

Experiment 3: pH and Solution Preparation

CH2250: Techniques in Laboratory Chemistry, Plymouth State University

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Suggested reading for background information: Sections 1.3-4, 8.1-4, *Exploring Chemical Analysis, 5th Edition*, Daniel C. Harris (2012).

Introduction:

pH is a convenient scale used to measure and report the concentration of the hydronium ion, H_3O^+ , in aqueous solutions, according to the equations:

$$\text{pH} = -\log [\text{H}_3\text{O}^+] \qquad [\text{H}_3\text{O}^+] = 10^{-\text{pH}}$$

where $[\text{H}_3\text{O}^+]$ is the concentration of hydronium ion in moles per liter (M). pH meters are common instruments that can directly measure pH based on the electrical potential across a porous glass membrane. The meter itself technically detects electrical potential (voltage) and converts this to pH. As a result, the meter must be calibrated using standard solutions of known, constant pH, so that the meter knows what pH corresponds to the voltages it reads. In this experiment, you will learn how to calibrate a pH meter, and then you will use the calibrated meter to measure the pH of solutions you prepare.

The preparation of solutions of known concentration is a necessary and vital skill for all chemists. This is a technique you will use over and over in this course and throughout the rest of your chemical career. Therefore it is important to learn early how to do it quickly and accurately.

Solutions can be prepared starting with solids or liquids (including solutions of previously dissolved solids). The final solution is always made in a volumetric flask, though the two routes use different equipment and techniques to measure out the solute. Regardless of the route, two general principles hold:

1. The solute must be *quantitatively transferred* into the flask. This means that 100% of the solute measured must end up in the volumetric flask. For solid solutes, this usually means triple rinsing the weighing vessel with the solvent. For liquid solutes, this usually means using glassware that is calibrated "to deliver" (TD).
2. The final level of the solution in the volumetric flask must be exactly at the "to contain" mark, after the solute is completely dissolved. *The level in the volumetric flask should NEVER be higher than the mark!!* **If you overshoot the mark, throw out the solution and start over.**

In this lab, you will make one solution starting with a solid acid and one solution starting with a solution containing a known concentration of base. You will test your final results using a pH meter.

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 B1 and B9) before coming to lab.

Procedure:

A. General Procedure

1. Boil 250 mL of distilled water for 2 min. Transfer the water to a polyethylene bottle and cap the bottle tightly. Allow the water to cool to room temperature. Use this water in any step below that calls for "Distilled Water." *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.*



B. Acid Solution using Solid Solute

1. In a 25 mL beaker, weigh out a mass of potassium hydrogen iodate ($\text{KH}(\text{IO}_3)_2$) equal to 1.25×10^{-4} mol. Calculate this theoretical mass as part of your pre-lab work. Record the exact mass of solid you weigh. This potassium hydrogen iodate must be quantitatively transferred. In other words, every single molecule of the solid you weighed must make its way into the volumetric flask! The next two steps, if done carefully, will ensure this happens.
2. Add about 5 mL of Distilled Water to the beaker. Dissolve the solid by gently swirling, and transfer this to a 25 mL volumetric flask. Pour the solution gently so that it is not aerated in the process. *If even one drop of this solution is spilled, start over at Step 1.*
3. Add another 5 mL of Distilled Water to the beaker, rinse, and add this to the volumetric flask. Repeat this step once more.
4. Gently swirl the volumetric flask to mix the solution. *Do not aerate the solution.*
5. Using a disposable pipette, add enough Distilled Water to bring the level of liquid *exactly* to the mark in the neck of the flask. *If you overshoot the mark, throw out the entire solution, rinse the flask with distilled water, and start over at Step 1.*
6. Cap the volumetric flask and gently invert it twice to ensure the solution is completely mixed. *After doing this, you may note the level of solution is slightly below the mark. This is normal--part of the solution is stuck in the top of the neck. As long as you mixed thoroughly, this is acceptable. Do NOT add more water to bring it back to the mark!*
7. Pour the solution into a beaker and measure the pH with a previously calibrated pH meter. See part D below for instructions on calibrating the meter. Record the pH in your notebook.
8. Properly dispose of the solution and clean the glassware.
9. Repeat steps 1-8 using a 50 mL volumetric flask and 2.50×10^{-4} mol of $\text{KH}(\text{IO}_3)_2$.

