Experiment 2: Reaction Stoichiometry by Thermometric Titration

Introduction

The net result of a reaction (a chemical change) is summarized by a chemical equation. In order to write a chemical equation, a chemist must determine experimentally what the reactants are, in what ratio they react (the reaction stoichiometry), what the products are, and the amount of each product. This experiment is designed to determine the relative amounts of reactants (reaction stoichiometry) for a particular reaction.

When two or more substances are mixed, it is possible to determine whether a reaction occurs by noting whether any property of the mixture changes. Further, by noting how the change in an observed property varies with the ratios in which reactants are mixed, the stoichiometry of the reaction can be determined.

Consider an example in which solutions of A and B are mixed and a precipitate P forms. If a fixed volume of solution A is placed in a beaker, and small increments of solution B are gradually added, the amount of product P will increase. This will continue until the stoichiometric point is reached. The maximum amount of precipitate is formed when A and B are mixed in the correct stoichiometric amounts, when there is just enough of each to react, with nothing left over. Any amount of solution B added to the reaction mixture after this point will not result in the formation of any more product, because no reactant A is left to react. This stoichiometric point can be determined by plotting the amount of precipitate formed vs. the amount of A used in the experiments, as in Figure 2-1.

![Figure 2-1](image)

**Figure 2-1.** Plot of the mass of precipitate P formed as a function of the volume of solution B added to a fixed volume of solution A.

If the concentrations of solutions A and B are known, then the moles of each reactant at the stoichiometric point can be determined.
Technique

The formation of a precipitate is just one of many properties that could be used to determine the stoichiometry of a reaction. In this experiment you will base your decision about the stoichiometric ratio of reactants on the amount of heat that is evolved during the reaction.

One of the reactants you will use is NaOCl (sodium hypochlorite). NaOCl is a strong oxidizing agent present in many commercial bleaches, and will react with a large number of reducing agents in an oxidation-reduction reaction. When NaOCl is reacted, for example, with Na$_2$S$_2$O$_3$ (sodium thiosulfate), the reaction is

$$x \text{NaOCl} + y \text{Na}_2\text{S}_2\text{O}_3 \rightarrow \text{products} + q$$  \hspace{1cm} (2-1)

The coefficients in the balanced equation are $x$ and $y$, and $q$ stands for the heat that is evolved. In this experiment, you will place a measured amount of a reducing agent in a beaker and add an NaOCl solution of known concentration dropwise to the beaker until the reducing agent has been completely consumed. By monitoring the temperature of the reaction, you will be able to determine the stoichiometric point--the point where the reaction no longer occurs and heat is no longer evolved. The technique of adding a solution of known concentration to another solution dropwise until an endpoint is reached is called a titration. Visualizing the endpoint by monitoring the reaction temperature makes this a thermometric titration. In this experiment, the endpoint of the titration occurs at the stoichiometric point, although we will continue adding the NaOCl solution beyond the stoichiometric point.

The MeasureNet workstation will be equipped with a drop counter and a temperature probe. By counting the number of drops of NaOCl solution added, and measuring the total volume of NaOCl used in the titration, the average volume of each drop can determined. This information can be used to make a plot of temperature vs. volume of NaOCl for the reaction. You will generate one plot for each of four reactions involving NaOCl and four different reducing agents. By determining the volume of NaOCl required to reach the stoichiometric point of the titration, you will be able to calculate the number of moles of each reactant used at the stoichiometric point and thereby determine the coefficients $x$ and $y$ for the two reactants as seen in Eq. 2-1.

Equipment Needed

- 5-mL volumetric pipet
- pipet pump
- stir plate
- buret clamp
- temperature probe
- beakers
- magnetic stir bar
- 50 mL buret
- ring stand
- drop counter

Chemicals Needed

**Reducing agents:**

- 0.20 M Na$_2$SO$_3$ in 0.04 M NaOH; sodium sulfite in sodium hydroxide solution
- 0.20 M Na$_2$S$_2$O$_3$ in 0.44 M NaOH; sodium thiosulfate in sodium hydroxide solution
- 0.20 M KNCS in 0.44 M NaOH; potassium thiocyanate in sodium hydroxide solution
- 0.20 M KI in 0.04 M NaOH; potassium iodide in sodium hydroxide solution

**Oxidizing agent:**

- standardized NaOCl solution; sodium hypochlorite
Procedure

Note: As is usually the case when using MeasureNet, you will work in pairs on this experiment.

Obtain ~75 mL of the NaOCl solution; be sure to record the labeled concentration. Obtain ~10 mL of one of the four reducing agents.

1. Setting up the workstation

   Press MAIN MENU on the MeasureNet workstation. Select TEMPERATURE from the menu, then TEMP VOLUME from the menu that appears next. Press the SETUP button on the workstation, then choose SET LIMITS FOR NEW ACQUISITION. This will allow you to set the temperature and time limits used for the graphical display on the workstation for the experiment. Follow the instructions on the display to set the temperature limits at 40°C maximum and 20°C minimum (y-axis) and the maximum number of drops at 900 drops (x-axis). Leave the minimum setting at 0. Press the DISPLAY button—you do not need to calibrate the temperature probe since, we are interested only in changes in temperature, not the actual value.

