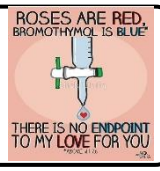
Lab 20G: Standardization of NaOH



Name:_____ **Block:** _____
Group Members:_____ **Date:** _____**/** _____**/ 2018**
Due Date:_____ **Drop Date:**_____

The report is submitted in full, **on the due date**. If you are absent on the day, the report is expected to be submitted electronically. Late reports are penalized, and will *not accepted past the drop date*.

Objective

To make a standard solution of Potassium hydrogen phthalate $C_8H_5KO_4$ (KHP).
To standardize an unknown sodium hydroxide solution.

Criteria	Student Self Evaluation	Teacher Assessment
Flow Chart: a flow chart diagram of the procedure completed individually by each group member ***before the lab****! (for part 2 of the lab only)* **Pre-Lab Questions:** displays a critical understanding of the background theory.	**/9**	**/9**
Data, Results: *(word processed)* Provides results & detailed observations (and diagrams where appropriate) that are presented in **correctly labelled** tables with **descriptive, numbered titles**.	**/4**	**/4**
Questions & Calculations: *(may be hand written **IF neat**, or typed up…all working out for calculations must be shown)* Correctly identifies and explains the theory relating to the experiment and supports this with accurate observations & data.	**/8**	**/8**
Presentation: Practical report is presented in the **correct format**, is written fluently and provides **appropriate section headings** and accurate referencing. ***Tables & graphs*** have numbered headings. Data & calculations may be hand written, however the remainder of the report is to be **word-processed.**	**/2**	**/2**
Conclusion: *(word processed)* Identifies and defines important concepts and principles relevant to the experiment by relating back to the objective and hypothesis. Be sure to address the points listed in the lab handout when answering the conclusion.	**/3**	**/3**
Practical: Demonstrates an organized and safe approach to experimental work during the lab. Shows maturity, cooperation and leadership during laboratory work. Titrated average volume falls within an accepted range. [NaOH] is accurate	**/8**	**/8**
Results Summary	**/34**	**/34**

We will be doing the lab on _____*. In order to be ready to go, you need to complete the following sections of your lab report:*

- *Flow Chart*
- *Pre-Lab Questions*
- *Data & Observations: Draw & set-up Table 1 & 2 into your lab notebook.*

Materials:

- ~500mL unknown [NaOH] *(approx.. 0.10M)*
- KHP (crystals)
- Phenolphthalein indicator
- Centigram balance
- Beaker (100mL)
- Beaker (250mL)
- Funnel
- Volumetric flask (250mL)
- Stopper (for Vol. Flask)
- Wash bottle
- Buret (50mL)
- 500mL Erlenmeyer Flask + stopper
- 250mL Erlenmeyer Flask
- 25mL Pipet
- Retort stand
- Buret clamp

Procedure:

**This experiment is based on Experiment 20G in Heath Laboratory Experiments page 253-260.*
See attached pages for reference & supplementary information you will find helpful.

Draw a flow chart for this experiment. For *PART II ONLY.* This should be **shown to your teacher** before beginning the lab.

NOTE CHANGES:

- *Students to complete Part I as a class and Part II in lab groups*
- Potassium hydrogen phthalate (KHP) KOOC-C_6H_4-COOH will be used **instead of oxalic acid**
- A 4-5th trial is **only** necessary should trial(s) 1-3 fall outside of an acceptable range due to errors or overshooting the equivalence point.

Safety glasses are to be worn at all times, for all experiments!

Reagent Disposal: all waste is to be collected in the **WASTE DISPOSAL**.

Clean Up: clean up all materials, wipe lab bench with disinfectant and wash hands *well* with soap and water before you leave the lab each day.

Pre-lab Questions: *(to be answered in full sentences)* *(8 marks)*
*Carefully read the pre-lab discussion; **attached** for reference & include supplementary information you may find helpful.*

1. Titration is a common method of quantitative analysis. What does titration involve? *(briefly explain equipment required and procedure)*
2. What is meant by a "primary standard"? What are the characteristics of a substance that can be used as a primary standard?
3. What substances are commonly used as a primary standard? What will we use?
4. It is impossible to make a *standard solution of NaOH* whose concentration is accurately known. Explain why.
5. Potassium hydrogen phthalate (KHP), KOOC-C6H4-COOH will be used as the primary standard to standardize NaOH. This primary standard has one acid proton per molecule. Write the balanced equation for this reaction.
6. Use basic stoichiometry to calculate [NaOH].

Mass of KOOC-C_6H_4-COOH	1.00 g
Volume of NaOH	35.60 mL

7. Calculate the mass of potassium hydrogen phthalate (KHP) KOOC-C_6H_4-COOH that you will need to make up 250.0 mL of a 0.0500M solution.

