Chemical Technology Program
General Education & Sciences Division
Cape Fear Community College
Wilmington Campus

Lab Activity: Quality Control Standardization of NaOH

STANDARDIZATION OF SODIUM HYDROXIDE

Chemical Technology Program, Cape Fear Community College
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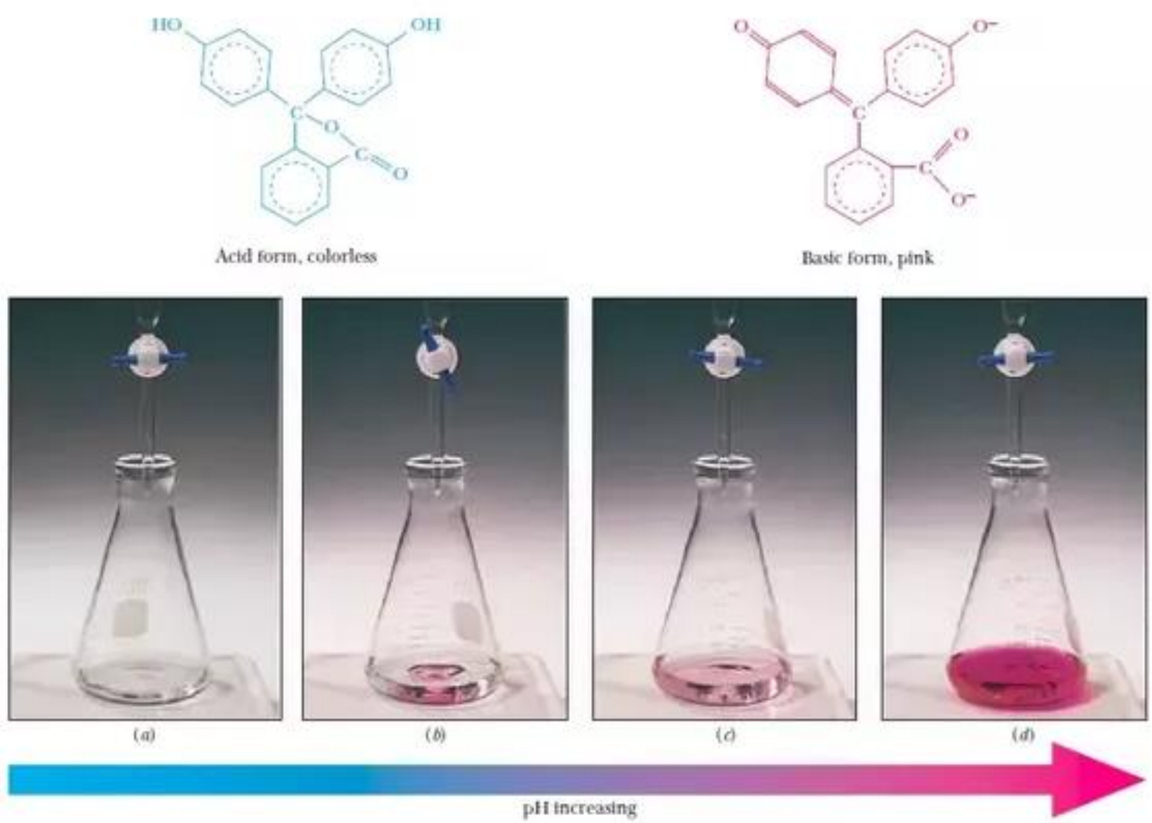
A **titration** is one type of analytical procedure often used in a wet laboratory. In a titration, an one substance is reacted with a known amount of another substance. Typically, an analyst will perform a titration to determine the concentration of a sample provided to them in a laboratory. The **endpoint** of a titration (the point at which the reaction is complete) can be determined by using an **indicator** which will change colors at a specific pH value. One of the most common indicators is phenolphthalein which is colorless in an acidic solution and pink in a basic solution.



