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Green Chemistry Analysis of a Mixture

AP* Chemistry Big Idea 3, Investigation 7

An Advanced Inquiry Lab

Introduction

The *Green Chemistry Program* was initiated by the Environmental Protection Agency in the 1990s with the goal of applying chemical principles to prevent pollution. The program calls for the design of chemical products and processes that will reduce the use and generation of hazardous substances. The purpose of this lab is to design an experiment for determining the percent composition of a solid by applying the principles of green chemistry.

Concepts

- Stoichiometry
- Percent composition
- Green chemistry
- Decomposition reaction

Background

Much of what makes this world modern is the result of the application of chemistry and chemical reactions. Oil and gasoline, prescription drugs, plastics materials, solvents, and fertilizers, to name a few, are all products of chemistry.

Over time, many of the processes used to create these products were found to be quite harmful, whether to workers, the consumers, or to the environment. In response to these pressing issues, various professional groups have created a different approach to the research and production of chemicals and chemical processes called *Green Chemistry*.

The Green Chemistry approach uses twelve principles that help evaluate the production and use of chemical products so that the use and generation of hazardous substances can be reduced or eliminated and, where possible, renewable starting materials can be used. These principles are listed below.

1. **Prevention**

   It is better to prevent waste than to treat or clean up waste after it has been created.

2. **Atom Economy**

   Synthetic methods should be designed to maximize the incorporation of all materials used in the process into the final product, leaving few or no atoms behind.

3. **Less Hazardous Chemical Syntheses**

   Synthetic methods should be designed to use and generate substances that possess little or no toxicity to human health and the environment.

4. **Designing Safer Chemicals**

   Chemical products should be designed to be fully effective while minimizing or eliminating their toxicity.

5. **Safer Solvents and Auxiliaries**

   Minimize the use of auxiliary substances (e.g., solvents, separation agents, etc.) wherever possible and make them innocuous when used.

6. **Design for Energy Efficiency**

   Energy requirements of chemical processes should be recognized for their environmental and economic impacts and should be minimized. If possible, synthetic methods should be conducted at ambient temperature and pressure.

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7. **Use of Renewable Feedstocks**

   Renewable raw material or feedstock should be used whenever technically and economically possible.

8. **Reduce Derivatives**

   Unnecessary derivatization (use of blocking groups, protection/deprotection, temporary modification of physical/chemical processes) should be minimized or avoided if possible, because such steps require additional reagents and can generate additional waste.

9. **Catalysis**

   Catalytic reagents are superior to stoichiometric reagents.

10. **Design for Degradation**

    Chemical products should be designed so that at the end of their function they break down into innocuous products that do not persist in the environment.

11. **Real-time Analysis for Pollution Prevention**

    Analytical methodologies need to be further developed to allow for real-time, in-process monitoring and control prior to the formation of hazardous substances.

12. **Inherently Safer Chemistry for Accident Prevention**

    Substances and the form of a substance used in a chemical process should be chosen to minimize the potential for chemical accidents, including releases, explosions, and fires.

In this lab, you will design a process to determine the weight percent of a metal bicarbonate, either sodium bicarbonate or potassium bicarbonate, in a mixture of itself and its carbonate counterpart. Sodium and potassium bicarbonates undergo decomposition when heated above 110 °C.

$$2MHCO_3(s) \rightarrow M_2CO_3(s) + H_2O(g) + CO_2(g) \qquad \textit{Equation 1}$$

At temperatures below 800 °C, potassium and sodium carbonate remain unreacted. Therefore, if a mixture of bicarbonate and carbonate salts is heated at low temperature, all that remains after heating is the carbonate solid.

You will use three of the twelve principles of green chemistry to evaluate the "greenness" of a current lab procedure used in some high school labs to teach stoichiometry. The three principles you will look at in this evaluation are: *prevention, atom economy,* and *use and production of nontoxic materials*.

