

# Protocol | Stoichiometric Characterization

## Stoichiometric Characterization of Compounds and Mixtures

### Introduction

Watch the [introduction and theory video](#) for this experiment.

*Stoichiometry* is the study of the mass ratios in which substances combine in compounds and chemical reactions. An understanding of stoichiometry is vital for the efficient planning of chemical reactions and the elucidation of the empirical formulas of compounds. In this experiment, we will apply stoichiometry to plan and execute chemical reactions, determine the empirical formula of a substance whose composition can vary, and determine the relative amounts of substances in a mixture.

To accomplish all of these goals, a balanced chemical equation for a chemical reaction of interest is essential. Balanced chemical equations are the “meat” of stoichiometry: they provide us with a wealth of information about the numbers and types of molecules that combine in reactions. Using the *molar ratios* implied in balanced chemical equations, we can deduce an unknown amount of a substance that has reacted from a known amount of a different substance. To complete this experiment, you should also be familiar with the concepts of amount of substance and the *mole*, *molar mass* (g/mol) and solution *molarity* (mol/L). In the laboratory, we measure solids using mass and solutions using volume. However, amount of substance in moles is really the key quantity for stoichiometry. Molar mass and molarity act as “bridges” from the macroscopic realm of the laboratory to the microscopic realm of chemical reactions. That is, if we know the amounts of substances (in moles) that combine with one another in a chemical reaction, we can use molar mass or molarity to decide how much of each substance to use in a practical laboratory setting. We can also work in reverse, from measured masses or solution volumes to numbers of moles.

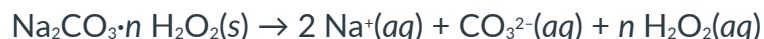
The *law of conservation of mass* will also be important for this experiment. This law states that matter is neither created nor destroyed; physical and chemical changes only alter its form. Conservation of mass guarantees that when examining a compound made of multiple elements, we can consume part of the compound through a chemical reaction without altering the mass of the rest of the compound. Knowing the molar masses of the part that reacted and the part that remained unchanged, we can calculate the molar ratios of the two parts within the compound, and thereby determine its empirical formula.

For example, this strategy can be applied to determine the number of waters of hydration in a hydrated metal salt  $\text{MX} \cdot n \text{H}_2\text{O}$ . Strong heating liberates the water ( $\text{H}_2\text{O}$ ), whose mass we can determine using the difference between the masses before and after heating. What remains is only the metal salt (MX), so the mass after heating belongs to the pure metal salt. If the formula of MX is known, we can use molar mass to calculate the amounts of MX and  $\text{H}_2\text{O}$ . The ratio of these amounts tells us  $n$ , the number of water molecules per hydrated metal salt molecule.

A similar procedure can be applied to determine the amounts of different substances within a mixture. If one of the substances reacts and the other does not under a particular set of reaction conditions, stoichiometry of the reaction that does occur can be used to determine the amount of the substance that does react.

Sodium percarbonate (SPC) is the active ingredient in several very strong cleaning products, including OxiClean. It consists of sodium carbonate molecules to which some number of hydrogen peroxide molecules have complexed, resulting in an empirical formula of  $\text{Na}_2\text{CO}_3 \cdot n \text{H}_2\text{O}_2$ , where  $n$  is generally a number between 1 and 2. The cleaning power of this reagent is based on the strong oxidizing power of the hydrogen peroxide molecules, which are liberated when SPC is dissolved in water. Thus, the value of  $n$  is a measure of the “strength” of a sample of SPC.

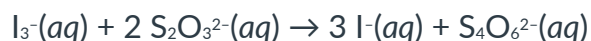
In Part A of this experiment, we will determine the value of  $n$  for a commercial sample of sodium percarbonate. To do this, we will dissolve a known mass of the sample in water and perform a reaction in which the hydrogen peroxide reacts selectively.



Aqueous hydrogen peroxide reacts with the iodide ion ( $\text{I}^-$ ) in acidic solution to form the aqueous triiodide ion ( $\text{I}_3^-$ ). Iodide does not react with sodium carbonate, so the amount of triiodide in solution is a direct indicator of the amount of hydrogen peroxide.

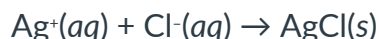


To determine the amount of  $\text{I}_3^-$  in solution, we will add to it a 0.10 M solution of sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) with known concentration *just* until all of the triiodide has been consumed. The thiosulfate anion ( $\text{S}_2\text{O}_3^{2-}$ ) reacts with triiodide ion to form iodide ion and  $\text{S}_4\text{O}_6^{2-}$  according to the balanced chemical equation below. Stoichiometry can then be used to work backwards from the volume of  $\text{Na}_2\text{S}_2\text{O}_3$  solution delivered to the moles of hydrogen peroxide originally dissolved, all of which came from the solid SPC sample. Because the amount of iodine is used to determine a different number of moles, this method is called *iodometry*.



