Experiment 5: Preparing and Standardizing an Acid

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, 5th Edition*, Daniel C. Harris (2013).

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 hydorchloric acid and standardize it by titration. This standardized solution will be used to analyze unknown samples in future labs. In a separate lab, you will prepare and standardize a solution of sodium hydroxide.

HCl is standardized by reacting with a known mass of sodium carbonate:

$$2HCl + Na_2CO_3 \rightarrow CO_2 + 2NaCl + H_2O$$

Sodium carbonate (Na_2CO_3) 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, there are two considerations you must keep in mind when using sodium carbonate. First, 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. Second, the reaction above generates CO_2, which dissolves into the solution to generate an acid. The presence of dissolved CO_2 thus interferes with the pH and the detection of the end point of the titration. However, the CO_2 can be driven off by boiling the solution, enabling an accurate titration.

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 1 and 3) before coming to lab. Read through the Procedure and set up appropriate tables to record the data.

Procedure:

1. Use the table inside the cover of the textbook to calculate the volume of ~37 wt% HCl that should be added to 1 L of distilled water to produce 0.1 M HCl. *This calculation should be performed as part of your pre-lab work.*
2. Prepare the HCl solution using the values you calculated, measured with graduated cylinders.
3. Calculate the mass of Na_2CO_3 needed to react with ~25 mL of 0.1 M HCl. *This calculation should be performed as part of your pre-lab work. Note the stoichiometry from the reaction in the Introduction!*
4. Obtain the primary standard Na_2CO_3 from your instructor. *Note*: Sodium carbonate will rapidly absorb water from the air. *If you are not actively weighing it, this primary standard should be stored in a desiccator.*
5. Use a permanent marker to mark four 125 mL flasks as "1," "2," 3," and "4." Measure and record the mass of each as accurately as possible.

6. Weigh four samples of Na_2CO_3 of approximately the mass you calculated in Step 3 directly into the flasks. *It is NOT necessary to weigh exactly the mass you calculated! Just get within 10% of that mass and record exactly the mass you have using all the digits on the balance.*
7. When you are ready to titrate each one, dissolve it in ~25 mL of distilled water and add 3 drops of bromocresol green indicator.
8. Titrate one sample rapidly to a green color to find the approximate end point. *This sample will NOT be used in the final calculation of the concentration of HCl.*
9. Use your result from Step 8 to calculate the approximate volume of HCl required for each of the other three samples.
10. Carefully titrate a sample until it turns from blue to green. During each 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 by careful tilting, and swirl the solution.
11. When you have just reached the endpoint, heat the solution just to boiling to expel CO_2. The color should return to blue. Carefully add HCl from the buret until the solution turns green again and report the volume of acid at this point. *Note: this may only take 1 drop!*
12. Repeat steps 11 and 12 for all samples. If you overshoot any end points, or otherwise do anything you believe caused your titration to be inaccurate, you MUST redo the titration. You need to have three good titrations with numbers that agree reasonably well with one another.
13. Obtain 50 mL of 0.05 M NaCl. Add 3 drops of indicator and titrate this blank using the same procedure as above.

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. Subtract the volume of HCl needed for the blank (Step 11) from the volume of titrant in each titration to obtain the Adjusted Volume of Titrant. *This should be a column in your table.*
3. Use the Adjusted Volumes to calculate the HCl molarity for each titration.
4. Calculate the average molarity of HCl. For this calculation, you should use three trials that agree reasonably well. If you do not have three, go back and do more!
5. 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. Write the value on the bottle of acid.

Conclusions

1. Why was it OK to use graduated cylinders in the measurement of volumes in Step 2?
2. Why is it not important to exactly measure and know the volume of water used to dissolve the primary standards (Step 7)?
3. Why must the titrated solution be boiled before the end point can be found (Step 10)?
4. Why must you titrate a "Blank" of NaCl (Step 11)?

Homework Problems

The following problems from your book must be completed in your lab notebook (see the Syllabus for other suggested problems): Ch 6: **1**, **2**, **4**, **5**; Ch 10: **26**