C. Base Solution by Diluting Solution of Known Concentration

1. Obtain about 100 mL of NaOH Stock solution from your instructor.
2. Pour 20-25 mL of the solution into a clean, dry beaker. Measure the pH with a pH meter. Record the pH in your notebook. Dispose of this solution--do not use it in the subsequent steps.
3. Use a 5 mL volumetric pipette to measure 5.0 mL of the Stock solution into a 25 mL volumetric flask.
4. Pour about 15 mL of Distilled Water into a 50 mL beaker. Add this water to the volumetric flask.
5. Using a disposable pipette, add enough Distilled Water to bring the level of liquid *exactly* to the mark in the neck of the flask. *If you overshoot the mark, throw out the entire solution, rinse the flask with distilled water, and start over at Step 1.*
6. Cap the volumetric flask and gently invert it twice to ensure the solution is completely mixed. *After doing this, you may note the level of solution is slightly below the mark. This is normal--part of the solution is stuck in the top of the neck. As long as you mixed thoroughly, this is acceptable. Do NOT add more water to bring it back to the mark!*
7. Pour the solution into a beaker and measure the pH with a previously calibrated pH meter. See part D below for instructions on calibrating the meter. Record the pH in your notebook.



- Properly dispose of the solution and clean the glassware.
- Repeat Steps 3-9 using 10.0 mL of Stock solution, a 10 mL volumetric pipette and a 50 mL volumetric flask. Use 30 mL of Distilled Water in Step 4.

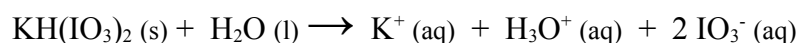
D. Calibrating a pH meter

- The pH electrodes should be soaked in buffer for at least 24 hours prior to use. *When not in use, always store the electrode in buffer.*
- To calibrate the meter, you will need 20-25 mL each of pH 4, 7, and 10 buffer in separate beakers. If you are the first person to calibrate the meter, label three 50 mL beakers as "4," "7," and "10" with a permanent marker and fill them approximately halfway with the appropriate solutions.
- Please note the following:
 - When you are done calibrating the meter, set the solutions aside for the next person to use.
 - Be very careful not to contaminate the buffer solutions. Only a clean, dry pH meter should be inserted into a buffer solution.
 - If you have any doubt whatsoever about whether a buffer solution is pristine, dispose of it down the sink, rinse the beaker a few times with distilled water, and re-fill it with buffer solution.
 - Absolutely do NOT pour used buffer back into the original reagent bottle!
 - Absolutely do NOT insert the pH probe into the original reagent bottle!!
- Remove the pH electrode from its storage solution and thoroughly wash the bulb with distilled water from a wash bottle. *Always rinse the electrode with distilled water before and after inserting it into any solution. Dry the electrode before inserting it into a new sample by gently touching the tip to a Kimwipe.* There should be a large waste beaker near the probe to collect the rinses. When this is full, empty into the sink.
- Calibrate the pH meter using the pH 4 and pH 7 buffer solutions.
- After calibrating the electrode, re-check the pHs of the three buffer solutions. If the pHs do not agree with those of the buffers, re-calibrate the electrode. You may need to calibrate the pH 10 point as well, depending on the meter.

Analysis

Acid Solutions

- Using the exact masses of potassium hydrogen iodate you weighed out, calculate the moles of hydronium ion this yielded in each solution. Note: the ratio of potassium hydrogen iodate to hydronium is 1:1, according to the reaction:



- From the moles of hydronium found in questions 1 and the volumes of the solutions calculate the hydronium concentration in moles per liter (M) you prepared in the two acid solutions.
- From the concentrations of hydronium found in question 2, calculate the pH that theoretically would have resulted from these concentrations.

Base Solutions

- From the pH you measured, calculate the hydronium concentration in the NaOH Stock solution.



2. Divide your answer from question 1 by 5 to calculate the theoretical hydronium concentration resulting from diluting the Stock solution by five (which is what you did!).
3. Using your result from question 2, calculate the theoretical pH that would have resulted from diluting the Stock solution by five.

Conclusions

1. Make a table of the theoretical pHs you calculated in "Analysis" and the experimental pHs you measured in the "Procedure" for the acid and base solutions you made. Calculate the percent error using the equation below, and include these in your table:

$$\% \text{ error} = \frac{|\text{theoretical value} - \text{experimental value}|}{\text{theoretical value}} \times 100$$

2. Comment on what may have resulted in your experimental values differing from the theoretical ones.
3. Why does Step B5 include the instruction "If you overshoot the mark, throw out the entire solution, rinse the flask with distilled water, and start over at Step 1."?
4. Why were you instructed to dispose of the 25 mL of Stock solution used to test pH in Step C2?
5. When CO₂ dissolves in water, it creates a weak acid that can dramatically change the pH of the solution. List two steps or instructions in this lab that were intended to prevent this source of error.

Homework Problems

The following problems from your book must be completed in your lab notebook (see the Syllabus for other suggested problems): Ch 1: 7, 10, 14; Ch 8: 3, 7

