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Determining the Empirical Formula of Zinc Chloride

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Purpose of the Experiment Determine the empirical formula of zinc chloride, a compound composed of only zinc (Zn) and chlorine (Cl).

Background Required You should be familiar with basic laboratory techniques for measuring mass and volume. You should understand the concepts associated with chemical formulas, ionic compounds, molar mass, and molarity.

Background Information We can represent a compound using two types of **chemical formulas,** each of which indicates something about the kinds and numbers of atoms making up one molecule of that compound. The **molecular formula** shows the actual numbers and kinds of atoms in one molecule, while the **empirical formula** shows the smallest, whole-number ratio of atoms in one molecule. For example, the molecular formula of acetylene is C_2H_2, indicating that there are two carbon (C) atoms and two hydrogen (H) atoms in one acetylene molecule. Therefore, the C:H atomic ratio in acetylene is 2:2. However, we can divide each number in this ratio by 2, yielding the smallest, whole-number C:H atomic ratio, 1:1. Acetylene's empirical formula is therefore C_1H_1, which we express as CH.

In this experiment, we will determine the empirical formula of zinc chloride. To do so, we will dissolve a known mass of zinc chloride in a measured volume of water, then determine the zinc content of the resulting solution by titration. In **titration,** we measure the volume of a **standardized** (known concentration) **solution** required to completely react with a target substance in the solution being analyzed. We add an indicator to the titration mixture in order to signal the **end point.** The indicator changes color when the exact volume of standardized solution needed to completely react with the sample has been added. The reaction is complete at this point, also called the **equivalence point.**

NOTE: The concentration of a standardized solution is usually expressed in units of molarity (*M*, or mol/L).

Under the proper conditions, ethylenediaminetetraacetic acid (EDTA) reacts with many metal ions, including zinc, to produce stable metal–EDTA complexes. Therefore, we can use a standardized EDTA solution in our titration to determine the zinc concentration in our zinc chloride solution. Whether or not a particular metal ion will complex with EDTA depends upon the pH of the reaction solution. Solution pHs of 10 or greater are required in order for EDTA to complex with calcium (Ca^{2+}), magnesium (Mg^{2+}), and zinc (Zn^{2+}) ions in water, particularly when Eriochrome Black T (EBT) is used as the indicator, which is the case in this experiment.

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The reaction of EDTA with Zn^{2+} is shown in Equation 1.

$$Zn^{2+}(aq) + EDTA^{4-}(aq) \rightarrow [ZnEDTA]^{2-}(aq) \qquad \text{(Eq. 1)}$$

EBT is red in zinc solutions containing Zn^{2+} that is not complexed with EDTA. At the equivalence point, when the number of moles of EDTA added exactly equals the number of moles of Zn^{2+} originally present, EBT turns blue, with no traces of red or purple. This color change marks the end point of the titration, indicating that the reaction is complete.

Example Calcium oxide is a white solid composed of only calcium (Ca) and oxygen (O). Suppose you dissolve 0.206 g of calcium oxide in a few milliliters of hydrochloric acid (HCl), then add enough water to make 100.0 mL of solution. You obtain a 25.00-mL sample of this solution, adjust the pH to 10, and titrate with 0.0225*M* EDTA, using EBT indicator. The titration requires 40.82 mL of EDTA solution to reach its end point, identified by a color change from red to blue. The titration reaction is shown in Equation 2.

$$Ca^{2+}(aq) + EDTA^{4-}(aq) \rightarrow [CaEDTA]^{2-}(aq) \qquad \text{(Eq. 2)}$$

Problem 1 Calculate the mass of calcium oxide in the 25.00-mL titrated sample.

Solution First we divide the starting mass of calcium oxide (0.206 g) by the total solution volume (100.0 mL) to determine the solution concentration in g/mL. Then we multiply the result by 25.00 mL, the volume of the titrated sample.

$$\begin{aligned}\text{mass of calcium oxide in 25.00-mL sample, g} &= \left(\frac{0.206\text{ g calcium oxide}}{100.0\text{ mL}}\right)(25.00\text{ mL})\\ &= 0.0515\text{ g calcium oxide}\end{aligned}$$

Problem 2 Calculate the mass of Ca in the titrated sample.

