Experiment 5
Chem 313

Preparation of Standard Sodium Hydroxide Solution and Titration of a Commercial Vinegar Product

In this experiment we prepare a standard solution of NaOH, and then use it to determine the concentration of acetic acid in vinegar. We will require knowledge of the exact concentration of the NaOH solution, but it is not convenient either weigh solid NaOH, because it is hydroscopoic. Therefore, we must use a primary standard to determine the sodium hydroxide concentration to four significant figures.

The primary standard that is most often used for standardizing sodium hydroxide solutions if potassium hydrogen phthalate, KHP. KHP of very high purity can be purchased, dried it in an oven, cooled it in a desiccator, and a sample can be accurately weighed on an analytical balance. After dissolving a known amount of KHP in distilled water, one can titrate the resulting solution with a sodium hydroxide solution. The volume of the sodium hydroxide solution required to reach the equivalence point and the mass of KHP is used to calculate the concentration of the sodium hydroxide solution to 4 or 5 significant figures. Once the NaOH solution is standardized it can be used to analyze acidic solutions, such as vinegar.

The presence of carbon dioxide is another reason why NaOH can not be used as a primary standard. Water can readily absorb carbon dioxide. Dissolved carbon dioxide acts as a weak acid in aqueous solutions.

$$CO_{2(g)} \leftrightarrow CO_{2(aq)}$$
$$CO_{2(aq)} + H_2O \rightarrow H_2CO_3 \rightarrow HCO_3^- + H^+$$

If basic solutions are prepared using water containing dissolved carbon dioxide, a portion of the base reacts with HCO_3^- to yield CO_3^{2-}.

Sodium hydroxide solutions are often prepared by diluting a 50 % aqueous solution of sodium hydroxide to the desired concentration, followed immediately by standardization against a primary standard. This removes possible sources of error due to varying amounts of carbon dioxide, because the dissolved CO_2 precipitates out as sodium carbonate in a 50 % NaOH solution.

We will also be using water that has recently been boiled to prepare all of the solution in this experiment. Boiling drives the above equilibriums to the left, driving off $CO_{2(g)}$ and dramatically reducing the bicarbonate concentration.

KHP is a weak acid. It is the intermediate species of the phthalic acid system
$Ka_2 = 3.90*10^{-6}$ $pKa_2 = 5.408$

KHP titration
$C_8H_5O_4^- + OH^- \rightarrow H_2O + C_8H_4O_4^{2-}$

Prelab Assignment

A 0.5123-g sample of KHP (FW 204.23) was dissolved in about 25 mL of distilled water, and titrated to the phenolphthalein end point with 28.75 mL of a sodium hydroxide solution. Calculate the molar (M) concentration of the hydroxide solution.

Apparatus
250 mL Erlenmeyer Flasks
50 mL burette
10 mL graduated cylinder
100 mL graduated cylinder
1000 mL boiling flasks
1 L polyethylene bottles
Weighing bottle for drying KHP

Chemicals
Vinegar
Phenolphthalein solution
Potassium hydrogen phthalate solid
50 % sodium hydroxide solution

PROCEDURE
PART A:

1. Dry the KHP in a 105 °C oven for at least one hour. Use your weighing bottle, without lid, to hold the solid. Label a 150-mL beaker, put the weighing bottle in the beaker and cover the beaker with a watch glass.

2. Clean the following glassware thoroughly; *25 mL pipet*, 250 mL volumetric, and a buret.

3. Add about 6 mL of the 50 % NaOH solution using a 10-mL graduated cylinder to a 1-L bottle. Fill the bottle almost to the top with the boiled water, screw on the cap, and mix the solution thoroughly. (Leaving little head space and screwing on the cap will prevent CO_2 in the atmosphere from dissolving into your NaOH). Label it! ***Handle the 50% NaOH with care!! Avoid contact with your skin or clothes. If you do spill some on yourself wash it off at once with a large amount of water.***

4. Obtain about 30 mL of vinegar. There will be two different brands available. Write down the brand of the vinegar that you sample. Pipet a 25 mL of this sample into a 250 mL volumetric flask. Dilute to the mark with the de-ionized water and mix thoroughly. Label it!

5. Pipet 25-mL aliquots of the diluted vinegar solution to five 250 mL Erlenmeyer flask. Add 50 mL of de-ionized water and 3 drops of phenolphthalein indicator and mix thoroughly. Label the flasks.

6. Rinse a clean 50-mL burette with 25 mL of your sodium hydroxide solution. Fill it with approximately 45 mL of the sodium hydroxide solution. Record the level of the of the NaOH solution to the nearest ±0.01 mL. Take your time with this to really understand how to read and work the burette.

7. Take the KHP out of the oven. Place it in a desiccator to cool for 15-20 minutes (until cooled to room temperature).

8. Titrate the diluted vinegar in the five labeled Erlenmeyer flasks. The endpoint is reached when the solution changes from colorless to faint pink. Be careful not to overshoot the endpoint. One drop takes it from clear to faint pink. Be sure to refill burette with NaOH in between titrations. Also, make sure that you recap your 1 L-bottle in between fills.

9. Weigh five portions of 0.4-0.7 g to the nearest 0.1 mg. Place each portion in a labeled 250-mL Erlenmeyer flask. Add about 30 mL of the boiled water, 3 drops of phenolphthalein indicator and mix thoroughly.

10. Titrate each of the five samples to the same phenolphthalein end point as with the vinegar samples.

11. Carefully label your NaOH solution.

12. Trade data with a classmate that sampled from the other brand of vinegar

PART 2 CLEAN-UP

Rinse the buret, pipet and volumetric flask with several (10) portions of de-ionized water and put them away. (**THIS IS CRITICALLY IMPORTANT). If it is not done the pipet and buret will essentially be destroyed.**

Use a brush and soapy water to wash the Erlenmeyer flasks. **(SCRUB!)**

Data Analysis and Discussion Points:

1. With the data from each of you each of your six KHP samples calculate values for the concentration of your NaOH solution. (5 pts)

2. Calculate the mean, s_e, s_m, and 95% CL of the concentration of your NaOH solution. (5 pts)

3. Calculate the mean, s_e, s_m, and 95% CL of the volume of NaOH used to titrate the diluted vinegar samples (5 pts)

4. Using the mean [NaOH] and the mean V* for the diluted vinegar titration, calculate the percentage of acetic acid in the vinegar (w/v). (5 pts)

5. Propagate the uncertainties in this calculation to estimate the uncertainty in the % acetic acid (use s_e values for [NaOH] and V^* and the uncertainties in the pipet and volumetric flask for the propagation). (5 pts)

6. Repeat 1-6 with your classmate's data. (25 pts)

7. Using your estimate of the propagated uncertainties obtained from Question 5 as estimates of standard deviations, s_e, determine whether the percentage of acetic acid in the different brands of vinegar is statistically different to the 95 % CL. (5 pts)

8. Produce a Master Table that summarizes the results from this experiment and incorporate it into your report. (5 pts)

Lab Report

Abstract (10 pts)

Brief procedure (less detail then in the write-up) (10 pts)

Data (10)

Data Analysis and Questions (60 pts)

Discussion (10 pts)