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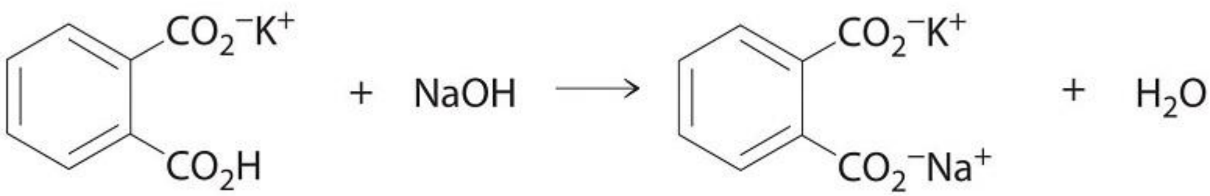
When performing any titration, it is critical to know the exact concentration of the titrant (the solution in the burette which will be added to the unknown). The exact concentration of the titrant must be determined to perform calculations on the sample being tested. **Standardization** is the process of determining the exact concentration of the titrant. Often, concentration is provided in terms of molarity (M), however normality (N), milligrams per milliliter (mg/mL), parts per million (ppm), and percentage (%) may also be reported.

In a working environment, it is not uncommon for laboratory analysts to perform various **quality control** tests on raw reagents and materials or finished products. A user should always confirm the identification and quality of the reagent or product that is used in a laboratory environment. For example, if a laboratory orders a 6 M sodium hydroxide solution, the concentration should be checked before it is used. In this laboratory procedure, you will make a solution of sodium hydroxide. However, your actual concentration will be different than the targeted concentration. Therefore, it is imperative that you standardize the solution. Standardization helps an analyst determine the actual concentration of a solution prepared in the laboratory. Sodium hydroxide will be standardized using potassium hydrogen phthalate (KHP) and using phenolphthalein as the indicator.

Reaction of KHP with Sodium Hydroxide



KHP

Notice in the above reaction, one molecule of KHP will react with one molecule of NaOH to procedure a “salt” and water. This ratio will become important in the standardization calculations.

When molecules react to form products, amounts of reagents are often important. The term used for “amount” in a laboratory environment is “**mole**”. In your daily life, the word “dozen” often means an amount: 12 items. Mole is similar; it is a term that represents a certain number of molecules or atoms (known as **Avogadro’s number**: 6.022×10^{23}). Mole is a universal term that can be applied to any atom or molecule. However, to calculate this value, the substance’s molecular mass is important. To calculate the number of moles of substance, an analyst should divide the starting mass by its molecular mass.

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For example:

Water has a molecular mass (MW) of 18.02 g/mol. A person uses 3.4516 g of water in an experiment. How many moles of water are used?

moles = grams / MW
moles = 3.4516 / 18.02
moles = 0.1915

To calculate concentration of a substance (known as **Molarity**), an analyst will need to take the moles of substance and divide by the volume (in liters) of the solution. For example:

Sodium chloride was used in the laboratory and a solution was made. An analyst took 2.5831 grams of sodium chloride and dissolved it in 250 mL of water. What is the concentration (in Molarity)?

moles = 2.5831 g / 58.44 g = 0.04420 moles
Molarity = moles / L
M = 0.04420 / 0.250 L
(since 250 mL = 0.250 L)
M = 0.177

INDUSTRY CONNECTION:

You are an employee of the Kraft Heinz Company, the third-largest food and beverage company in North America and the fifth-largest food and beverage company in the world. As a R&D employee, you work in their wet laboratory as a technician that performs various tests on their products. One product, distilled white vinegar, has been submitted to the laboratory for analysis. The label states that the vinegar product should contain 5% acidity. Your laboratory supervisor would like for you to confirm this statement by titrating a sample of vinegar with sodium hydroxide.

However, before the titration can be attempted, you need to confirm the actual concentration of NaOH provided to you by the laboratory. To accomplish this, the NaOH will need to be standardized. Only when the NaOH is standardized, can the vinegar sample can be titrated.