Prevention:

It is better to prevent waste than to treat or clean up the waste after it has been created. In designing a lab activity, evaluate alternative reactions wherever possible, and identify the reaction that produces the least waste.

Atom Economy:

When choosing among various reactions, identify which reaction produces the least amount of by-product waste. A typical reaction can be represented by the following equation.

$$\text{Reactants} \rightarrow \text{Desired product} + \text{By-product waste}$$

The greater the ratio of desired product to reactants, the greener the reaction. Atom economy can be calculated as a percentage of the mass of the desired product to the mass of all reactants. The higher this percentage, the greener the process.

Let's look at an example of a product that can be produced by two different reactions. Aluminum can be oxidized by water to aluminum oxide (Equation 2). If solid aluminum hydroxide is strongly heated, aluminum oxide and water are produced.

$$2Al(s) + 3H_2O(l) \rightarrow Al_2O_3(s) + 3H_2(g) \qquad \textit{Equation 2}$$

$$2Al(OH)_3(s) \rightarrow Al_2O_3(s) + 3H_2O(g) \qquad \textit{Equation 3}$$

The atom economy for Equation 2 is equal to:

$$\frac{(\text{Mass of 1 mole } Al_2O_3)}{(\text{Mass of 2 moles Al } + \text{ mass 3 moles } H_2O)} \times 100 = \frac{101.96}{[(2)(26.98) + 3(18)]} \times 100 = 94.4\%$$

The atom economy for Equation 3 is equal to:

$$\frac{(\text{Mass of 1 mole } Al_2O_3)}{(\text{Mass of 2 moles } Al(OH)_3(s))} \times 100 = \frac{101.96}{(2)(78.00)} \times 100 = 65.4\%$$

Of the two reactions, the first reaction is the greenest in terms of atom economy. Remember, atom economy does not address the toxicity or hazards of either the reactants or the by-products.

Use and Production of Nontoxic Materials—Less Hazardous Chemical Syntheses:

When possible, choose chemicals that have the least toxic effect on humans and the environment. Check the toxicity of all the chemicals used in the production of the desired products, including the products themselves. Chemists can use this toxicity information when making their selection decisions.

Experimental Overview

The purpose of this advanced inquiry lab is to design and carry out a green chemistry experiment that can quantitatively measure the weight percent of one compound in a mixture of two compounds. The investigation begins with an introductory activity to verify the decomposition reaction of a solid bicarbonate, either potassium or sodium bicarbonate. These solids undergo the reaction outlined in the *Background* section.

Stoichiometry is defined as the quantitative relationship among constituents in a chemical reaction. Based on the bicarbonate balanced chemical equation and its stoichiometry, you will calculate your atom economy using the experimental data and compare this to the theoretical value. The results provide a model for the guided-inquiry design of an experiment that can quantitatively measure the weight percent of either a sodium carbonate/bicarbonate solid mixture or a potassium carbonate/bicarbonate mixture. You will assess your procedure in terms of the three green principles and then compare this assessment to that of the procedure examined in the *Pre-Lab Questions*.

Pre-Lab Questions

Carefully read the Stoichiometry Laboratory Procedure on the determination of a chemical reaction, and then answer the following questions. Use reference books and the Internet when needed. In the experiment, the silver chromate that is produced forms a dense, colorful precipitate that is easy to see and accurately measure.

1. The products of this lab are silver chromate solid, $AgCrO_4$, and a solution of potassium nitrate.
   a. Is either or both of these products hazardous? If so, in what way?
   b. What are the proper disposal methods for each of these products?
2. The balanced equation for the experimental reaction is listed below.

   $$2AgNO_3(aq) + K_2CrO_4(aq) \rightarrow Ag_2CrO_4(s) + 2KNO_3(aq)$$

   The desired product of this reaction is the solid silver chromate. Calculate the atom economy of this reaction.
3. What are the hazards, if any, of the reactants silver nitrate, $AgNO_3$, and potassium chromate, K_2CrO_4? What safety practices in the lab should be used to mitigate these hazards?

