2: Standardization of Acids and Bases (Experiment)

Introduction

This experiment affects several experiments that follow. You should strive for maximum precision in this experiment since inaccuracies here will result in inaccuracies later.

Safety Precautions

Be especially careful when using the strong acid or strong base solutions as they can cause severe burns. Since concentrated HCl has a pungent odor it would be wise to dispense this solution in the hood. All waste solutions may be disposed of by rinsing them down the drain.

Part I. **Preparation of Solutions**

1. An approximately 0.1 M NaOH solution will be prepared by dilution of a 50 weight-percent NaOH solution. This solution has a density of 1.53 g/mL. Compute the volume of this solution that is required to prepare 1000 mL of 0.1 M NaOH. If you are unsure of your answer, confirm it with your TA.

2. Obtain 1000 mL volumetric flask, add around 400-500 ml (halfway) of deionized water from the proper carboy in the laboratory. Using the 50% by mass stock solution, measure the calculated volume of NaOH solution into a clean, dry 10 mL graduated cylinder.

3. Pour the contents of the graduated cylinder into your flask. Fill the graduated cylinder with five successive 10 mL portions of deionized water, each time emptying it into the flask.

4. Swirl then add more deionized water until the final volume of the system is 1000 mL. Pour the contents into the 1 L storage bottle and cap the bottle.

5. Label the bottle with chemical name and concentration. Allow the solution to return to room temperature. Sodium hydroxide solutions should never be left uncapped. Base solutions will absorb carbon dioxide from the atmosphere, thereby changing the titer of the solution.

Question 2.1

Give the balanced chemical equation for the formation of carbonic acid and for the reaction of carbonic acid with sodium hydroxide.

6. An approximately 0.1 M HCl solution will be prepared by dilution of concentrated HCl, which is about 12 M. Compute the volume of this solution that is required to prepare 1000 mL of 0.1 M HCl. If you are unsure of your answer, confirm it with your TA. **DO NOT REMOVE THE CONCENTRATED HCl BOTTLE FROM THE FUME HOOD!**

7. Obtain 1000 mL volumetric flask, add around 400-500ml (halfway) of deionized water from the proper carboy in the laboratory. Measure the calculated volume of concentrated HCl solution into a clean, dry 10 mL graduated cylinder. Pour the contents of the graduated cylinder into your flask.

8. Always remember to add acid to water and never the reverse. Swirl then add more deionized water until the final volume of the system is 1000 mL. Pour the all the contents into the 1L storage bottle and cap the bottle. Label the bottle with chemical name and concentration. Allow the solution to return to room temperature.

Question 2.2

Why do we always add acid to water and never the reverse?

Part II. Standardization of the NaOH Solution

1. (Please note: KHP has already been dried in the oven for your convenience). Label three 125 mL Erlenmeyers Flasks (ensure that they are cleaned and dried.)

2. On a weigh boat, weigh out approximately 3 grams of KHP, and record the value. Keep the KHP and weigh boat on the balance.
3. From this mass of KHP, take about 0.7-0.9 gram of material and transfer it to one of the Erlenmeyer Flasks. Record the new mass of KHP on the weigh boat. The difference in masses is the mass of KHP in your Erlenmeyer Flask.
4. Repeat step 3 for the remaining Erlenmeyer Flasks. This process of weighing starting material is weight by difference.
5. To each of the Erlenmeyer flasks, add 50-75 mL of deionized water and 2 drops of phenolphthalein indicator solution. Against a white background, titrate each flask with your NaOH solution to the first appearance of a faint pink color that persists for 30 seconds. You will have to learn to "split" drops to get a very faint pink endpoint. Tip: you should use a black-lined background card to accurately read the meniscus.
6. Wash down the sides of the flask with deionized water using your water bottle to be sure all the reactants are in the solution.
7. Given that the molecular weight of KHP is 204.23 and that it is a monoprotic acid, calculate the molarity of NaOH for each of your samples. Your triplicate molarities should agree to within three parts per thousand. Calculate the average, standard deviation, 95% confidence limit, and relative deviation. This is easily done on a computer spreadsheet.
8. **Carefully cap and store the solution for use in later experiments.**

Question 2.3

Why does it not matter how much water you add when dissolving the acid or when carrying out the titration?

Part III. Unknown KHP Analysis

You will be given an unknown sample of KHP (KHC8H4O4). Analyze the sample in the same way that you standardized your NaOH solution. A brief outline of the procedure is described below.

1. Label three 125 mL Erlenmeyers Flasks (ensure that they are cleaned and dried.)
2. (An assigned unknown will be provided by your TA). Weigh by difference three samples of your dry, cooled unknown KHP into three separate 125 mL Erlenmeyer Flasks.
3. On a weigh boat, weigh out approximately 3.3 grams of unknown KHP, and record the value. Keep the KHP and weigh boat on the balance.
4. From this mass of KHP, take about 0.9-1.1 grams of material and transfer it to one of the Erlenmeyer Flasks. Record the new mass of KHP on the weigh boat. The difference in masses is the mass of KHP in your Erlenmeyer Flask.
5. Repeat step 4 for the remaining Erlenmeyer Flasks.
6. Dissolve in deionized water and titrate with standard NaOH.
7. Calculate the mass percent of KHP in each sample. Report an average, standard deviation, 95% confidence limit, and relative deviation.
8. **This is a good stopping point for day 1 of the lab. Please clean all glassware and report to TA before leaving.**

Question 2.4

Write a balanced chemical equation for this reaction.