2. Setting up the Apparatus

   Using a volumetric pipet, deliver 5.0 mL of the reducing agent to a 50 mL beaker. Place a magnetic stir bar in the beaker, and place the beaker on a stir plate.

   Rinse the inside of a buret with a small amount of the NaOCl solution, then open the stopcock and allow the solution to drain into a 400 mL waste beaker. Fill the buret close to the 0 mL mark with the NaOCl solution. Clamp the buret to a buret clamp attached to a ring stand, and drain a small amount of the NaOCl solution into the waste beaker until the tip of the buret is completely filled with solution.

   Connect the drop counter to the MeasureNet workstation, and clamp it to a second ring stand. Position the drop counter over the beaker containing the reducing agent so that the notch on the side of the drop counter is over the solution. Make sure the temperature probe is connected to the MeasureNet workstation, then insert the probe into to small hole in the center of the drop counter. Position the probe so that it is slightly above the bottom of the beaker, then tighten the white thumbscrew to hold it in that position.

   Position the buret so that the tip is slightly above and centered over the notched portion of the drop counter, making sure that it is also over the beaker.

3. Measuring Temperature Change

   When everything is arranged over the beaker, begin the titration by pressing START/STOP on the workstation. The station will first ask you to enter the starting reading on the buret. It need not be exactly 0.00 mL. Just read what it is and enter the appropriate value, to the nearest 0.01 mL, and then press ENTER.

   Press START/STOP again--the station display will now show the temperature and the number of drops counted (0 so far). Carefully turn the stopcock on the buret to begin adding the NaOCl solution. The red LED on the drop counter will flash each time a drop is counted; if the light does flash, stop the titration and adjust the position of the buret tip so that it is centered over the notch. Try to maintain a rate of approximately 1 drop per second. While the titration is running, monitor the drip rate and make small adjustments of the stopcock if needed.
Continue adding the NaOCl solution dropwise until you observe a significant change in the slope of the temperature curve on the workstation screen. Allow the titration to continue for about 10 seconds after this point, then simultaneously close the stopcock and press **START/STOP**. The station will ask you to enter the final buret reading; again determine this value to the nearest 0.01 mL.

Press **FILE OPTIONS**, then the function key for **PRINT STANDARD**.

Pour the reaction solution into the waste beaker, rinse the 50 mL beaker with distilled water and dry the beaker. Obtain ~10 of another of the reducing agents and repeat the steps above. Follow the same procedure with the other reducing agents, refilling the buret with NaOCl solution only when the buret becomes less than ½ full.

At the end of the experiment, combine all leftover reagents in the waste beaker, and pour this waste down the sink.

**Results**

For each titration:

1. The endpoint is visualized as the point on the titration curve where the slope of the curve changes drastically. Determine, to the nearest 0.1 mL, the volume of NaOCl solution required to reach the endpoint of the titration.

2. Recall that the endpoint represents the stoichiometric point of the titration. Calculate the number of moles of NaOCl and reducing agent that reacted with each other at the stoichiometric point.

3. Calculate the mole ratio, that is, the number of moles of NaOCl per mole of reducing agent.

4. Round the mole ratio to the nearest whole number, and determine the coefficients $x$ and $y$ for the chemical equation.

**Questions**

1. Why does the temperature of the reaction mixture drop (as opposed to remaining constant) once the reaction reaches the stoichiometric point?

2. For one of the reactions, a colored product temporarily appeared. What do you think this product was?
**Experiment 2**  
**Report Sheet**

Name: ________________________________ Date: ____________

**Data**  
Concentration of NaOCl solution ______________________

<table>
<thead>
<tr>
<th>Reducing agent</th>
<th>Concentration of Reducing Agent</th>
<th>Volume of Reducing Agent</th>
<th>Volume of NaOCl at Stoichiometric Point</th>
</tr>
</thead>
<tbody>
<tr>
<td>KI</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Na$_2$S$_2$O$_3$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Na$_2$SO$_3$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>KNCS</td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>

\[
x \text{NaOCl} + y \text{KI} \rightarrow \text{products} + q
\]

Moles of NaOCl reacted ____________________________

Moles of KI reacted ____________________________

Mole ratio, NaOCl /KI ____________________________

Simplest whole number mole ratio, NaOCl /KI ____________________________

\[
x \text{NaOCl} + y \text{Na}_2\text{S}_2\text{O}_3 \rightarrow \text{products} + q
\]

Moles of NaOCl reacted ____________________________

Moles of Na$_2$S$_2$O$_3$ reacted ____________________________

Mole ratio, NaOCl / Na$_2$S$_2$O$_3$ ____________________________

Simplest whole number mole ratio, NaOCl / Na$_2$S$_2$O$_3$ ____________________________
\[ x \text{NaOCl} + y \text{Na}_2\text{SO}_3 \rightarrow \text{products} + q \]

Moles of NaOCl reacted

Moles of Na\(_2\)SO\(_3\) reacted

Mole ratio, NaOCl / Na\(_2\)SO\(_3\)

Simplest whole number mole ratio, NaOCl / Na\(_2\)SO\(_3\)

\[ x \text{NaOCl} + y \text{KNCS} \rightarrow \text{products} + q \]

Moles of NaOCl reacted

Moles of KNCS reacted

Mole ratio, NaOCl / KNCS

Simplest whole number mole ratio, NaOCl / KNCS