Materials

Potassium carbonate, 2 g

Sodium carbonate, 2 g

Solid mixture samples:

- Potassium carbonate(K_2CO_3)/potassium bicarbonate($KHCO_3$) mixture, 2g
- Sodium carbonate(Na_2CO_3)/sodium bicarbonate($NaHCO_3$) mixture, 2g

Balance, 0.001-g precision (shared)

Bunsen burner

Crucible and cover

Matches or lighter

Ring clamp

Spatula

Stoichiometry Lab Procedure

Support stand

Triangle, pipe stem

Tongs, crucible

Wire gauze

Safety Precautions

Potassium carbonate and sodium bicarbonate are slightly toxic by ingestion and are skin irritants. Handle the crucible only with tongs. Do not touch the crucible with fingers or hands. There is a significant burn hazard associated with handling a crucible—remember that a hot crucible looks like a cold one. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Thoroughly wash hands with soap and water before leaving the laboratory. Follow all laboratory safety guidelines.

Introductory Activity

Decomposition of Group 1 Bicarbonates

1. Set up a Bunsen burner on a support stand beneath a ring clamp holding a pipe-stem triangle (see Figure 1). Do NOT light the Bunsen burner.
2. Adjust the height of the ring clamp so that the bottom of a crucible sitting in the clay triangle is about 1 cm above the burner. This will ensure that the crucible will be in the hottest part of the flame when the Bunsen burner is lit.
3. Place a crucible with a cover in the clay triangle and heat over a burner flame until the crucible is red hot.

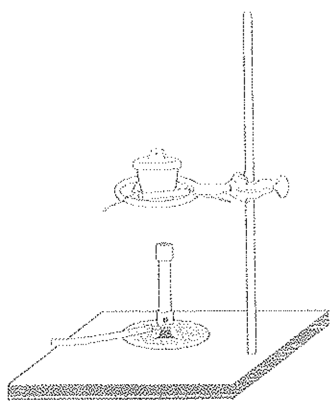

Figure 1.

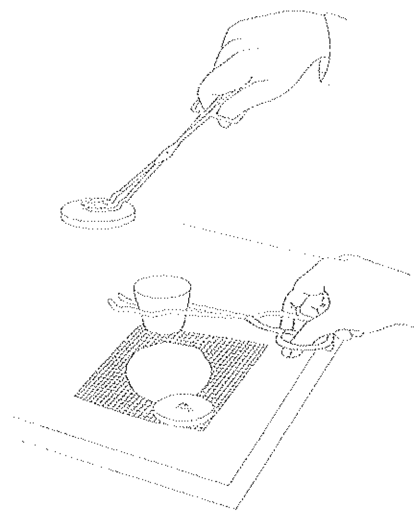

Figure 2.

4. Turn off the gas source and remove the burner.
5. Using crucible tongs, remove the crucible cover and place it on wire gauze on the bench top. With the tongs, remove the crucible from the clay triangle and place it on the wire gauze as well (see Figure 2).
6. Allow the crucible and its cover to cool completely on the wire gauze for at least 10 minutes.
7. Use an analytical balance to find the mass of the crucible and crucible cover. Handle with tongs to avoid getting fingerprints on the crucible and cover.
8. Record their mass.
9. Now add about 2 g of your bicarbonate sample to the crucible. Weigh the crucible, cover, and sample. Record their combined mass.

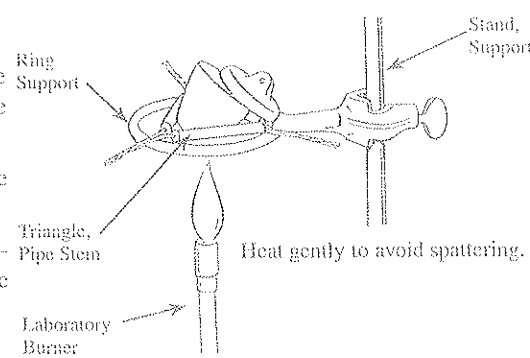

Figure 3.

