Experiment 9

ANALYSIS OF CALCIUM CARBONATE TABLETS

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PURPOSE

To perform a gravimetric exercise to determine weight percent of active ingredient in a commercial calcium carbonate tablet.

CALCIUM

Calcium is an essential nutrient for the body. It is involved in the normal function of nerves and muscles (including the heart). Calcium ions in the blood are also necessary for blood clotting. In order to prevent blood from clotting, it is necessary that the calcium ions be precipitated out by adding some other substance. Blood samples used in the laboratory often have potassium oxalate added to them (causing calcium oxalate to precipitate out). Since oxalates are poisonous, during transfusions sodium citrate is added which causes solid calcium citrate to form.

A deficiency in the amount of calcium ion in the blood stream will cause calcium from bones to dissolve to replace it. Calcium is normally obtained through consuming milk and milk products. However, this can be supplemented by taking calcium tablets. Commercial tablets primarily consist of calcium carbonate (chalk). The solid calcium carbonate tablets are dissolved by stomach acid (hydrochloric acid, HCl) freeing the calcium ion to go into solution:

$$CaCO_3(s) + 2\,HCl(aq) \rightarrow CaCl_2(aq) + CO_2(g) + H_2O(l) \quad (1)$$

Reaction (1) also shows how calcium carbonate can act as an antacid. Here it is the carbonate part that is important, not the calcium. $CaCO_3$ is the active ingredient in many commercial antacids (such as Tums®). Other antacids use ingredients such as magnesium hydroxide (Milk of Magnesia®) which also acts as a laxative. Thus, often the magnesium hydroxide is mixed with aluminum hydroxide (which constipates, neutralizing the laxative effect of the magnesium hydroxide). Maalox® is an example of such an antacid.

MASS PERCENT

The mass percent calcium carbonate in the original tablet can be determined using the following formula:

$$\%\,CaCO_3 = \frac{mass\ CaCO_3}{mass\ tablet} x\ 100\% \quad (2)$$

The mass percent calcium in calcium carbonate can be determined using the following formula (as described in your textbook):

$$\%\,Ca = \frac{mass\ of\ Ca}{molar\ mass\ of\ CaCO_3} x\ 100\% \quad (3)$$

IN THIS EXPERIMENT

A calcium carbonate tablet will be dissolved, by HCl, to form calcium ions. Impurities will be filtered out and the calcium ions will be precipitated by adding sodium carbonate to reform calcium carbonate:

$$CaCl_2(aq) + Na_2CO_3(aq) \rightarrow CaCO_3(s) + 2\,NaCl(aq) \quad (4)$$

The mass percent calcium carbonate in the tablet will then be determined. Finally, the amount of calcium in the tablet will be calculated. This can be compared with the amount claimed by the manufacturer.

PRE-LABORATORY PREPARATION

1. Read the procedure and data analysis sections of this experiment.

2. Complete the computer-generated PRELAB assignment. Refer to the procedure and data analysis sections of the experiment as needed. The prelab questions for this experiment exactly replicate the questions in the data analysis section.

EXPERIMENTAL SECTION

REAGENTS PROVIDED

Hydrochloric acid, 1 M.
Sodium carbonate, 1.0 M.
Calcium carbonate tablets.

Hazardous Chemicals

Handle the hydrochloric acid solution with care. If any gets on you, wash thoroughly with water.

WASTE DISPOSAL

All liquid wastes can go down the drain. Your final solid product should be put in the appropriate waste container. Used filter paper can go in the garbage cans.

SPECIAL EQUIPMENT

Buret stands and suction filtration apparatuses should be available in the lab.

PROCEDURE

You will do this experiment individually.

1. Obtain a calcium carbonate tablet. Tare a balance, place the tablet on the balance pan, and record the mass on your data sheet (to the nearest 0.001 g, as always).

2. Place the tablet in a 100 mL beaker. Clean and dry a 50-mL graduated cylinder. Measure 30 mL of 1 M HCl(aq) in the graduated cylinder and then pour it into the beaker containing the tablet.

$$CaCO_3(s) + 2\,HCl(aq) \rightarrow CaCl_2(aq) + CO_2(g) + H_2O(l) \quad (1)$$

Recall 1 M HCl means there is 1 mole of HCl per liter of solution. Therefore, we are adding (30 mL)(1 mole HCl/L)(1 L/1000 mL) = 0.03 moles of HCl. This is enough HCl to react with 0.015 moles of $CaCO_3$ (note the 1:2 stoichiometry in the above reaction). This is an excess of HCl since all of the tablets contain less than 0.015 moles of $CaCO_3$. By using an excess of HCl, the $CaCO_3$ becomes the limiting reactant and determines how much product will be formed.

3. Allow the tablet to dissolve, it may take a few minutes.

If it is taking a while, you can speed up the dissolution by using a glass stirring rod to crush the tablet and stir the mixture. Not all of the tablet will dissolve, only the calcium carbonate will. The binders and fillers will still be insoluble. Note: once you put the stirring rod in the solution you should leave it in the solution. If you remove it, any liquid (containing calcium ions) clinging to the rod will be lost.

4. While the tablet is dissolving, prepare a hot water bath by adding about 100 +/- 25 mL of hot tap water to a 250-mL beaker.

Caution: the hot water from our taps can get very hot. Be careful not to burn yourself!

5. After the foaming has pretty much subsided (the generation of $CO_2(g)$ indicates the reaction is still proceeding), place the beaker containing the reaction mixture in the hot water bath for a couple of minutes.

