Synthesis of Zinc Iodide Revisited

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An experiment concerning the synthesis of zinc iodide from its elements was discussed in an article published in this *Journal (1).* The author suggested that the experiment could help introductory chemistry students understand some fundamental concepts related to stoichiometry. In this article, two additional experiments related to zinc iodide synthesis are presented; the experiments involve an alternate synthesis as well as an investigation of a side reaction. These two experiments provide an inquiry-based experience that is thematically connected to the previous work with zinc iodide. The students conduct the experiments, isolate the products (which are all white solids), and seek to identify the products by searching the literature, and choosing appropriate tests on the products and standards. Students also evaluate the two reaction pathways. Teachers can use these experiments as laboratory experiences for their chemistry classes or as independent projects for selected students.

Experiment 1: An Alternative Synthesis of Zinc Iodide, A Double Replacement Reaction

In this experiment students investigate an alternative method of synthesizing zinc iodide and compare it to the previous method synthesizing zinc iodide from its elements. From their understanding of double replacement reactions, solubility rules, and molarity, students hypothesize that barium iodide reacts with zinc sulfate to produce zinc iodide. The equation for this reaction is

$$BaI_2(aq) + ZnSO_4(aq) \rightarrow ZnI_2(aq) + BaSO_4(s)$$

Two questions are posed to the students: (1) Will barium iodide and zinc sulfate react to make zinc iodide as postulated? and (2) Which reaction is preferred from a chemist's perspective, the synthesis of zinc iodide from its elements or from the double displacement reaction? Students compare both syntheses in terms of the amount of product that is made, efficiency (reaction time and the ease of isolating the product), safety issues, and cost. This activity can also be used to discuss limiting and excess reagents in an introductory chemistry course both on the college and high school level.

After the students synthesize[W] and isolate[W] the two solid products, qualitative and quantitative tests are conducted to confirm their identities. The tests are also performed on standards to aid in the interpretation.

Qualitative Tests on the Dried White Solid

Three tests are performed to qualitatively identify the dried white solid isolated from the supernatant as zinc iodide. The tests are performed on the reaction product and standard(s). The isolated product is dissolved in deionized water. The presence of the zinc ion can be confirmed by reaction with magnesium metal. A shiny magnesium turning is covered in black zinc solid when placed in a solution containing zinc ions. The presence of zinc ions can also be assessed with dithizone (diphenylthiocarbazone) paper.[W] The paper turns red-purple when a drop of Zn^{2+}-containing solution is applied to it. Addition of silver nitrate to the aqueous sample gives a pale yellow precipitate if the solution contains iodide ion.

Qualitative Tests on the White Precipitate

Three tests are performed to qualitatively identify the precipitate as barium sulfate. The tests are performed on the reaction product and standard(s). The presence of barium in the white precipitate is assessed by adding deionized water to the solid and using the supernatant to carry out the following tests. When a solution containing barium ions is placed into a flame a faint greenish color is observed. When carbonate ion (0.1 M) is added to the solution a white precipitate is observed. This precipitate is soluble in acetic acid.

The presence of the sulfate ion (SO_4^{2-}) is confirmed by adding several drops of 6 M HCl to the solution and a few drops of barium chloride. A fine white precipitate forms if SO_4^{2-} ions are present.

Results

Based on the qualitative tests, students confirm that barium iodide reacts with zinc sulfate to produce zinc iodide. Quantitatively, zinc iodide can be synthesized by the double replacement reaction with close to a 100% yield[1] (Table 1). Comparison of the two syntheses for zinc iodide shows that both reactions produce comparable yields, do not produce any hazardous wastes, and do not necessitate any unusual safety demands (Table 2). The key difference between the two syntheses involves the efficiency and the cost. The synthesis of zinc iodide from its elements involves an easier separation technique and is faster to perform since centrifuging is not necessary. Based on the cost per kilogram of the starting materials, the elemental synthesis is less expensive than the double displacement reaction. From this analysis, it is concluded that the synthesis of zinc iodide from its elements is the preferred method of preparing the product.

