## **Lab Practical: Acid-Base Titration**

### **Pre-lab Assignment**

1) Potassium hydrogen phthalate (KHP) is a primary standard used to determine the molarity of bases such as NaOH. The equation for this reaction is:

$$C_8H_5O_4K(aq) + NaOH(aq) \rightarrow Na^+(aq) + K^+(aq) + C_8H_4O_4^{2-}(aq) + H_2O(l)$$

Write the net ionic equation for this reaction.

2a) When 0.198 g of KHP is titrated with NaOH, it requires 27.80 mL of NaOH to reach the endpoint of the reaction. Calculate the molarity of the NaOH solution.

b) The standardized solution of NaOH (part a) is used to titrate a solution of HCl. Write the balanced equation for this reaction. Calculate the molarity of the HCl solution if it requires 13.50-mL of NaOH to titrate 15.0-mL HCl solution to the endpoint.

c) Finally, the standardized solution of NaOH (part a) is used to titrate a solution of  $H_2SO_4$ . Write the balanced equation for this reaction. Calculate the molarity of the  $H_2SO_4$  solution if it requires 22.35-mL of NaOH to titrate 15.0-mL  $H_2SO_4$  solution to the endpoint. (Be careful as the stoichiometry is different than in the last problem.)

### **Acid-Base Titration: A Lab Practical**

#### Introduction

In this experiment, you will work with **standardized** solutions. A standardized solution is a solution of known molarity. Some chemicals are very pure and easy to handle. These chemicals, called **primary standards**, can be used directly to make a solution with a very accurate molarity. You will make a standard solution using the primary standard potassium hydrogen phthalate (KHP). Primary standards are used to find the molarity of other solutions, which are then called **secondary standards**. The process of determining the concentration of a solution by titration with a solution of known molarity is called standardization. The concentration unit we use in this lab is molarity.

The technique you will use is titration. In titration you use a solution of known molarity to find the molarity of some other solution. You can do this because the volume times the molarity of the known solution gives you *moles* of that reactant. The stoichiometry of the reaction will give you moles of the second reactant. Molarity is moles solute per liter of solution, so you can calculate molarity if you have measured the volume of the second reactant. These are acid-base reactions, so you should be able to write the equation for the reaction that occurs with each titration. (acid + base  $\rightarrow$  salt + water).

In this experiment you will make solutions of "KHP" (potassium hydrogen phthalate), a primary standard with **molar mass 204.223 g/mol**. KHP has one titratable H. You will use your KHP solution to find the molarity of a stock solution of NaOH. Lastly, you will use the NaOH solution to find the molarity of HCl and of  $H_2SO_4$  solutions. Your final result depends strongly on the intermediate results; hence this is a test of lab techniques you have developed this semester.

The indicator for use in this experiment has been chosen for its sharp endpoint when only a fraction of a drop of excess NaOH has been added to a solution of an acid. The indicator is *phenolphthalein*. It turns from colorless to a faint-pink at the endpoint and goes on to a dark pink or purple color when the endpoint is overshot. The approach to the endpoint is suggested by the temporary appearance of a pink color that fades when the solution is swirled for up to 10 seconds. A pink color that persists for more than 30 seconds signals the actual endpoint.

#### **Procedure**

**NOTE:** All work in this experiment is to be done on an individual basis.

## Part A - Standardization of a solution of sodium hydroxide using a primary standard.

#### Needed:

- Three 125-mL or 250-mL Erlenmeyer flasks—be sure they are clean and dry.
- Three samples of potassium hydrogen phthalate; measure between 0.20 0.25 g into each Erlenmeyer flask. Record the *exact* weight added to each flask. Add about 25 mL of water to each sample. Set the samples aside.
- 300.0-mL of NaOH stock solution..
- A buret for the NaOH

Clean, rinse well (with deionized water), and dry three 125-mL or 250-mL Erlenmeyer flasks. Measure between 0.20 - 0.25 g of potassium hydrogen phthalate into each Erlenmeyer flask. Record

the exact weight added to each flask to the nearest milligram. Add about 25-mL of water to each sample. Set the samples aside.

Find out from your teaching assistant which unknown you will use. Obtain about 500 mL of the stock NaOH solution in a clean, dry beaker. Make a note of its unknown code.

Prepare to titrate the KHP solutions by filling a buret with NaOH solution. Your buret must be clean! Rinse the buret with de-ionized water. Then, pre-rinse it with a portion of the NaOH solution before you fill it up for the titration. Pre-rinsing is necessary to insure that *all* of the solution in the buret is the desired solution, not a diluted or contaminated solution. To do this, add about 10 mL of the NaOH solution to the clean buret. *Carefully* turn the buret on its side so the liquid slowly runs out the top. Rotate the buret on its axis during this time to make sure the solution wets the sides all the way to the top. Pour the rinse from the buret into a waste beaker. Repeat the rinsing process with a second portion of the NaOH solution.