Heat usually speeds up chemical reactions. If you stir the mixture and don't see any more bubbles forming, the reaction is done.

6. While the beaker is sitting in the bath, prepare to do a gravity filtration.

To do this you will need a 125-mL Erlenmeyer flask, a funnel, and a piece of 110 mm (large) filter paper. Place the funnel in the top of the Erlenmeyer flask. Fold the piece of filter paper in half, and then fold it in half again. **Do NOT crease the folds**; this weakens the paper and makes it more likely to tear. Open it into a cone (one layer thick on one side and three layers thick on the other). Place the filter paper cone into the funnel and wet it down by spraying it with distilled water from a spray/wash bottle to hold it in place. There should be a supply of wash bottles next to the back sink (or sometimes the other sinks, as well).

7. Remove the beaker from the water bath.

If it is too hot to handle, use your crucible tongs to remove it.

8. Pour the solution from the beaker into the funnel.

Add as much liquid as possible to the funnel, making sure that the liquid level does not rise above the top of the filter paper. Continue pouring the liquid into the funnel until all of it has been transferred. Spray the beaker and stirring rod (still in the beaker) with a few mL of distilled water to rinse them off. Pour this rinse water into the funnel. This insures every possible drop of solution containing calcium ions is collected.

9. Remove the funnel from the Erlenmeyer flask and discard the filter paper in the waste basket. If the solution in the flask is <u>not</u> clear: put a clean piece of filter paper in the funnel and repeat the filtration process (using a clean 125-mL Erlenmeyer flask).

If it is still not clear after the second filtration, continue with the experiment. This is not unusual.

10. Rinse the 100-mL beaker and funnel with distilled water, dry them, and return them to your drawer.

11. Measure 30 mL of 1.0 M $Na_2CO_3(aq)$ in a 50-mL graduated cylinder.

It's okay to add a couple of mL too much; like the HCl, it is being added in excess. But, don't add too little.

12. Slowly pour the 30 mL of 1.0 M $Na_2CO_3(aq)$ into the flask.

Two reactions will occur.

(a) The dissolved Ca^{2+} reacts to form $CaCO_3$:

$$CaCl_2(aq) + Na_2CO_3(aq) \rightarrow CaCO_3(s) + 2\,NaCl(aq) \quad (4)$$

(b) The excess HCl also reacts, as follows:

$$2\,HCl(aq) + Na_2CO_3(aq) \rightarrow CO_2(g) + H_2O(l) + 2\,NaCl(aq) \quad (5)$$

13. Let the flask sit for a couple of minutes to allow the $CaCO_3$ precipitate to form.

14. Check for completeness of the reactions. Visually, precipitate (solid) should be observed. Additionally, a piece of litmus paper can be used to test the pH of the solution. If the solution is still acidic, more Na_2CO_3 needs to be added.

Use a clean stirring rod to transfer a drop of liquid onto a piece of red litmus paper. Do not dip the stirring rod into the precipitate which may stick to it (causing a loss of product). The paper should turn blue (basic). If the paper is still red, add 5.0 mL more of the $Na_2CO_3(aq)$ and retest.

15. Take a one-inch piece of labeling tape and attach it to the bottom of a clean watch glass. Write your station number on the tape. Place a dry piece of 42.5 mm (small) filter paper on the watch glass and weigh them together. Record the mass on your data sheet.

16. Place the (just-weighed) filter paper in the funnel of a suction-filtration apparatus and collect the solid precipitate by suction filtration, as described in the Techniques section (see the next page).

Be sure to rinse the beaker thoroughly to insure complete transfer of precipitate. Remember this time it is the solid precipitate that you want to keep.

17. If solid remains in the Erlenmeyer flask after all of the liquid has been transferred, use distilled water to rinse out the beaker so that all of the precipitate gets transferred.

18. Draw air through the funnel until all of the liquid is gone and then continue drawing air for another five minutes to partially dry it.

19. Turn off the suction and carefully remove the filter paper and product from the funnel and place it on the watch glass. Do not lose any of your solid. (A spatula or tweezers can be used to facilitate this transfer.)

20. Pour the solution from the filtration flask into a clean, but not necessarily dry, beaker.

A) If there is no precipitate in the solution (normally the case), you can dump the solution down the drain.

B) If you see any precipitate, you need to refilter the solution. Weigh a new piece of filter paper, reassemble the suction filtration apparatus and repeat the filtration.

If you need to refilter, you will need to add the second piece of filter paper to the same watch glass and add the weights together.

Either your filter paper tore or it wasn't wetted down sufficiently.

21. Place the watch glass and filter paper into the oven for 15 minutes to dry.

Try to remember where you put your watch glass. It is recommended that the top shelf of the oven be used since occasionally rust from the top shelf drifts down to land on products on the lower shelves. Be careful not to disturb other students' products.

22. Use crucible tongs to remove the watch glass from the oven. Inspect your product. If there are any chunks, use a spatula to break them up. If the product appears to be wet, or chunks were present, put your product back in the oven for another 5 to 10 minutes to complete the drying.

23. Use crucible tongs to remove the watch glass from the oven. Carry it back to your station and allow it to cool for 5 minutes. Weigh the sample (precipitate + filter paper + watch glass) on the same balance used earlier. Record the mass on your data sheet.

When a sample is hot it heats the air around it causing the air to rise. The rising air current affects the balance reading. Thus, the sample must be allowed to cool before it is weighed.

24. Dispose of the product and filter paper in the appropriate waste container in the hood. All liquids can go down the drain. Wash and store your glassware. Wash your hands.