10. Set the crucible at an angle in the clay triangle held in the ring on the support stand. Cover the crucible loosely with the crucible cover, and heat very gently. It is important that the escaping vapor does not carry any of the solid along with it, so be sure that the crystals are heated very gently for at least five minutes (see Figure 3).

11. Turn off the gas source and remove the burner.
12. Use tongs to remove the crucible cover and place it on wire gauze on the bench top. With the tongs, remove the crucible from the clay triangle and place it on the wire gauze as well.
13. Allow the crucible and its cover to cool completely on the wire gauze for at least 10 minutes.
14. Measure and record the mass of the crucible, cover, and carbonate product.
15. Repeat the drying procedure until constant mass is obtained.
16. Record the final mass of the crucible, cover, and carbonate product.
17. Dispose of the crucible contents according to your instructor's directions. Carefully clean the crucible and crucible cover for use in the next part of the lab.
18. Calculate the percent yield for the carbonate product.

Guided-Inquiry Design and Procedure

Form a working group with other students and discuss the following questions.

1. Based on your results in the *Introductory Activity*, what, if anything, is the product when potassium or sodium carbonate is heated at 100 °C to 200 °C?
2. Review the stoichiometry of the bicarbonate decomposition lab. How does mass loss relate to the mass of the starting material?
3. Review your laboratory procedure on the percent bicarbonate in a mixture, and then answer the following questions. Use reference books and the Internet when needed.
   a. What are the products of this lab?
   b. Are any or all of these products hazardous? If so, in what way?
   c. What are the proper disposal methods for each of these products?
4. The balanced equations for the experiment are listed below.

   $2KHCO_3(s) \rightarrow K_2CO_3(s) + H_2O(g) + CO_2(g)$

   or

   $2NaHCO_3(s) \rightarrow Na_2CO_3(s) + H_2O(g) + CO_2(g)$

   The desired products of these reactions are the solid metal carbonates. Calculate the atom economy of each of these reactions.
5. What are the hazards, if any, of the reactants potassium bicarbonate, $KHCO_3$, and sodium bicarbonate, $NaHCO_3$?
6. Write a detailed step-by-step procedure for the experiment. Include the materials, glassware and equipment that will be needed, safety precautions that must be followed, the mass of reactants, accuracy of any equipment, the required data table, and calculations.
7. Review additional variables that may affect the reproducibility or accuracy of the experiment and how these variables will be controlled.
8. Carry out the experiment and record results in an appropriate data table.

Analyze the results:

Calculate the mass percent of the bicarbonate compound in the solid mixture. Determine your percent recovery.

Opportunities for Inquiry

As an alternative stoichiometric lab, students could determine the optimum mole ratio of two green reactants using the method of continuous variation.

AP Chemistry Review Questions

1. From a mass of 1.678 g of sodium bicarbonate reactant and the balanced chemical equation, calculate the theoretical mass of sodium carbonate solid that should be produced.
2. If 1.018 g of sodium carbonate were produced from the sodium bicarbonate in question 1, calculate the percent yield for the bicarbonate decomposition reaction.
3. Calculate the mass of water vapor and carbon dioxide that would be produced by gently heating a mixture of 1.550 g of sodium bicarbonate and 0.463 g of sodium carbonate. What mass of sodium carbonate would remain in the crucible?
4. A classic gravimetric high school lab experiment involves combining a solution of barium nitrate, $Ba(NO_3)_2$, with a sodium sulfate solution, Na_2SO_4, forming a precipitate of barium sulfate.

   $Ba(NO_3)_2(aq) + Na_2SO_4(aq) \rightarrow BaSO_4(s) + 2NaNO_3(aq)$

   a. Indentify the hazards associated with the chemicals in this reaction.

   b. The purpose of this lab is to teach the techniques and principles of analytical gravimetric analysis. Use your knowledge of solubility products to devise a greener set of solutions that meets the purpose of this lab.