Solution First we calculate the number of moles of EDTA required to reach the equivalence point of the titration. To do so, we multiply the volume (in L) of EDTA solution added by its concentration. Then we use Equation 2 to determine that 1 mol of EDTA reacts with 1 mol of Ca^{2+}.

$$\begin{aligned}\text{number of moles of } Ca^{2+} \text{ in titrated sample, mol} &= (40.82\text{ mL})\left(\frac{1\text{ L}}{1000\text{ mL}}\right)\left(\frac{0.0225\text{ mol EDTA}}{1\text{ L}}\right)\left(\frac{1\text{ mol } Ca^{2+}}{1\text{ mol EDTA}}\right)\\ &= 9.18\times10^{-4}\text{ mol } Ca^{2+}\end{aligned}$$

We note that the number of moles of Ca^{2+} in the titrated sample must equal the number of moles of Ca atoms in the sample. Therefore, we can use the molar mass of Ca (40.08 g/mol) to determine the mass of Ca in the titrated sample.

$$\begin{aligned}\text{mass of Ca in titrated sample, g} &= (9.18\times10^{-4}\text{ mol Ca})\left(\frac{40.08\text{ g Ca}}{1\text{ mol Ca}}\right)\\ &= 0.0368\text{ g Ca}\end{aligned}$$

Problem 3 Calculate the mass of O in the titrated sample.

Solution Calcium oxide is composed of only Ca and O, so if we subtract the mass of Ca in the titrated sample from the mass of calcium oxide in the titrated sample, the remaining mass must be O.

$$\begin{matrix}\text{mass of O in}\\ \text{titrated sample, g}\end{matrix} = 0.0515 \text{ g calcium oxide} - 0.0368 \text{ g Ca} = 0.0147 \text{ g O}$$

Problem 4 Calculate the number of moles of O in the titrated sample.

Solution We use the molar mass of O (16.00 g/mol) to convert mass to moles.

$$\begin{matrix}\text{number of moles of O}\\ \text{in titrated sample, mol}\end{matrix} = (0.0147 \text{ g O})\left(\frac{1 \text{ mol O}}{16.00 \text{ g O}}\right) = 9.19 \times 10^{-4} \text{ mol O}$$

Problem 5 Determine the empirical formula of calcium oxide.

Solution First we determine the molar ratio of Ca to O in our calcium oxide sample. To do so, we divide the number of moles of each element by the smaller number of moles.

$$\text{Ca: } \left(\frac{9.18 \times 10^{-4} \text{ mol Ca}}{9.18 \times 10^{-4}}\right) = 1.00$$

$$\text{O: } \left(\frac{9.19 \times 10^{-4} \text{ mol O}}{9.18 \times 10^{-4}}\right) = 1.00$$

Thus, the molar ratio of Ca to O in the compound is 1:1, which is also the atomic ratio. Because this ratio cannot be simplified further, the empirical formula of calcium oxide is Ca_1O_1, expressed as CaO.

Procedure

Caution: Wear departmentally approved safety goggles while doing this experiment.

Use caution when working with all chemicals. Many chemicals are potentially harmful. Prevent contact with your eyes, skin, and clothing. Do not ingest any of the reagents.

Note: Record all measurements on your Data Sheet.

Record all masses to either the nearest milligram (0.001 g) or centigram (0.01 g), as indicated by your laboratory instructor.

Label a 400-mL beaker "Discarded Solutions", and use it throughout the experiment. Dispose of all solids and solutions according to your laboratory instructor's directions.

1. Determine and record the mass of a clean, dry watch glass. Place about 0.2 g of zinc chloride on the watch glass. Record the exact mass of the zinc chloride plus watch glass. Set the glass aside on your laboratory bench [NOTE 1].

NOTE 1: Place the watch glass and zinc chloride where they will not be disturbed during the laboratory period.

2. Tare a clean, dry 50-mL beaker. Measure between 0.29 and 0.32 g of zinc chloride into the tared beaker. Record the exact mass of zinc chloride in the beaker [NOTE 2].

NOTE 2: Make sure you tightly stopper the zinc chloride reagent bottle after use.

3. Dissolve the zinc chloride in the beaker by adding 10–20 mL of distilled or deionized water. ***Being careful not to lose any solution,*** pour the liquid into a clean, 100-mL volumetric flask. Use a wash bottle to rinse the beaker with about 10 mL of distilled water. Transfer the rinse into the flask. Repeat the rinse procedure three more times, transferring the rinses into the flask. Next, use a 10-mL graduated cylinder to add 10 mL of 0.1*M* HCl to the flask. Fill the flask exactly to the calibration mark on its neck with distilled water. Stopper the flask. With your index finger firmly holding the stopper in place, invert the flask at least ten times to thoroughly mix the solution.