By this time, the KHP samples should all be dissolved. If not, swirl their flasks gently. When you are sure they are fully dissolved, add 2 drops of phenolphthalein indicator to each. The solution should be colorless. Begin to titrate with the NaOH until you reach the endpoint.

The first titration should be performed by adding 0.50 mL portions of the NaOH solution, then swirling the solution. The approach of the endpoint is suggested by the temporary appearance of a pink color that fades when the solution is swirled for up to 10 seconds. A pink color that persists for more than 30 seconds signals the actual endpoint. (Note that the solution may lose its color after this; all you are looking for is persistence for 30-seconds or more.) This first titration gives you a rough idea of the amount of NaOH you need to neutralize the KHP.

Repeat the titration on the second and third samples of KHP. For these you can add about 70% of the needed NaOH quickly and then, near the end, add the NaOH *one drop* at a time until you see the endpoint.

# Part B - Determination of the molarity of a solution of hydrochloric acid using the NaOH secondary standard.

You will need:

- Approximately 125-mL sample of HCl to titrate with your NaOH solution. Make a note of the unknown code of the HCl.
- Four clean 125-mL or 250-mL Erlenmeyer flasks for the HCl
- A 25.00-mL pipet to transfer 25.00 mL of HCl solutions into each of the four flasks
- 2 drops of phenolphthalein indicator added to each HCl sample.

Clean and rinse well (with de-ionized water) three 125-mL or 250-mL flasks. Use a 25.00-mL pipet to transfer 25.00-mL of HCl solutions into each of the three flasks. Add 2 drops of phenolphthalein indicator to each.

Titrate the HCl solutions with the NaOH from the buret. Again, expect that you will overshoot the endpoint on the first run. Make an effort to be much more precise on the second and third trials.

## Part C - Determination of the molarity of a solution of sulfuric acid using the NaOH secondary standard.

You will need:

- Approximately 125-mL sample of H<sub>2</sub>SO<sub>4</sub> to titrate with your NaOH solution. Make a note of the unknown code of the H<sub>2</sub>SO<sub>4</sub>.
- Four clean 125-mL or 250-mL Erlenmeyer flasks for the H<sub>2</sub>SO<sub>4</sub>
- a 25.00-mL pipet to transfer 25.00 mL of H<sub>2</sub>SO<sub>4</sub>solutions into each of the four flasks
- 2 drops of phenolphthalein indicator added to each H<sub>2</sub>SO<sub>4</sub>sample.

Use a clean dry beaker to obtain about 125-mL of your assigned solution from the stock bottle.

Clean and rinse well (with de-ionized water) three 125-mL or 250-mL flasks.

Use a 25.00-mL pipet to transfer 25.00-mL of H<sub>2</sub>SO<sub>4</sub> solutions into each of the three flasks. Add 2 drops of phenolphthalein indicator to each.

Titrate the H<sub>2</sub>SO<sub>4</sub> solutions with the NaOH from the buret. Again, expect that you will overshoot the endpoint on the first run. Make an effort to be much more precise on the second and third trials.

#### Calculations.

**A. Standardization of a solution of sodium hydroxide.** Your calculations begin with the determination of the concentration of sodium hydroxide you used. The mass of the KHP divided by the molar mass of KHP gives the number of moles of KHP. This is equal to the number of moles of NaOH needed to reach the endpoint.

For this part of the experiment, you will be graded on the *precision* and the *accuracy* of your results for the second and third trials. (The first trial is not graded.)

**B and C. Determinations of the Molarity of HCl and H<sub>2</sub>SO<sub>4</sub>.** In these titrations you will know the volume and concentration of NaOH required to reach the endpoint of the titration. With HCl, the number of moles of NaOH used is equal to the number of moles of HCl present in the flask. With H<sub>2</sub>SO<sub>4</sub>, two moles of NaOH are required for each mole of H<sub>2</sub>SO<sub>4</sub>. The number of moles of acid divided by the volume of acid in liters gives the molarity of the acid.

For these parts of the lab, the second and third trials for each acid will be graded for *precision*, since we do not wish to penalize you twice if your calculated concentration of NaOH is inaccurate.

### Report.

Present your results in a table. You must give molarities for your assigned solutions of NaOH and HCl. Indicate the amount of primary standard used in each of the three trials and show sample calculations used (a) to determine the concentration of the secondary standard sodium hydroxide and (b) to determine the concentration of the HCl solution. (c) to determine the concentration of the H<sub>2</sub>SO<sub>4</sub> solution. Report your final results as an average of the second and third titrations in each part.

