

# 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.

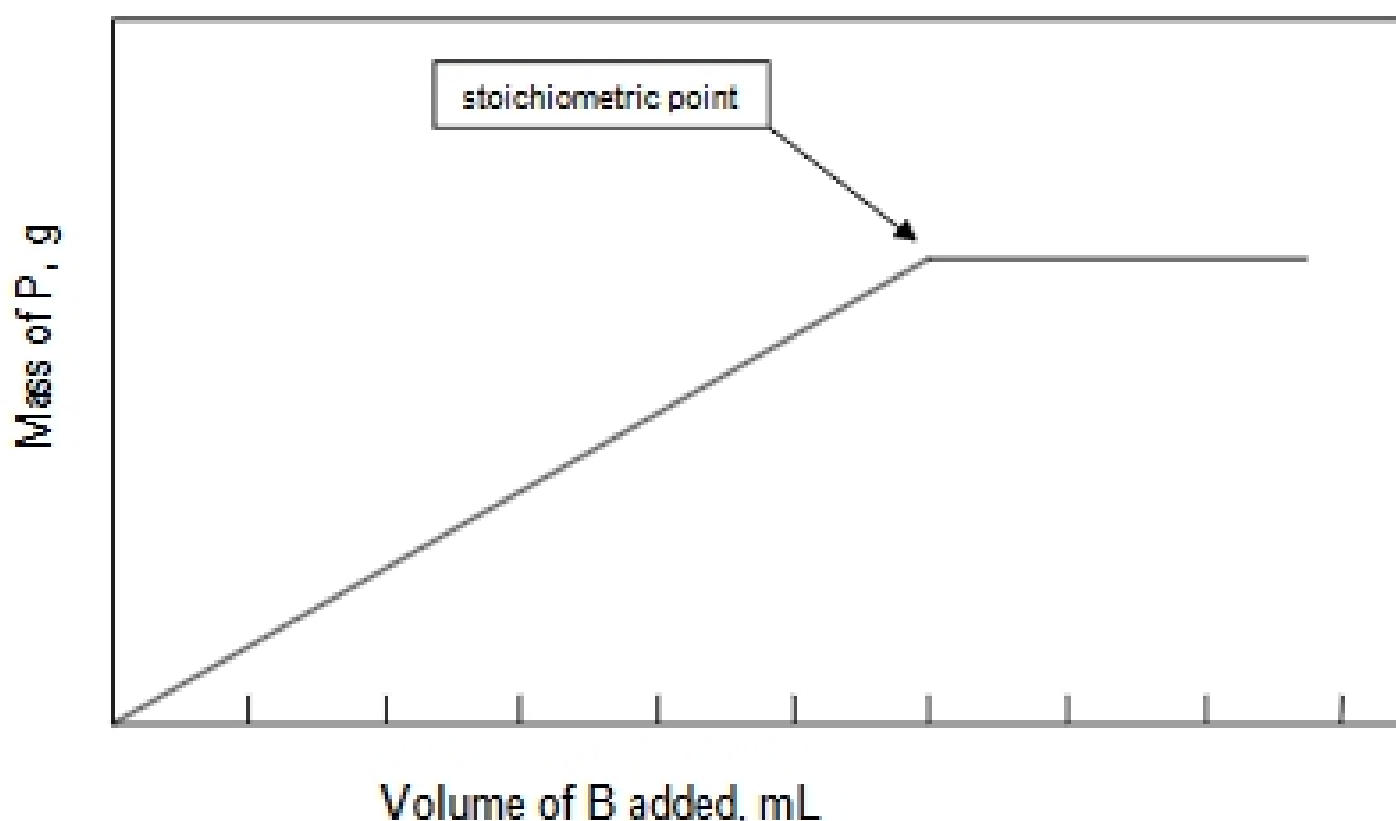


Figure 2-1. Plot of the mass of precipitate P formed as a function of the volume of solution B added to a fixed volume of solution A.

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<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (sodium thiosulfate), the reaction is



The coefficients in the balanced equation are  $x$  and  $y$ , and  $q$  stands for the heat that is evolved. Since the objective is to determine the ratio of the coefficients  $x$  and  $y$ , it is not important that we know the chemical identity of the products. Also note that hydroxide ion is required as a reactant; it is added in excess to the solution containing the reducing agent to ensure that the reaction goes to completion.

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. Once the stoichiometric point is reached, the temperature of the solution will begin to decrease; not only is heat no longer being generated, but the temperature of the titrant added past the endpoint is at a lower (room) temperature than the reaction solution.

The LabQuest workstation will be equipped with a drop counter and a temperature probe. By first calibrating the buret to determine the average number of drops per mL being delivered, a plot of temperature vs. volume of NaOCl for each reaction can be obtained. 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  
drop counter

beakers  
magnetic stir bar  
50 mL buret  
ring stand  
LabQuest

## CHEMICALS NEEDED

### reducing agents:

0.20 M  $\text{Na}_2\text{SO}_3$  in 0.04 M NaOH; sodium sulfite in sodium hydroxide solution

0.20 M  $\text{Na}_2\text{S}_2\text{O}_3$  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

**Note:** Hydroxide ion is added in excess to each solution to ensure reaction completion

### oxidizing agent:

standardized NaOCl solution; sodium hypochlorite (concentration is given on the container)

## PROCEDURE

Obtain ~60 mL of the NaOCl solution; be sure to record the labeled concentration. Obtain ~10 mL of one of the four reducing agents (note: whenever you obtain a solution used by all students, you obtain an approximate amount. You will measure out 5.00mL of this solution more precisely with a volumetric pipet once you return to your workstation).

### *Setting up the workstation and calibrating drops/mL of the buret*

**Note:** If the workstation is displaying a plot of data from a previous user's experiment, select **New** from the **File** menu and touch **Discard**.

1. The temperature probe should already be plugged into a side port on the workstation, and a temperature (the temperature of the lab room) displayed on the screen. If the drop counter has not yet been plugged in, do so using the DIG1 port at the back of the station.
2. Attach the drop counter to a ring stand. Position the drop counter above a 50 mL beaker so that the beaker is directly below the notch in the drop counter (this is where the drop detector is located).
3. Clamp a buret to the ringstand with a buret clamp and position the tip of the buret directly above the notch in the drop counter so that the drops will be collected in the beaker. Fill the buret close to the 0.0mL mark with tap water and allow some water to drain out the tip so that tip is filled with water. Record the initial volume to the nearest 0.01 mL.
4. Touch the **Volume** box on the workstation and select **Calibrate**. Ignore instructions 1 and 2 on the workstation screen; we will be using the volume readings from the buret rather than a graduated cylinder to ensure greater precision.
5. Touch the **Calibrate Now** box. Carefully open the buret stopcock so that the drip rate is ~1 drop per second. Make sure the drops are being detected by the drop counter (the red LED light on the drop counter will flash when a drop is detected) and that the drops are being counted in the upper right corner of the workstation.
6. When approximately 10 mL of water has been drained from the buret, close the stopcock. Record the final volume and determine the precise volume of water drained from the buret. Touch the box on the workstation after **Enter the precise volume** and enter this volume. Touch **Equation** to see the calculated drops/mL. Record this value.
7. Touch **Calibrate** and then **Redo Calibration**, and repeat the calibration steps. Continue repeating the calibration until you have two drops/mL values that agree within 0.5 drops/mL. Touch **OK**.