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C-TEACH ACADEMY
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OBJECTIVES

In this experiment, you will

- Standardize a solution: base: Sodium Hydroxide (NaOH).
- Determine the actual concentration of sodium hydroxide presented to the lab.

MATERIALS

Burette, 50 mL
Ring Stand
Magnetic Stirrer
Magnet
125-mL Erlenmeyer flask

Sodium hydroxide solution
Phenolphthalein indicator
Potassium Hydrogen Phthalate (KHP)
Analytical or Top-Loading balance

PRE-LAB EXERCISE

Complete the pre-lab exercise below. These questions should only be attempted if you have covered the basics of a titration and the concept of moles and molarity.

1. In the standardization method, potassium hydrogen phthalate will be used. What is the formula weight (g/mol) for KHP?

2. If an analyst weighs 0.2156 g of KHP, how many moles of KHP are present?

3. If 0.00415 moles of KHP were used in the standardization method, how many moles of sodium hydroxide (NaOH) would it take to neutralize this amount?

4. During a standardization method, the endpoint to neutralize 0.00415 moles of KHP was reached at 26.5 mL of sodium hydroxide. Based on these values, what is the concentration of NaOH?

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PROCEDURE

Part I: Preparation and Standardization of Sodium Hydroxide

1. Obtain and wear goggles.

2. You will need to make a 0.1 M NaOH solution. You will need to make 500 mL. Therefore, you will need to use 2.000 g of sodium hydroxide and dissolve it in a 500 mL volumetric flask. Do not fill to the line! Ensure that all crystals are dissolved and that the solution has cooled down before filling to the meniscus. Once cooled and dissolved, fill to the line.

<u>Math to Prove Why 2.000 g is needed:</u>

M = mol / L
0.100 = mol / 0.500
0.0500 = moles of sodium hydroxide needed
0.0500 moles x MW (of NaOH) = grams needed
0.0500 moles x 40.00 = 2.00 g

3. Set up a burette, a burette clamp, and ring stand (see Figure 1). Rinse and fill the burette with your ~0.1 M sodium hydroxide (NaOH) solution.

Figure 1: Burette Assembly

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4. Using a balance, measure approximately 0.300 g of potassium hydrogen phthalate (KHP). Record the exact weight in your data sheet. Add the KHP to the 125-mL Erlenmeyer flask and add enough deionized or distilled water to fully dissolve the crystals. You should not need more than 50 mL of water.

   <u>If using a magnetic stirrer:</u>
   Place the flask on a magnetic stirrer, beneath the burette of NaOH, and add a magnet. Turn the stirrer on to a low or medium-low setting. The magnet should turn and stir your solution.

   <u>If no magnetic stirrer is available:</u>
   You will need to hold the beaker and continuously swirl the solution throughout the addition of sodium hydroxide.

5. Add an indicator (phenolphthalein; phth) to the KHP solution. You will only need 2-3 drops of indicator. The color should not change (it should stay colorless)!

6. Using the burette, turn the stopcock and slowly add the sodium hydroxide solution to the flask containing the KHP and indicator.

7. Continue to add the NaOH to the solution. Your goal is to add NaOH until the indicator turns a faint pink. The pink color should persist for 30 seconds. Once this endpoint is reached, record the volume of NaOH required for the titration.

8. Repeat the procedure and titrate three more KHP samples.

CHEMICAL TECHNOLOGY:
Part II: Using Instrumentation to Standardization Sodium Hydroxide

In today's analytical laboratory environment, it is not uncommon for workers to use automated titrators. Overall, these systems are simplistic, easy to use, and will include automated end point detection. Some automated titrators are controlled by computer software and can be connected to autosamplers to increase sample throughput.

The machine will slowly add titrant, detects the endpoint, and calculates results based on report templates determined by the user. Automated titration is growing in popularity as they often improve accuracy, repeatability, safety, traceability, and meet requirements of regulatory compliance.