Part IV. Standardization of HCl Solution

This is a more complicated procedure than the one you have just accomplished. The reason for this is that there are very few primary base standards. One of the best is sodium carbonate. However, the use of this standard is complicated by the fact that carbonate is a weak acid and that a final product of the titration is a gas. The two acid/base reactions are:

$$\mathrm{H^+ + CO_3^{2-} \rightleftharpoons HCO_3^-} \tag{2.1}$$

$$\mathrm{H^+ + HCO_3^- \rightleftharpoons H_2CO_3 \longrightarrow H_2O + CO_2(g)} \tag{2.2}$$

These two reactions combine to from a solution which is called a buffer and which is described in Chapter 10. We will boil the solution near the end of the titration to drive out the carbon dioxide and thus destroy this buffer and get a much sharper endpoint.

Procedure

1. After the $\mathrm{Na_2CO_3}$ has dried for at least 1 hour, remove the beaker from the oven using tongs, and set it on a pad of paper on the bench top.

2. Allow it to cool there until you can put the inside of your forearm against the beaker without discomfort. Then put the beaker into a desiccator and allow it to cool for at least another half-hour. The weighing bottle should remain uncapped throughout this operation.
3. Label three 125 mL Erlenmeyers Flasks (ensure that they are cleaned and dried.)
4. On a weigh boat, weigh out approximately 0.8 grams of Na_2CO_3, and record the value. Keep the Na_2CO_3 and weigh boat on the balance.
5. From this mass of Na_2CO_3, take about 0.2 to 0.25 grams of material and transfer it to one of the Erlenmeyer Flasks. Record the new mass of Na_2CO_3 on the weigh boat. The difference in masses is the mass of Na_2CO_3 in your Erlenmeyer Flask.
6. Repeat step 5 for the remaining Erlenmeyer Flasks.
7. Dissolve the first sample of sodium carbonate in 50 mL of deionized water and add 20 drops of bromocresol green indicator. Titrate the solution with HCl until the solution *just* changes from blue to green.
8. Wash down the sides of the flask with deionized water using your water bottle to be sure all the reactants are in the solution. Boil the solution for 2 to 3 minutes, during this time the color should revert back to blue. If it does not, then a measured amount of base must be added to change the color to blue and this volume of added base must be included in the calculations. Cool to room temperature, and complete the titration to the final green endpoint.

Question 2.5

What is the reason for boiling the solution? Why is it important?

6. Compute the HCl molarity. Report an average, standard deviation, 95% confidence limit, and relative deviation.

Question 2.6

Write balanced equations for the reactions involved in this standardization.

7. **Carefully store and cap the solution for use in later experiments.**

Part V. Unknown Soda Ash Analysis

Obtain an unknown sample of soda ash from the stockroom. Soda ash is a mixture of sodium carbonate and an unreactive substance. Analyze the sample in the same way that you standardized your HCl solution. A brief outline of the procedure is described below.

1. Label three 125 mL Erlenmeyers Flasks (ensure that they are cleaned and dried.)
2. On a weigh boat, weigh out approximately 1.8 grams of unknown soda ash, and record the value. Keep the unknown soda ash and weigh boat on the balance.
3. From this mass of unknown soda ash, take about 0.56 to 0.60 grams of material and transfer it to one of the Erlenmeyer Flasks. Record the new mass of unknown soda ash on the weigh boat. The difference in masses is the mass of unknown soda ash in your Erlenmeyer Flask.
4. Repeat step 3 for the remaining Erlenmeyer Flasks.
5. Dissolve in deionized water and titrate with standard HCl.
6. Calculate the mass percent of sodium carbonate in each sample. Report an average, standard deviation, 95% confidence limit, and relative deviation.

Clean-up

After the experiment is completed, drain any remaining solution from the burette. Rinse each burette with deionized water. Then, fill the burette with deionized water and carefully place it in the burette rack.

Helpful hint

You will use your burette in many of the Experiments in Chemistry 4B. **It is important that you always clean it at the end of the day and fill it with deionized water before storage**. It is also important that when you use a burette that you follow the recommended procedure for changing a solution. First empty the burette out the top and half-fill it with deionized water. Open the stopcock and drain ~5 mL out the tip. Empty the burette out the top and repeat the water washing, this time also opening the stopcock when the burette is inverted to drain most of the water from the tip. Wait ~30 seconds for drainage and close the stopcock.

Add the new solution to about the 48 mL mark. Drain some of it out the tip and close the stopcock. At the sink, cradle the top of the burette between the thumb and index finger of one hand and turn the burette horizontal. Then twirl the burette and slowly empty it through the top, being careful to wet the entire interior wall with the new solution. Repeat this operation two more times. Finally, fill the burette above 0.00 mL mark and drain the excess out the tip until a reasonable starting mark is reached. Follow this procedure whenever you change the solution in a burette.