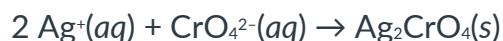
Determining the amount of an ion in a solid or aqueous sample is a very common problem in analytical chemistry. In part B of this experiment, we will apply stoichiometry to determine the composition of a solid mixture that contains sodium chloride ( $\text{NaCl}$ ) and

sodium nitrate ( $\text{NaNO}_3$ ). To do this, we will take advantage of the selective reactivity of the chloride anion with the silver cation to form a solid precipitate of silver chloride.



The sodium cation and nitrate anion are spectator ions in this process. Thus, addition of a known amount of silver cation can tell us the amount of chloride anion in a solution of the solid  $\text{NaCl}/\text{NaNO}_3$  mixture, and therefore the amount of  $\text{NaCl}$  in the original solid mixture.

Silver chloride is a very fine precipitate that forms a cloudy suspension in water. As a result, it is difficult to tell when *just* the right amount of silver has been added to completely precipitate chloride. To get around this issue, we will make use of potassium chromate ( $\text{K}_2\text{CrO}_4$ ) as an indicator. Silver combines with the chromate anion to form a red-orange precipitate of silver chromate ( $\text{Ag}_2\text{CrO}_4$ ).



As we add silver cation to a solution of sodium chloride, sodium nitrate, and a small amount of potassium chromate, silver chloride will precipitate first because it is less soluble in water than silver chromate. Red-orange silver chromate will start to precipitate when very little chloride remains in solution; we will assume that  $[\text{Cl}^-] = 0$  at this point. Thus, at the moment we observe a *persistent* red-orange precipitate, we can conclude that we've added just enough silver cation to consume all of the chloride anion in the original solution.

This method for the determination of chloride is called the [Mohr method](#). Check out [this video \(5:55\)](#) to see this method in action.

## Safety and Materials

The following reagents will be available:

- [1.0 M sulfuric acid \( \$\text{H}\_2\text{SO}\_4\$ \) solution \(Links to an external site.\)](#)
- [Potassium iodide \(KI\) solid \(Links to an external site.\)](#)
- [Sodium percarbonate \( \$\text{Na}\_2\text{CO}\_3 \cdot n \text{H}\_2\text{O}\_2\$ \) solid \(Links to an external site.\)](#)
- [1.70% w/w silver nitrate \( \$\text{AgNO}\_3\$ \) solution \(Links to an external site.\)](#)
- [Sodium chloride \( \$\text{NaCl}\$ \) \(Links to an external site.\)/sodium nitrate \( \$\text{NaNO}\_3\$ \) \(Links to an external site.\) solid mixture](#)
- [5% w/w potassium chromate \( \$\text{K}\_2\text{CrO}\_4\$ \) solution \(Links to an external site.\)](#)

Discard all chemical waste in the waste bottles under the hood, including excessed unused reagents.

## Relevant Experimental Techniques

- [Weighing Solids](#)
- [Pipetting Liquids](#)
- [Titration](#)

## Research Questions

1. How many moles of hydrogen peroxide per mole of sodium carbonate are present in a sodium percarbonate sample?
2. What is the molar composition of an unknown mixture of sodium chloride and sodium nitrate?

## Procedures

### A. Characterization of Sodium Percarbonate by Iodometry

1. Prepare a table in your lab notebook with entries for the mass of sodium percarbonate (in grams) and the volume of sodium thiosulfate solution (in milliliters). Prepare a second table for the results of calculations with the following entries. This table does not need to be filled in during lab, but will be helpful later.

Quantity
Amount of $\text{S}_2\text{O}_3^{2-}$ Delivered (moles)
Amount of $\text{H}_2\text{O}_2$ Consumed (moles)
Mass of $\text{H}_2\text{O}_2$ in Sample (grams)
Mass of $\text{Na}_2\text{CO}_3$ in Sample (grams)
Amount of $\text{Na}_2\text{CO}_3$ in Sample (moles)
Molar Ratio of $\text{H}_2\text{O}_2:\text{Na}_2\text{CO}_3$ in Sample

2. *Prepare an acidified solution of potassium iodide and add a precisely known mass of sodium percarbonate (SPC) to it.* To a 50 mL Erlenmeyer flask, add approximately 20 mL of **1.0 M sulfuric acid solution** (the exact amount is not critical).