4. Using a buret clamp, attach a clean, dry 50-mL buret to a support stand, as shown in Figure 1. Rinse the buret with a ***small amount*** (less than 10 mL) of the solution you prepared in Step 3 [NOTE 3]. Collect the rinse in your Discarded Solutions beaker. Then use the same solution to fill the buret to a level just above the 0.00-mL mark. Slowly drain the solution from the buret into the Discarded Solutions beaker, until the bottom of the **meniscus** (the curved surface of the liquid in the buret) aligns with, or is slightly below, the 0.00-mL mark. Label the buret, "Zinc Chloride".

NOTE 3: Use a clean, dry, short-stem funnel to transfer solution into the buret.

5. Clamp, rinse, and fill a second clean, dry 50-mL buret with EDTA solution, following the procedure in Step 4. Label the buret "EDTA". Record the exact EDTA solution concentration.

6. Record the initial volume reading for the Zinc Chloride buret [NOTE 4]. Deliver 20–25 mL of the zinc chloride solution into a clean, 125-mL Erlenmeyer flask. Record the buret's final volume reading.

NOTE 4: A 50-mL buret is calibrated in 0.1-mL units, but measurements to the nearest 0.02 mL can be reproducibly estimated. Therefore, you should estimate and record every buret reading to the nearest 0.02 mL. When reading the liquid level in a buret, hold a white card with a dark stripe directly behind the buret, with the top edge of the stripe slightly below the bottom of the meniscus, as shown in Figure 2. Keep your line of sight level with the bottom of the meniscus.

7. Use a clean, 10-mL graduated cylinder to transfer 2 mL of pH 10 buffer solution to the Erlenmeyer flask. Mix by swirling the flask and its contents, or use a clean stirring rod. Add 3 drops of EBT indicator solution to the flask. Swirl again to mix.

8. Record the initial volume reading for the EDTA buret. Titrate the solution in the Erlenmeyer flask, adding 1–2 mL of EDTA solution at a time, until you see the titration mixture turning blue at the point where the

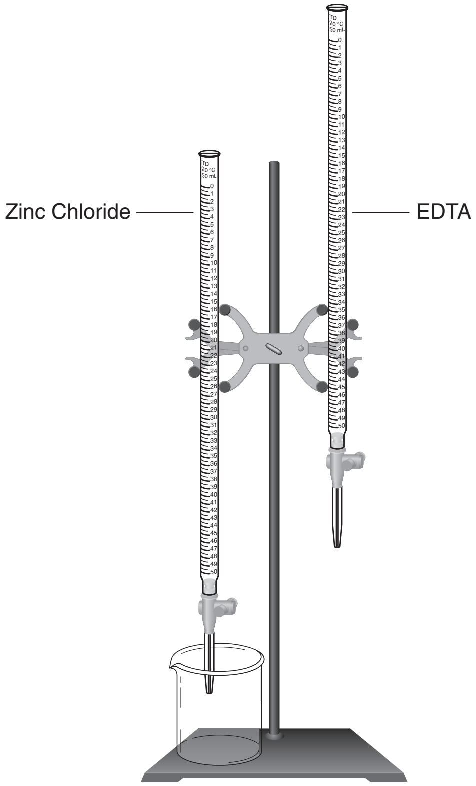

Figure 1 *A titration assembly*

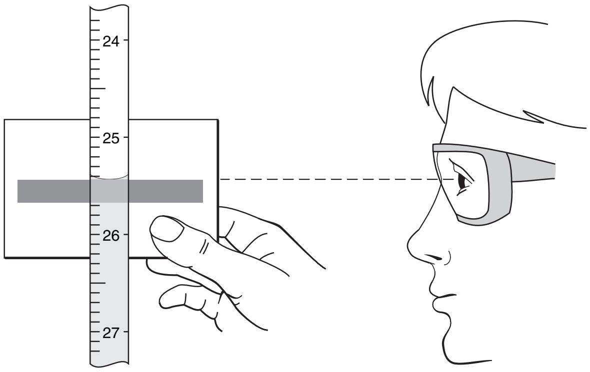

Figure 2 *Reading a buret*

EDTA enters the mixture. Then begin adding EDTA in drops. After each EDTA addition, gently swirl the flask to mix. As soon as the mixture in the flask turns completely blue, with no trace of red or purple, the titration has reached the end point. Stop adding EDTA. Record the final volume reading in the EDTA buret.

9. Pour the reaction mixture from the flask into the Discarded Solutions beaker. Rinse the flask with tap water, then distilled water.

10. Refill the Zinc Chloride and EDTA burets with their respective solutions.

11. Perform a second and a third titration by repeating the procedure in Steps 6–10.

12. Once again, determine and record the mass of the watch glass plus zinc chloride that you put aside at the beginning of the Procedure. Record any changes in the appearance of the zinc chloride.