Experiment 2: The Role of Acetic Acid and the Identification of Zinc Hydroxide

Dilute aqueous acetic acid was used as a reaction medium in the synthesis of zinc iodide from its elements *(1).* An aqueous medium allows the transfer of electrons between reactants. In this experiment students react zinc and iodine in deionized water, omitting acetic acid. Without the acid students observe a fluffy white precipitate, zinc hydroxide, whereas no precipitate is observed with the acid. The purpose of this experiment is to allow students to isolate and identify the product and then to hypothesize the reason dilute acetic acid was used in the synthesis. This experiment would be appropriate for introductory college students who are studying acid–base chemistry.

Table 1. Calculations for the Zinc Iodide Double Replacement Reaction

Data	Student 1	Student 2
Raw Data		
Mass of $ZnSO_4$	0.26 g	0.25 g
Mass of $BaI_2{\cdot}2H_2O$	0.66 g	0.68 g
Mass of large test tube, two boiling chips, and dried solid (ZnI_2)	23.78 g	19.42 g
Mass of large test tube and two boiling chips	23.27 g	18.93 g
Mass of dried solid (ZnI_2) – Actual Yield	0.51 g	0.49 g
Calculated Data		
Moles of $ZnSO_4$	0.0016	0.0015[a]
Moles of $BaI_2{\cdot}2H_2O$	0.0015[a]	0.0016
Moles of ZnI_2	0.0015	0.0015
Theoretical yield of ZnI_2	0.49 g	0.49 g
Percentage yield of ZnI_2	103%[b]	99%

[a]This is the limiting reagent.

[b]Yields greater than 100% are most likely due to the incomplete evaporation of water during the drying process or the absorption of water from the atmosphere after drying.

Table 2. Comparison of the Two Syntheses of Zinc Iodide

Comparison Feature	Elemental Synthesis	Double Replacement Synthesis
Percentage Yield	Close to100%	Close to100%
Efficiency	Fast reaction Easy to separate product from excess reactant	Fast reaction Separation is slow due to centrifuging
Safety	Chemicals as used are not hazardous	Barium compounds are poisonous
Cost	Less expensive	More expensive

Zinc iodide is produced by a redox reaction between zinc metal and iodine. In the absence of acetic acid, zinc iodide is hydrolyzed to zinc hydroxide by the following equation *(2)*:

$$ZnI_2(aq) + 4H_2O(l) \longrightarrow Zn(OH)_2(s) + 2H_3O^+(aq) + 2I^-(aq)$$

Dilute acetic acid prevents observation of the white zinc hydroxide precipitate by immediately reacting with it as represented in the following acid–base equation

$$Zn(OH)_2(s) + 2HC_2H_3O_2(aq) \longrightarrow Zn^{2+}(aq) + 2C_2H_3O_2^-(aq) + 2H_2O(l)$$

The reaction is carried out in dilute acetic acid is used is to prevent students from mistakenly identifying the white precipitate as zinc iodide, which is also a white compound.

Observations and Deductions

A fine white precipitate, which accumulates with time, is formed when zinc and iodine react in the absence of acetic acid. When diluted with water the precipitate is more visible. It appears as a fluffy white suspension.

The students are asked to identify the precipitate. Possible identities of the white precipitate based on the knowledge that the synthesis of zinc iodide involves iodide ions, zinc ions, and water, are:

1. $Zn(OH)_2$: This is an ionic substance and the literature describes this as a white solid.
2. ZnO: This is an ionic substance and it is also described as a white solid in the literature.