Data & Observations *(4 marks)*

*Tables are to be **prepared** as part of the **pre-lab** work. They should be **completed** neatly during the lab.*
*It is **always expected** that you make your own experimental observations and record data.*

Part I Preparation of a Primary Standard Acid

Table 1

Calculated mass of oxalic acid $H_2C_2O_4 \cdot 2H_2O$ (required for 250.0 mL of 0.0500*M* solution (g))	
Mass of beaker (g)	
Mass of beaker + oxalic acid (g)	
Mass of oxalic acid (g)	

COMPLETE IN YOUR NOTEBOOK

Part II Standardization of an Unknown NaOH Solution

Table 2 Volume of NaOH Needed to Neutralize 25.00 mL of Oxalic Acid

	TRIAL 1	TRIAL 2	TRIAL 3	TRIAL 4 (if necessary)	TRIAL 5 (if necessary)
Initial reading of buret					
Final reading of buret					
Volume of NaOH required (mL)					
Average volume of NaOH (mL)					

COMPLETE IN YOUR NOTEBOOK

*NOTE: if your first titration result is somewhat larger than subsequent results, do not include it in calculating your average (but do make note of this). Only then is "trial 4" required.

Questions & Calculations

PART I: Preparation of a Primary Standard Acid

1. Calculate the mass of one mole of KHP ($KOOC\text{-}C_6H_4\text{-}COOH$) (1 mark)
2. Determine the number of moles in the measure mass of KHP used. (1 mark)
3. Calculate $[KOOC - C_6H_4 - COOH \cdot 2H_2O]$ when the mass in table 1 is dissolved in 250.0mL of solution. (1 mark)

PART II: Standardization of an Unknown NaOH Solution

1. Calculate the number of moles of KHP in 25.00mL of standard solution. (1 mark)
2. Calculate the number of moles of NaOH required to neutralize this amount of KHP. *(note, KHP is a monoprotic acid; show all equations to explain)* (2 marks)
3. Calculate & use your average volume of NaOH required and the number of moles present as obtained above, calculate [NaOH] in the standardized solution in mol/L. (2 marks)
   ***NOTE:** if your first titration result is somewhat larger than subsequent results, do not include it in calculating your average (but do make note of this). (in the first titration, students often overshoot the endpoint)

Conclusion:

Your conclusion should summarize your experimental results and answer your objective. Be sure to discuss any experimental errors and how this may/or may not have impacted your results. (3 marks)

Practical Evaluation

In this lab, you will be given a practical evaluation, based on the quality of your data obtained. (4 marks)

Preparation and Standardization of Acid and Base Solutions, and Testing of Unknowns

EXPERIMENT 20G

A common laboratory procedure is to determine the concentration of an acid or base solution by titrating it against a solution of known concentration. In an earlier experiment (16B) you were introduced to the method of preparing a standard solution. In a subsequent experiment (20C) you performed an acid-base titration to determine the concentration of acetic acid in vinegar, having been given a sodium hydroxide solution of known molarity. In this experiment you will have to standardize the NaOH yourself.

You will first have to prepare a solution of an acid of known concentration by weighing out a sample and making it up to a known volume in a volumetric flask. To be suitable for such a use, a substance must be very pure and stable. Also, it must not absorb water from the air. A chemical such as this is called a *primary standard*. Sodium hydroxide cannot be used as a primary standard because it is difficult to obtain 100% pure, it readily adsorbs moisture from the air, and it reacts with the carbon dioxide in the air. A common primary standard for acid-base titrations is oxalic acid, which occurs in the crystalline form as the dihydrate $H_2C_2O_4 \cdot 2H_2O$ (or $(COOH)_2 \cdot 2H_2O$). This is the primary standard that you will use in Part I of this experiment. (It must be of analytical reagent purity.)

After preparing the solution of oxalic acid of known molarity, you will carry out a titration with sodium hydroxide solution in order to determine the molarity of the NaOH (Part II). This standardized NaOH can have a variety of uses, such as determining the molar mass of an unknown solid acid (Part III) or the molarity of an unknown acid solution (Part IV). Keep in mind that it is important to make all measurements with as much accuracy as possible.

In this experiment you will also have the opportunity to do some optional procedures which you will design yourself (Parts V and VI).

