

Experiment 4

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 hygroscopic. 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 is potassium hydrogen phthalate, KHP. KHP of very high purity can be purchased, dried in an oven, cooled 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. We will be saving part of our sodium hydroxide solutions for use in future experiments so it is critical that the standardization is performed as carefully as possible.

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.



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 equilibria to the left, driving off $\text{CO}_{2(\text{g})}$ and dramatically reducing the bicarbonate concentration. The department has a still in the adjacent laboratory and fresh water was brewed the morning of this experiment.

KHP is a weak acid. It is the intermediate species of the phthalic acid system

$$K_{a2} = 3.90 \times 10^{-6} \quad pK_{a2} = 5.408$$

KHP titration



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

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 to the shoulder with the boiled water, screw on the cap, and mix the solution thoroughly. 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 distilled 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 distilled 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 with your name and store in one of the drawers at your station.
12. Trade data with a classmate that sampled from the other brand of vinegar

Questions:

1. With the data from each of you each of your five KHP samples calculate values for the concentration of your NaOH solution.
2. Calculate the mean, s_e , s_m , and 95% CL of the concentration of your NaOH solution.
3. With the data from each of you each of your five vinegar samples calculate values for the percent acetic acid in the vinegar (w/v).
4. Calculate the mean, s_e , s_m , and 95% CL of the concentration of your NaOH solution percent acetic acid in the vinegar (w/v).
5. Repeat questions 1-4 with your classmate's data.
6. Determine whether the percentage of acetic acid in the different brands of vinegar is statistically different to the 95 % CL.