13. Dispose of the solutions in your burets, volumetric flask, and Discarded Solutions beaker, as well as the zinc chloride on your watch glass, as directed by your laboratory instructor. Rinse all your glassware with tap water, then distilled water. Allow the glassware to drain.

Caution: Wash your hands thoroughly with soap or detergent before leaving the laboratory.

name ____________________ section ____________ date ____________

Data Sheet

mass of watch glass, g ____________

mass of watch glass plus zinc chloride at beginning of laboratory period, g ____________

mass of watch glass plus zinc chloride at end of laboratory period, g ____________

observations
changes in the appearance of zinc chloride on watch glass:

mass of zinc chloride in 50-mL beaker, g ____________

concentration of EDTA solution, mol/L ____________

	determination		
	1	*2*	*3*
initial volume reading for zinc chloride buret, mL	________	________	________
final volume reading for zinc chloride buret, mL	________	________	________
initial volume reading for EDTA buret, mL	________	________	________
final volume reading for EDTA buret, mL	________	________	________

Results Sheet

Complete the following calculations for each determination. Record your answers in the spaces provided. Use additional paper for calculations, if necessary.

		determination	
	1	*2*	*3*
volume of zinc chloride solution sample, mL			
mass of zinc chloride in sample, g			
volume of EDTA solution used in titration, mL			
number of moles of Zn^{2+} (Zn) in titrated sample, mol			
mass of Zn^{2+} (Zn) in titrated sample, g			
mass of Cl in titrated sample, g			
number of moles of Cl in titrated sample, mol			
molar ratio of Zn:Cl in zinc chloride			
empirical formula of zinc chloride			

name section date

Interpretation of Your Results

Use the spaces provided for the answers and additional paper if necessary.

1. In this experiment, you prepared a stock solution of zinc chloride, then analyzed measured samples of that solution.

(a) Would it have been practical for you to titrate the entire 100-mL zinc chloride solution directly? Briefly explain your answer.

(b) Would your results have been more accurate if you had weighed three separate samples of solid zinc chloride and dissolved each in 25.0 mL of distilled water, rather than preparing a 100-mL solution and analyzing portions of it? Briefly explain.

2. Solid zinc chloride is often described as **deliquescent,** meaning that it readily absorbs water from the atmosphere.

(a) What experimental evidence do you have that indicates zinc chloride is deliquescent?

(b) If the zinc chloride you used to make the 100-mL solution had absorbed some atmospheric water before you weighed it in the beaker, would your experimentally determined number of moles of Zn have been incorrectly high, incorrectly low, or unaffected? Briefly explain.

(c) Based on your experimentally determined molar ratio of Zn to Cl, do you suspect that your zinc chloride had absorbed some water before you weighed it? Briefly explain.

3. Tap water, particularly in locations where the water is "hard", may contain relatively high concentrations of Ca^{2+} and Mg^{2+}. Suppose that the tap water in your laboratory is hard, and that you used tap water, not distilled water, in Step 3 to prepare the zinc chloride solution.

(a) What effect would the tap water have had on the volume of EDTA required for the titration? Briefly explain.

(b) What effect would the tap water have had on the calculated mass of Zn in the sample? Briefly explain.

(c) What effect would the tap water have had on the calculated mass of Cl in the sample? Briefly explain.

(d) What effect would the tap water have had on your determination of the empirical formula of zinc chloride? Briefly explain.

name

section

date

Pre-Laboratory Assignment

1. Why is it important to add a buffer solution to the titration mixture when titrating Zn^{2+} with EDTA, using EBT as the indicator? Briefly explain.

2. Determine the empirical formulas of the compounds listed below.

(a) hydrazine (N_2H_4), a compound used in rocket fuel

(b) aspartame ($C_{14}H_{18}O_5N_2$), an artificial sweetener

3. In order to determine the empirical formula of a compound composed of only Ca and carbon (C), a student prepared a solution of the compound by dissolving 0.114 g of the compound in 100.00 mL of distilled water. Titration of a 25.00-mL sample of the solution at pH 10 required 36.18 mL of 0.0122*M* EDTA solution, using EBT as the indicator.

(a) Calculate the mass of dissolved compound in the 25.00-mL titrated sample.

(b) Calculate the number of moles of Ca in the titrated sample.

(c) Calculate the mass of Ca in the titrated sample.

(d) Calculate the mass of C in the titrated sample.

(e) Calculate the number of moles of C in the titrated sample.

(f) Determine the molar ratio of Ca:C in the compound.

(g) Determine the empirical formula of the compound.

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