solution into a clean, dry, labeled beaker from your drawer. You will pipet the HCl(aq) from this beaker and <u>NOT</u> from the stock bottle. A total of 30 mL will be used for the titrations and you need a little extra to rinse your pipet, so 35 mL should suffice. Do not waste the HCl solution and <u>DO NOT</u> pour any excess back into the stock bottle. Excess HCl(aq) should be discarded in the proper waste container.

Slowly and carefully pipet 10.00 mL of 1.0 M HCl solution into each of your labeled Erlenmeyer flasks. Swirl the flasks to wet all of the solids. Heat the solutions in the flasks until they begin to boil and the solid egg shell dissolves. It is important that all of the eggshell solids dissolve because this contains the material you want to analyze. Eggshell is dense and will settle on the bottom of the flask. A white proteinaceous substance may form, but it will be suspended in the solution. Maintain a consistent fluid level in the flask by periodically washing down the walls of the flask with deionized water from your squirt bottle. DO NOT allow the liquid to completely evaporate or your eggshell will burn to a brown crust! The process of dissolving the eggshell is complete when no more "sinkers" (the eggshell) are visible in the flask; you'll be left with only "floaters" (the white proteinaceous material). Allow the flasks to cool. Rinse the walls of the flasks one last time with water from your wash bottle.

Part 3. *Titration of the Unreacted HCl(aq) With Your ~0.1 M NaOH(aq)*

Add 3-4 drops of phenolphthalein indicator to each flask. Using a funnel, partly fill a clean buret with your standardized ~0.1 M NaOH solution (saved from the last experiment) to rinse it. Drain the buret into a waste beaker. Fill the buret with your ~0.1 M NaOH solution. Run some solution out to remove all air bubbles from the tip. Replenish the solution in the buret if necessary. Read and record the initial volume to ± 0.01 mL. Titrate one sample to the first persistent barely-pink color. When you are close to the endpoint the color will fade slowly. Add more ~0.1 M NaOH(aq) dropwise until the color remains for at least 30 seconds. Read and record the final volume to ± 0.01 mL. Repeat the titration for the other two samples.

Waste Disposal and Cleanup:

- Dispose of all titrated solutions into the waste container. Wash the egg residue out of the Erlenmeyer flasks using hot soapy water and a test tube brush.
- Dispose of any excess ~0.1 M NaOH from the buret, and from your 1 liter polyethylene bottle, into the waste container. Rinse the 1 liter polyethylene bottle ***three times*** with deionized water and flush the rinses down the sink drain. Return the 1 liter polyethylene bottle to the stockroom cart.
- Rinse the buret ***three times*** with deionized water, and return the buret.
- Rinse the 10.00 mL pipet ***three times*** with deionized water and return the pipet.

Calculations: Use the following steps to calculate the percent mass of $CaCO_3$ in an eggshell. Use the correct number of significant figures and include ***all*** units.

A. Calculate the moles of HCl added to the eggshells.
B. Calculate the volume (L) of NaOH dispensed.
C. Calculate the moles of NaOH dispensed = moles HCl ***unreacted*** with eggshells.
D. Calculate the moles of HCl ***reacted*** with eggshells.

$$\text{total moles HCl} = \text{moles HCl reacted} + \text{moles HCl unreacted}$$

E. From the balanced chemical equation, convert moles HCl to moles $CaCO_3$.
F. Convert moles of $CaCO_3$ to mass (g) of $CaCO_3$.
G. Calculate the mass percent of $CaCO_3$ in an eggshell.

$$\text{mass \%} = \frac{\text{mass of } CaCO_3\text{, g}}{\text{mass of eggshell, g}} \times 100$$

Conclusion:

Report the average percent $CaCO_3$ by mass in the eggshell and the standard deviation. Comment on the accuracy of your average mass percent of $CaCO_3$ in an eggshell by comparing it to reported values. (Be sure to cite your references.) Comment on your precision as shown by your standard deviation. What are possible sources of error that may have affected, or could affect your accuracy and precision? In your opinion, do you trust your calculations and results, and was the lab successful?