Supplementary Material

Determining the Stoichiometry of a Chemical Reaction

Background

A balanced chemical equation gives the mole ratios of reactants and products for chemical reactions. If the formulas of all reactants and products are known, it is relatively easy to balance an equation to find out what these mole ratios are. When the formulas of the products are not known, experimental measurements must be made to determine the ratios.

This laboratory uses the method of continuous variations to determine the mole ratio of two reactants. Several steps are involved. First, solutions of the reactants are prepared in which the concentrations are known. Second, the solutions are mixed a number of times using different ratios of reactants. Third, some property of the reaction that depends on the amount of product formed or on the amount of reactant that remains is measured. This property may be the color intensity of a reactant or product, the mass of a precipitate that forms, or the volume of a gas evolved.

In the method of continuous variations, the total number of moles of reactants is kept constant for the series of measurements. Each measurement is made with a different *mole ratio* of reactants. The optimum ratio, which is the *stoichiometric ratio* in the equation, should consume the greatest amount of reactants, form the greatest amount of product, and, if the reaction is exothermic, generate the most heat and maximum temperature change.

In this laboratory, the amount of precipitate formed in a *double replacement reaction* is the property that will be measured. Seven different mole ratios of reactants are added to 50-mL graduated cylinders. The volume of precipitate formed for each mole ratio is measured and these volumes are plotted versus the mole ratio.

Materials

Potassium chromate solution, K_2CrO_4, 0.5 M, 210 mL

Silver nitrate solution, $AgNO_3$, 0.5 M, 210 mL

Marker or labeling pen

Graduated cylinders, 50-mL, 2

Graduated cylinders, 100-mL, 7

Stirring rods, long, 2

Safety Precautions

Potassium chromate solutions are known carcinogens, may be corrosive to skin and eyes, and are moderately toxic by ingestion. Avoid contact with eyes and skin. Silver nitrate solutions are moderately toxic; they will stain skin and eyes. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Wash hands thoroughly with soap and water before leaving the laboratory. Follow all laboratory safety guidelines.

Disposal

Review all federal, state and local regulation that may apply, before proceeding. All solid chromate compounds and solutions must be saved for disposal by a licensed waste disposal company.

Procedure

Reaction of Silver Nitrate with Potassium Chromate

1. Label seven 100-mL graduated cylinders 1–7.
2. Using a clean, 50-mL graduated cylinder, add the appropriate volume of silver nitrate solution to each 100-mL graduated cylinder, as shown in Table 1.
3. Use a second 50-mL graduated cylinder to add the appropriate volume of potassium chromate solution to each 100-mL graduated cylinder, as shown in the data table.

4. Use a large stirring rod to thoroughly mix the reactants. Observe the signs of chemical reaction in each cylinder. (Mixing the clear solution of silver nitrate with the yellow potassium chromate solution gives a rust-colored precipitate and a pale yellow supernatant.)

5. Let the reaction mixtures sit undisturbed for at least 10 minutes to allow the precipitates to settle.

6. After the precipitates have settled, record the volume of precipitate in each graduated cylinder in the Data Table.

7. Collect the waste in the hood as directed by the instructor—chromium compounds are heavy metals and require licensed hazardous waste disposal.

Data Table

Cylinder	1	2	3	4	5	6	7
$AgNO_3$, 0.5 M, mL	10	15	20	30	40	45	50
K_2CrO_4, 0.5 M, mL	50	45	40	30	20	15	10
$Ag:CrO_4$ Mole Ratio	1:5	1:3	1:2	1:1	2:1	3:1	5:1
Precipitate, mL							

Analysis

1. On graph paper, plot the milliliters of reactant #1 versus volume of precipitate for each reaction. Draw the two best-fit straight lines through the data points and determine their point of intersection.

2. From the point of intersection, determine the stoichiometric mole ratio for the reaction. Write out the correct balanced equation for the reaction.