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The image below shows an Eco Titrator, available from Metrohm. This compact design does not require a computer or computer software for operation. It can perform potentiometric titrations that are common within the quality control (ie. confirming concentrations and label information), food (ie. acid content, Vitamin C), water quality (ie. hardness), and pharmaceutical (ie. USP monographs for most acids and bases) fields.

Eco Titrator from Metrohm

If an automated titrator is available: Complete Steps 9-11:

9. Once four hand titrations have been completed, measure another sample of KHP, transfer it to a 250 mL beaker, and dissolve it as before.

10. Take the KHP sample to the instrumentation room and titrate it using the automated titrator. You will need one successful titration. If the instrument malfunctions, you will need to reattempt. (Note: The automated titrator will NOT have YOUR sodium hydroxide solution. It will have a pre-loaded sodium hydroxide solution with an unknown concentration.

11. Save and print the data report sheet. You will need to attach it to your laboratory submission.

12. Once everyone completes their titration, use the data to calculate classroom statistics.

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Part III: Calculations & Excel

13. Using the equations in the background information, calculate the "actual" concentration of sodium hydroxide used in the manual titration portion of the laboratory experiment. A data sheet is provided. Once raw data has been recorded, complete the Excel spreadsheet template.

14. Using statistics, calculate an average and standard deviation of the concentration via Excel. Do not include data from the automated titrator (it a different source of NaOH).

15. Transfer any remaining sodium hydroxide into a container and save this solution for future titrations (in other lab experiments). Alternatively, the laboratory instructor will collect and combine the NaOH solutions from everyone and use it for future experiments. Do **not** store in a container with a **glass** stopper.

16. Rinse and clean the volumetric flask and all other glassware.

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DATA ANALYSIS

1. **Please complete the table below, reporting your laboratory data. An Excel spreadsheet has been provided. Complete all calculations using Excel. DO NOT manually type in results that have been performed by a calculator (Excel should do all calculations)!**

Standardization of Sodium Hydroxide:

	Trial 1	Trial 2	Trial 3	Trial 4
Mass of KHP (g)				
Moles of KHP				
Moles of NaOH				
Initial Volume, NaOH (mL)				
Final Volume, NaOH (mL)				
Volume of NaOH used (mL)				
Volume of NaOH used (L)				
Concentration (M)				
Average				
Standard Deviation				
%RSD				

Chemistry connection/check: In this titration, one molecule of sodium hydroxide will react with one molecule of KHP. Therefore, at the endpoint, the amounts of KHP and NaOH should be the same. To calculate the concentration, the formula weight of KHP is needed (204.22 g/mol).

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Shortcut method to double check your calculations from above

$$\text{Molarity} = \frac{(\text{g KHP} * 1000)}{(204.22 * \text{mL NaOH})}$$

2. **Attach your instrumentation report (automated titrator) to this data sheet.**

3. **Suppose that the quality control department required you to prepare a solution of sodium hydroxide with a concentration of 0.100 +/- 0.040. Does your sample "pass" the laboratory regulations?**

4. **Calculate the number of moles of sodium hydroxide in each of the following (MW: 40.00 g/mol):**

Sample	Grams of NaOH	Moles
1	**0.1245 g**	
2	**2.3314 g**	
3	**14.2497 g**	

5. **If each of the above amounts were dissolved in 250.0 mL of water, what is the concentration (in M) of each solution?**

Grams of NaOH	M
1	
2	
3	

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6. **Calculate the number of moles of KHP in each of the following (MW: 204.22 g/mol):**

Sample	Grams of KHP	Moles
1	0.1245 g	
2	2.3314 g	
3	14.2497 g	

7. **If each of the above amounts were dissolved in 250.0 mL of water, what is the concentration (in M) of each solution?**

Grams of KHP	M
1	
2	
3	

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