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Determination of an Empirical Chemical Formula: I with Zn

Introduction.

Review section 5.5 of your text for more background information.
The purpose of this experiment is to determine the empirical formula of the substance that results from the reaction of elemental zinc with elemental iodine.

$$Zn + I_2 \rightarrow Zn_xI_y$$

You will carry out this reaction in methanol. Iodine is soluble in methanol but Zn is not. When a soluble substance reacts with an insoluble solid, this is referred to as a heterogeneous reaction since the two reactants are in different phases. Your heterogeneous reaction will take place with a stoichiometric excess of zinc; therefore, some zinc will remain after the reaction is over, but all of the iodine will be consumed. By measuring the change in mass of the zinc and by assuming that all of the iodine was consumed by its reaction with zinc, you can calculate the mole ratio of the two elements that form the product. Through your data analysis you will find the empirical formula of the zinc-iodine compound, Zn_xI_y, where x and y are whole numbers.
In this experiment, you will need to accurately measure the masses of your reactants (zinc and iodine), the mass of unreacted zinc and the mass of your product. This enables you to confirm the Law of Conservation of Mass by comparing the amount of zinc and iodine that are consumed in the reaction with the amount of product that is formed.

Method.

For this experiment you will need the following items:
1) 125 mL Erlenmeyer flask
2) 150 mL beaker
3) Stir rod
4) Variable temperature hot plate
5) Aluminum foil
6) Weighing paper

Goal: React an excess of zinc with iodine and determine the empirical formula of the resulting product. Once you have determined the masses of zinc and iodine that were consumed in the reaction and the mass of your product, you can verify the Law of Conservation of Mass.

Note: when weighing the flask or beaker, you must record their weight with milligram precision; this means three decimal places on the scale. If the scale shows four decimal places, record all four.

1. Use an analytical balance to weigh a clean, dry 125 mL Erlenmeyer flask. Record the mass to milligram precision or better.
2. On a piece of weighing paper, weigh out 2.0 ± 0.1 g of granular zinc metal.

3. Transfer the granular zinc to the Erlenmeyer flask and reweigh it on the analytical balance and record the mass of the flask containing zinc to milligram precision or better.
4. Using a weigh boat, weigh out 2.0±0.1 g of iodine crystals. **Clean up any spilt iodine to avoid damage to the scales.**
5. Add the iodine crystals to the Erlenmeyer flask that contains the zinc metal. Again reweigh the flask on the analytical balance and record the mass with milligram or better precision.
6. Obtain 25 mL of methanol and add it to the Erlenmeyer flask. The methanol serves as the solvent to facilitate the reaction and dissolve the product of the reaction.
7. Because methanol is volatile it produces a toxic, flammable vapor. For this reason cap your Erlenmeyer with aluminum foil.
8. Place the flask on a hot plate under your fume hood.
9. Set the hot plate to a low setting and allow the solution to warm. Adjust your temperature until you observe the methanol begin to boil, and keep it at a low boil. The boiling point of methanol is about 65°C. **Keep the boiling action mild. Do not bring it to a rapid boil since you will risk evaporating your methanol.**
10. While the methanol is at a low boil, you should be able to see the methanol change color as the iodine is consumed during the formation of the zinc-iodine product. Record these observations on your data sheet.
11. While the reaction is proceeding (this takes 20 minutes) you can weigh a clean, dry, empty 150 mL beaker. Record the mass with milligram precision or better.
12. Let the methanol continue to boil for 20 minutes. By this point the iodine color should have disappeared (it may remain very slightly pale yellow). The absence of color indicates that all of the iodine has been consumed by its reaction with zinc. The excess zinc should still be visible in its solid form at the bottom of the beaker. Turn down the heat on the hot plate and record your observations on the data sheet.
13. Remove the flask from the hot plate. Use tongs or a paper towel wrapped around the neck of the flask to avoid burning yourself on the hot flask.
14. Working at your fume hood, carefully pour the liquid contents of your Erlenmeyer flask into the beaker; be careful not to allow any of the remaining solid zinc to get in the beaker. You can use your glass stir rod to help guide the liquid stream into your beaker.
15. Your Erlenmeyer will now contain the unreacted zinc and a small amount of the methanol solution which contains some of your reaction product. To recover as much of the reaction product as possible you will now add an additional 5 mL of methanol to the Erlenmeyer. Swirl the methanol and then allow the zinc to settle back to the bottom of the Erlenmeyer. With the unreacted zinc settled in the flask, once again carefully pour the liquid contents into the beaker.
16. Repeat step 15 two more times with 5 mL volumes of methanol. This will help ensure you have retrieved as much of your product as possible. At this point, you should have flask containing wet, unreacted zinc and a beaker containing your methanol washings. Your zinc-iodine product is in the methanol washings.
17. Place both the beaker and the Erlenmeyer flask (without the aluminum foil covering the top) back on the hot plate. Keep the plate set to a medium temperature and let the methanol evaporate from both vessels. **The zinc will become dry first. Follow steps 18-20 while you are waiting for the methanol in the beaker to evaporate.**