All calculations must be completed and turned in before you leave the lab today.

## This must be completed and handed in before you leave the lab today.

| *mass of KHP  *moles KHP (204.22 g/mol)  Volume NaOH used  *Molarity of NaOH  Average [NaOH]  Standard Deviation  *Titration of HCl solution with your standardized NaOH (above) Unknown Sample Num HCl + NaOH → H₂O + NaCl  Trial 1 Trial 2 Trial 3  Volume HCl used  Volume NaOH used  *Molarity of HCl  Standard deviation  Titration of H₂SO₄ solution with your standardized NaOH (above)  Unknown Sample Number  Write the balanced equation.  Trial 1 Trial 2 Trial 3  Volume H₂SO₄ used  Volume H₂SO₄ used  Volume NaOH used  *Molarity of H₂SO₄  |   | ution H <sup>+</sup> (from<br>Trial 1 | $\frac{\text{KHP)} + \text{NaOH} \rightarrow \text{H}_2}{\text{Trial 2}}$ | Trial 3 |
|---|---|---------------------------------------|---|---------|
| *moles KHP (204.22 g/mol)  Volume NaOH used  *Molarity of NaOH  Average [NaOH]  Standard Deviation  Titration of HCl solution with your standardized NaOH (above) Unknown Sample Num HCl + NaOH → H <sub>2</sub> O + NaCl  Trial 1 Trial 2 Trial 3  Volume HCl used  Volume NaOH used  *Molarity of HCl  Average molarity of HCl  Standard deviation  Titration of H <sub>2</sub> SO <sub>4</sub> solution with your standardized NaOH (above)  Unknown Sample Number  Write the balanced equation.  Trial 1 Trial 2 Trial 3  Volume H <sub>2</sub> SO <sub>4</sub> used  Volume NaOH used  *Molarity of H <sub>2</sub> SO <sub>4</sub> used  Volume NaOH used  *Molarity of H <sub>2</sub> SO <sub>4</sub> | mass of KHP   | 11101 1                               | 11141 2   |         |
| Volume NaOH used   *Molarity of NaOH   Average [NaOH]   Standard Deviation   Titration of HCl solution with your standardized NaOH (above) Unknown Sample Num HCl + NaOH → H₂O + NaCl   Trial 1 Trial 2 Trial 3   Volume HCl used Volume NaOH used   *Molarity of HCl Average molarity of HCl   Standard deviation Standard deviation    Titration of H₂SO₄ solution with your standardized NaOH (above)  Unknown Sample Number Write the balanced equation.  Trial 1 Trial 2 Trial 3   Volume H₂SO₄ used Volume NaOH used   *Molarity of H₂SO₄ *Molarity of H₂SO₄  |   |                                       |   |         |
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| Average [NaOH]  Standard Deviation  Fitration of HCl solution with your standardized NaOH (above) Unknown Sample Num $HCl + NaOH \rightarrow H_2O + NaCl$ $Trial 1$ $Trial 2$ $Trial 3$ Folume HCl used  Folume NaOH used  Find arity of HCl  Standard deviation  Fitration of $H_2SO_4$ solution with your standardized NaOH (above)  Unknown Sample Number  Write the balanced equation.  Trial 1  Trial 2  Trial 3  Folume $H_2SO_4$ used  Folume NaOH used  |   |                                       |   |         |
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| Titration of HCl solution with your standardized NaOH (above) Unknown Sample Num HCl + NaOH $\rightarrow$ H <sub>2</sub> O + NaCl  Trial 1 Trial 2 Trial 3  Volume HCl used  Volume NaOH used  Molarity of HCl  Standard deviation  Titration of H <sub>2</sub> SO <sub>4</sub> solution with your standardized NaOH (above)  Unknown Sample Number  Write the balanced equation.  Trial 1 Trial 2 Trial 3  Volume H <sub>2</sub> SO <sub>4</sub> used  Volume NaOH used  Volume NaOH used  Volume NaOH used  |   |                                       |   |         |
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|   | Unknown Sample Number   | <u> </u>                              | NaOH (above)  |         |
| *Molarity of H <sub>2</sub> SO <sub>4</sub>   |   |                                       | Trial 2   | Trial 3 |
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| Standard deviation  | Volume NaOH used<br>Molarity of H <sub>2</sub> SO <sub>4</sub>  |                                       |   |         |
|   | Volume NaOH used<br>*Molarity of H <sub>2</sub> SO <sub>4</sub>   |                                       |   |         |
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| Standardization of NaOH: Calculations                          |
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| Calculation for the molarity of H <sub>2</sub> SO <sub>4</sub> |
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