OBJECTIVES

1. to prepare a standard solution of potassium hydrogen phthalate (KHP) and use it to standardize an unknown sodium hydroxide solution

MATERIALS

Apparatus

centigram balance
beaker (100 mL)
beaker (250 mL)
funnel
volumetric flask (250 mL)
wash bottle
buret
pipet (25 mL)
suction bulb
pH meter (or universal indicator paper or solution)
stoppered bottle (500 mL)
label
stand
buret clamp
lab apron
safety goggles

Reagents

potassium hydrogen **phthalate** (KHP), KOOC-C6H4-COOH (crystals)
sodium hydroxide (NaOH) solution (approx. 0.1*M*)
phenolphthalein indicator

PROCEDURE

Part I Preparation of a Primary Standard Acid

CAUTION:

KHP is a toxic irritant.

Slightly hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.

Always use the suction bulb to withdraw the KHP into the pipet.

1. Before coming to the laboratory, calculate the mass of **potassium** hydrogen **phthalate** (KHP) KOOC-C6H4-COOH, that you will need to make up 250.0 mL of a 0.0500*M* solution.
2. Put on your lab apron and safety goggles.
3. Accurately determine the mass of an empty (clean and dry) 100 mL beaker and record it in Table 1 in your notebook.
4. Measure into the beaker the amount of oxalic acid that you have calculated you need and accurately determine the mass of the oxalic acid and the beaker. Record this figure in Table 1. Do not spend much time trying to get exactly the same mass as you calculated. The important thing is to record accurately the mass you do have, and to calculate the molarity from this mass. For example, the mass you use may give the solution a molarity of 0.0496*M*. This is perfectly acceptable, provided that you use this figure in your calculations.
6. Dissolve the KHP in water, and pour the solution through a funnel into a 250 mL volumetric flask. Wash the beaker with water twice, and add these washings to the flask. Now add water to the flask until the level is up to the mark. (Use a wash bottle as you get close to the mark.) Stopper the flask, and shake to ensure that the solution is homogeneous. You now have your standard solution of oxalic acid.

Part II Standardization of an Unknown NaOH Solution

1. Obtain a 500 mL bottle with a stopper and fill it with NaOH solution of unknown molarity. Label it with your name and class.

2. Add about 15 mL of the NaOH solution to a buret through a funnel, rinse it back and forth, then discard it through the tip into the sink.

3. Fill up the buret with more NaOH solution and allow some to drain in order to remove any air bubbles in the tip. Remove the funnel.

4. Using the suction bulb on the end of your pipet, withdraw about 5 mL of potassium hydrogen phthalate (KHP), rinse it around in the pipet, and discard it. Then withdraw 25 mL of the standard oxalic acid solution and transfer it to a 250 mL Erlenmeyer flask. The correct volume is delivered when you have touched the tip of the pipet to the side of the flask. Do not blow through the pipet. (*Note*: Depending on the shape and size of your pipet and volumetric flask, you may have to transfer the potassium hydrogen phthalate (KHP) first to a clean, dry beaker since the pipet will not reach deep enough into the volumetric flask.)

5. *Add 3 drops of phenolphthalein solution* to the acid in the Erlenmeyer flask.

6. Read the initial volume of NaOH in the buret as accurately as you can, and record it in Table 2. Then open the valve on the buret. Allow the NaOH solution to run into the flask and swirl constantly to ensure thorough mixing.

7. After a time, you will notice a pink color that appears where the NaOH enters the liquid in the flask. When this color takes a longer time to disperse and disappear, slow down the rate of addition of NaOH until eventually you are adding it a drop at a time. Stop the titration when the faintest possible pink color stays in the flask for about 20 s. Read the final volume of the NaOH in the buret and record it in Table 2. (The difference between the initial reading and the final reading represents the volume of NaOH required to neutralize the oxalic acid.)

8. If you are at all in doubt as to whether you have a pale pink color, take the reading anyway, then add one more drop. If the color immediately becomes much darker, the reading you took was probably the most accurate result. This is called the *endpoint* of the titration. Discard the solution down the sink.

9. Pipet another 25 mL sample of potassium hydrogen phthalate (KHP) into the flask and again add 3 drops of phenolphthalein. Refill the buret (if necessary) and repeat the titration. *Run in NaOH to within 1 mL of the volume needed in the first titration, then add the solution a drop at a time,* swirling after each drop, until you get the faint pink endpoint. Repeat the titration until you have two readings that agree to within 0.1 mL.

10. Dispose of your standardized NaOH (as instructed by your teacher).

CAUTION: Sodium hydroxide solution is corrosive to skin, eyes, and clothing. When handling NaOH, wear safety goggles, full face shield, gloves, and lab apron. Wash spills and splashes off your skin and clothing immediately using plenty of water. Call your teacher.

CAUTION: Phenolphthalein solution is flammable. Make sure there are no burner flames in the vicinity.

CAUTION: You must assume that any unknowns you are dealing with could be poisonous. Do not get any in your mouth and do not swallow any.