18. Swirl the flask occasionally as the zinc metal dries. Once the zinc is dry and moves freely around the bottom of the flask when you swirl, remove the flask from the hot plate and let it cool to room temperature.
19. If the zinc looks like the original metal you used, then you are ready to weigh the unreacted zinc. If it has a white residue, then you need to rinse again with methanol and transfer that rinsed methanol to the beaker. Repeat until you have clean, dry zinc in the bottom of your flask.
20. Once you have dry, unreacted zinc in your flask, reweigh the flask and record its weight on the data sheet.
21. Continue heating the beaker. As your product begins to crystallize place a watch glass **over the beaker in case the remaining solvent starts to "pop". Keep heating** until the methanol has boiled and evaporated to dryness. Once the methanol appears to be completely evaporated, wait an additional two or three minutes to allow your product to become completely dry.
22. Let the beaker cool to room temperature, weigh it and record the weight.

Literature Cited DeMeo, S. "Synthesis and Decomposition of Zinc Iodide: Model Reactions for Investigating Chemical Change in the Introductory Laboratory." J. Chem. Ed. 1995, 72, 836-839.

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Data Sheet and Calculations

A) Mass of empty Erlenmeyer flask_____.

B) Mass of Erlenmeyer flask containing zinc granules _____.

C) Mass of Erlenmeyer flask containing both zinc granules and iodine crystals_____.

D) Report what you observe during the heating of the reaction and at the end of 20 minutes.

E) Mass of empty 150 ml beaker _____

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F) Mass of Erlenmeyer flask containing the dry unreacted zinc from step 20 _____

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Once you know the mass of the unreacted zinc you can find the number of moles of zinc that reacted. In the space below show your calculations and fill in the indicated values. **Report all masses in milligram or better precision by taking the differences in the weights recorded on your data sheet. Show your calculations.**

Initial mass of zinc _____.

Mass of unreacted zinc _____.

Mass of reacted zinc_____.

Moles of reacted zinc _____.

Next, you can make the assumption that all of the iodine was consumed in the reaction. Under this assumption it is possible to calculate a mole ratio for the zinc-iodine product.

The assumption is: Mass of iodine consumed during the reaction is equal to the initial mass of iodine. **Report all masses in milligram or better precision by taking the differences in the weights recorded on your data sheet. Show your calculations.**

Mass of iodine consumed during the reaction _____.

Moles of iodine consumed during the reaction _____.

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Mole ratio moles of I reacted/moles of Zn reacted = _____.

Using your mole ratio – determine the empirical formula for your zinc-iodine product and report it here.

Empirical formula of product _____

Conservation of Mass. Compare the mass of the dry product that remained in your beaker after evaporating the methanol to the sum of the masses of the two reactants that were consumed.

Mass of beaker with your dry reaction product from step 22 _____.

Theoretically the mass of your product should equal the sum of the masses of zinc and iodine that were consumed in the reaction, but will vary due to experimental error.

Mass of product in beaker _____.

Combined mass of reacted zinc and reacted iodine _____.

Calculate the percent error between these two values. Explain the possible sources of error.