3. Weigh out approximately 1 gram of **potassium iodide** and dissolve it completely in the sulfuric acid solution. This acidified iodide solution will react with hydrogen peroxide supplied by sodium percarbonate.
4. *Weigh out a precisely known mass of sodium percarbonate.* Carefully, with as much accuracy as possible, weigh out approximately 25 mg (0.025 g) of **sodium percarbonate**. Record the exact mass in your lab notebook.
5. Add the sodium percarbonate to your acidified iodide solution along with 6 drops of **starch solution**. Record your observations; the mixture should darken as the sodium percarbonate dissolves.
6. *Construct an apparatus for titration.* Obtain a buret with stirrer stand and buret clamp. Mount the buret in the stand. Obtain about 50 mL of **0.10 M sodium thiosulfate solution** in a small beaker.
7. Wash the buret with the sodium thiosulfate solution. To do this, ensure the stopcock at the end of the buret is closed, then move the stand to the edge of the bench and lower the buret so that you can fill at eye level. Add a buret funnel and fill the buret with about 10 mL of sodium thiosulfate solution. Drain this liquid into a waste beaker. While doing this, practice opening and closing the stopcock carefully to achieve dropwise flow of the solution.
8. Once the liquid has drained completely, close the stopcock. Fill up the buret with the remainder (about 40 mL) of the sodium thiosulfate solution and return it to the stand. Record the initial volume of sodium thiosulfate solution in the buret.
9. *Prepare the analyte solution for titration.* Place the Erlenmeyer flask under the nozzle of the buret.
10. *Titrate the analyte solution to a colorless endpoint, using starch as an indicator.* Carefully open the stopcock slightly to deliver thiosulfate dropwise into the Erlenmeyer flask with gentle swirling of the flask. Do **not** allow the liquid level to fall below the bottom line!
11. Continue adding thiosulfate with swirling until the solution becomes colorless. Record the final volume of thiosulfate solution in the buret and calculate the volume of solution delivered.
12. Discard any remaining sodium thiosulfate solution, the contents of the Erlenmeyer flask, and any other excess reagents from this part of the experiment down the sink drain with water.

## B. Determining the Composition of a Mixture of Sodium Chloride and Sodium Nitrate

1. *Prepare a solution of sodium chloride + sodium nitrate and indicator.* Obtain a dropper bottle of **potassium chromate (indicator) solution**. To a clean and dry 125 mL Erlenmeyer flask, add about 40 mL of water and 8 drops of indicator solution.

2. Weigh out approximately 0.05 grams of **sodium chloride + sodium nitrate solid** and record its exact mass. Dissolve the solid in the solution from step 1, stirring with a stirring rod to ensure complete dissolution.
3. Determine and record the total mass of the Erlenmeyer flask and solution, noting the balance that you use to weigh. Rather than measuring the volume of silver nitrate solution added as in Part A, we will measure the mass of titrant added and make use of the weight-percentage concentration of silver nitrate in calculations.
4. *Prepare the silver nitrate solution.* Obtain about 15 mL of **1.70% w/w silver nitrate solution** in a 50 mL beaker. Designate a Pasteur pipet and [Pasteur pipet bulb](#) to use for the addition of silver nitrate solution to the Erlenmeyer flask.
5. *Titrate the chloride solution with silver nitrate to a persistent orange endpoint.* Add about one pipet's worth of silver nitrate solution to the Erlenmeyer flask and gently swirl. You should observe a chemical change; record your observations.
6. Continue to add silver nitrate solution *dropwise* via Pasteur pipet to the Erlenmeyer flask. Every few drops, swirl the flask until its color stabilizes.
7. Stop adding silver nitrate solution immediately when you observe a persistent orange color in the flask.
8. *Measure the mass of silver nitrate solution added.* Reweigh the flask and solution using the same balance you used in step 3. Record the final mass and determine the mass of silver nitrate solution delivered into the flask.
9. Discard any excess silver nitrate solution and the contents of the Erlenmeyer flask in the waste bottle in the hood. Use your wash bottle and some water to rinse the precipitate out of the flask and into the waste bottle.

## Group Argumentation

Merge data with the other students at your lab bench. Assign one part for initial analysis to each set of partners. Discuss the data calculations necessary and obtain a value of moles for the component of interest stated in the research question. Discuss and write an argument for each research question in lab notebooks with the other students at the bench. Include an answer to the question, evidence from your experimental results, and an explanation of your reasoning based on the mathematics involved. If there is time, the arguments can be discussed with other groups.

## Post-lab Calculations and Data Workup

| [SpreadsheetDownload Spreadsheet](#)

Use your Part A data to determine the value of  $n$  in  $\text{Na}_2\text{CO}_3 \cdot n \text{H}_2\text{O}$  for your sample of sodium percarbonate. Complete the table in your lab notebook to tackle this problem systematically.

Use your Part B data to calculate the moles of chloride anion present in the original solid sample used in the titration (approximately 0.05 g, the total mass of the mixture).

Determine the moles and mass of sodium chloride in the original sample. Use the mass to calculate the weight percent of sodium chloride in the mixture. Finally, determine the mass percentages of the *elements* sodium, chlorine, nitrogen, and oxygen in the mixture and create a pie chart in Excel that illustrates these percentages.