

Experiment 9: Preparing and Standardizing a Base

CH2250: Techniques in Laboratory Chemistry, Plymouth State University

Adapted from "6. Preparing Standard Acid and Base," *Experiments To Accompany Exploring Chemical Analysis, 4th Edition*, Daniel C. Harris (2008), available at <http://www.whfreeman.com/exploringchem4e>.

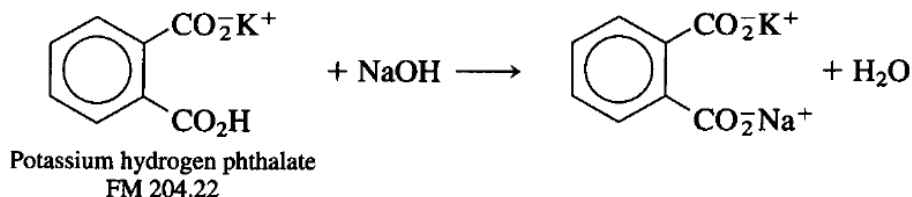
Suggested reading for background information: Section 10.5, *Exploring Chemical Analysis, 4th Edition*, Daniel C. Harris (2008).

NOTE: You will perform this lab AND Experiment 10a "Preparation of Sample for Measuring Manganese in Steel with Standard Addition" in the same lab period. Be sure to do the pre-lab for BOTH Experiment 9 and Experiment 10a before coming to lab!

Introduction:

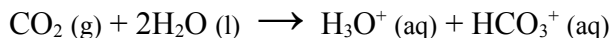
Hydrochloric acid and sodium hydroxide are the most common strong acids and bases used in the laboratory. It is impossible to purchase completely pure HCl or NaOH to be used in making solutions, so solutions of approximate concentrations are made, then standardized to learn their exact concentrations. In this experiment, you will prepare a Stock solutions of sodium hydroxide and standardize it by titration. This standardized solution will be used to analyze unknown samples in future labs.

NaOH is standardized by reacting with a known mass of potassium hydrogen phthalate:



Potassium hydrogen phthalate is a primary standard, meaning it can readily be purchased in very pure form and weighed out in such a way that you know to the greatest extent possible exactly how much material you have. However, the solid will rapidly absorb water from the air. It is possible to remove the water by heating the solid in an oven for several hours (this will be done for you before you arrive in lab), but care must be taken to store the material in a desiccator after it has been dried.

Whenever preparing and using any standardized base, it is important to remember the role of dissolved carbon dioxide on pH. Carbon dioxide dissolves in water to generate an acid:



In a solution containing a base, the produced hydronium ion reacts with hydroxide, driving the above reaction further to the right and changes the concentration of the base. In other words, the concentration of any solution of base exposed to air will, over time, slowly be reduced. Thus, if knowing the exact concentration of the base is important, the solution should be stored in a tightly sealed bottle with as little head space as possible, and it should be re-standardized every few weeks.

Equipment: Read through the procedures and make a list of the equipment you will need.

Safety Considerations: Read through the procedures and note any safety considerations.



Pre-lab work: In addition to the normal pre-lab write up (Title, Date, Purpose, etc.), you must perform the needed calculations in the Procedure (Steps 2 and 5) before coming to lab. **PUT THESE CALCULATION IN YOUR “PRE-LAB” SECTION**, not as part of the “Procedures.” Read through the Procedure and set up appropriate tables to record the data.

Procedure:

1. Boil about 500 mL of water in a large beaker for 5 min to expel CO₂. Pour the water into a polyethylene bottle, which should be tightly capped whenever possible. Place the bottle in ice to speed the process of cooling the water to room temperature. *Begin this step as soon as you walk into the lab, so the water will be cooled and ready to use when you need it.*
2. Calculate the volume of 50 wt% NaOH needed to prepare 500 mL of 0.025 M NaOH. (The density of 50 wt% NaOH is 1.50 g per milliliter of solution.) Use a graduated cylinder to transfer this much NaOH to the bottle of water. (CAUTION: 50% NaOH eats people. Flood any spills on your skin with water.) *Do this calculation as part of your pre-lab work!*
3. Mix the solution well and cool it to room temperature. Start cooling the solution by placing the bottle in ice.
4. Calculate the mass of solid potassium hydrogen phthalate needed to react with 25 mL of 0.025 M NaOH. *Do this calculation as part of your pre-lab work!*
5. Obtain the primary standard potassium hydrogen phthalate from your instructor. *Note: Potassium hydrogen phthalate will rapidly absorb water from the air. If you are not actively weighing it, this primary standard should be stored in a desiccator.*
6. Accurately weigh the mass of solid potassium hydrogen phthalate calculated in Step 4 and dissolve it in ~25 mL of distilled water in a 125-mL flask. *Use the "Mass Transferred" method of weighing. Do NOT waste time trying to weigh the "exact" amount you calculated.*
7. Add 3 drops of phenolphthalein to the flask
8. Do a quick titration to find the end point. *This sample will NOT be used in the final calculation of the concentration of NaOH.*
9. Repeat Step 6 for another sample of potassium hydrogen phthalate. Use the result from Step 8 to calculate the approximate volume of NaOH required to titrate this sample.
10. Carefully titrate the sample until it turns faint pink (the color should persist for 15 s, though it may slowly fade as CO₂ from the air dissolves in the solution). *It is helpful to place a sheet of white paper under the flask to help you see the pink solution.* During the titration, periodically tilt and rotate the flask to wash all liquid from the walls into the bulk solution. You may also rinse down the sides with a very small amount of distilled water from a squirt bottle. Near the end, deliver less than 1 drop of titrant at a time. To do so, carefully suspend a fraction of a drop from the buret tip, touch it to the inside wall of the flask, wash it into the bulk solution, and swirl the solution.
11. Repeat Step 9 and 10 for 2 more samples (four total). If you overshoot any end points, or otherwise do anything you believe caused your titration to be inaccurate, you **MUST** redo the titration. By the end of lab, you need to have three good titrations with numbers that agree reasonably well with one another.

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Analysis

Be sure to use the proper number of sig figs in all your calculations!

1. Set up a table to analyze your data.
2. Calculate the NaOH molarity for each titration. Calculate the uncertainty in your answers (note: the uncertainty of our balances is 0.0002 g and that of our burets is 0.02 mL). Report the NaOH concentrations along with their associated absolute uncertainties.
3. Calculate the average molarity of NaOH. For this calculation, you should use three trials that agree reasonably well. If you do not have three trials that are close to one another, go back and do more!
4. Show your values and the average to the instructor before leaving lab. If your values are too far apart, you may be instructed to repeat the Standardization.
5. Write the concentration of your Standardized Base on the bottle using a permanent marker.

Conclusions

1. Why was it OK to use graduations on the beaker and graduated cylinders in the measurement of volumes in Steps 1 and 2?
2. Why is it not important to exactly measure and know the volume of water used to dissolve the primary standards (Step 5)?
3. Why must the NaOH solution be cooled to room temperature before standardizing it?
4. Consider the concentrations with expected uncertainty and the average molarity you calculated in Analysis questions 2 and 3. Is the average within the uncertainty of the individual concentrations? What does this tell you about the accuracy and/or precision of your answers?

Homework Problems

The following problems from your book must be completed in your lab notebook (see the Syllabus for other suggested problems): Ch 6: **8, 9, 11**; Ch 10: **7, 23**