Some unlikely combinations are also proposed by the students. Students often use handbooks to look for compounds containing some of the elements that exist in the solution and then combine them even though they do not make sense theoretically. The following are some examples: (i) HI—This substance is a gas, therefore it is an impossible choice as a precipitate. Dissolved in water, HI is a strong acid that dissociates to iodide and hydronium ions.; (ii) $ZnIO_3$—This ionic substance forms by the combination of zinc ion and iodate ion. There is no iodate in solution, therefore this possibility is ruled out. If students want to perform a qualitative test for themselves, one is available.;[W] and (iii) I_2—Iodine is not an ionic compound. Iodine and the equilibrium mixture iodide–iodine–triiodide are not white solids. A qualitative test is available to students.[W]

Qualitative Test for Zinc Hydroxide[2]

Deionized water is added to the isolated product. A shiny magnesium turning changes to black when placed in a solution of the sample indicating the presence of zinc ions.

Dithizone (diphenylthiocarbazone) paper turns red-purple when a drop of the sample solution is applied to it again indicating the presence of zinc ions. The unknown white precipitate is soluble in 0.17 M acetic acid and the white precipitate forms a colorless solution when 20 mL of 1 M NaOH is added.

These tests indicate that the unknown precipitate is probably zinc hydroxide; however zinc oxide is still a possibility. Thus tests for zinc oxide must be conducted.

Qualitative Test for Zinc Oxide

Tests are conducted on the white precipitate and compared directly to tests run on a sample of known zinc oxide. The solubility of the white unknown precipitate in 20 mL of 0.17 M acetic acid is very different from the solubility of zinc oxide in this acidic solution. The solubility of the white unknown precipitate in 20 mL of 1 M sodium hydroxide is very different from the solubility of zinc oxide in this basic solution. Thus zinc oxide is eliminated.

Hazards

When properly handled zinc and solid iodine are nontoxic or only pose slight hazards. Iodine gas is toxic, but is not produced in this activity *(3).* Solids should not be disposed of down the drain. Zinc, iodine, and zinc iodide can be disposed of by changing the solids to ions and then diluting them in a drain attached to a sewer system at a rate that is in compliance with local authorities *(4).* Recycling of the excess zinc after the synthesis also can be done if desired. Barium sulfate is hygroscopic as well as air and light sensitive. It should be stored in an amber glass container surrounded by desiccant. Barium iodide and barium sulfate are harmful if ingested *(2)*; safety precautions such as washing hands after lab should be enforced. The strong base, NaOH, and the strong acid, HCl, should be used carefully. Base spills should be neutralized with a dilute acid, and acid spills should be neutralized with a dilute base. If either substance gets on the skin, it should be rinsed off with plenty of water. Protective eyeglasses must be used.

Conclusion

Two inquiry-based experiments are described that complement a previously published synthesis of zinc iodide *(1).* In the first experiment, students draw upon their knowledge of solubility and molarity to pose an alternative synthesis of zinc iodide using a double replacement reaction. Students then conduct a comparative analysis in terms of percent yield, efficiency, safety, and cost, and discover that the zinc iodide synthesized from its elements is the preferred reaction. In the second activity, students identify zinc hydroxide by making predictions, referring to the literature, and conducting qualitative tests.

These activities have been used by introductory chemistry students as well as high school chemistry teachers enrolled in our graduate science education program. Those who have used the experiments in their classes report that their chemistry students enjoyed making predictions and "discovering" the intended outcomes as opposed to verifying what is taught in lecture. Teachers have indicated that students commonly draw upon their previous knowledge of zinc and iodine to understand these two new activities. While this is not a formal assessment, this anecdotal information stressing the importance of prior knowledge and inquiry supports a constructivist view of learning in science.

[W]Supplemental Material

Written materials used by students (procedure, qualitative tests, a postlab checklist, pre- and postlab questions), as well as instructor information (comments, list of equipment and chemicals, chemical preparation instructions, and answers to questions posed in the activity) are available in this issue of *JCE Online*.

Notes

1. Yields greater than 100% are most likely the result of the incomplete evaporation of water during the drying process or the absorption of water from the atmosphere after drying.
2. Aldrich Chemical Co. does not sell a standard of zinc hydroxide. One can be prepared if necessary.

Literature Cited

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