Synthesis & Purification of Lead Iodide

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Purpose:

Chemical reactions are preformed allowing the identification of the limiting and excess reactants in a chemical reaction. Different methods of product purification are used to assist in this identification, showing the benefits and costs of each.

Procedure Citation:

Please refer to: "Synthesis and Purification of Lead Iodide." Chemistry 1210: General Chemistry Laboratory Manual, Hayden-McNeil, Plymouth, 2019, pp. 29-33 for the proper procedure.

Presentation of Data

Data Preparation

Reproduce your data preparation section here, including your calculations. These are the calculations you did in lab on Day 1.

Target mass of PbI_2, g	0.0525 g
Stoichiometric mass of $Pb(NO_3)_2$ needed, g	0.0378 g
Stoichiometric mass of KI needed, g	0.0378 g

Part A. Mass Reactants (Day 1)

Mass of $Pb(NO_3)_2$, g	0.0370 g
Mass of KI, g	0.0360 g
Moles of $Pb(NO_3)_2$	0.000112 moles
Moles of KI	0.000217 moles

Part C. Purification via vacuum filtration

Vacuum Filtration (Day 1)

Mass of dry filter paper, g	0.2783 g
Mass of weigh boat, g	2.8061 g
Mass of dry filter paper + weigh boat, g	3.0844 g
Mass of weigh boat + filter paper + "wet" sample, g	3.5932 g
Mass of "wet" sample, g	0.5088 g

Part E. Mass Reactants (Day 2)

Mass of $Pb(NO_3)_2$, g	0.0374 g
Mass of KI, g	0.0366 g
Moles of $Pb(NO_3)_2$	0.000113 moles
Moles of KI	0.000220 moles
Mass of casserole dish, g	54.9714 g
Mass of watch glass, g	55.8448 g

Part G. Purification via evaporative collection

Mass of casserole dish + collected sample, g	55.0293 g
Mass of casserole + collected sample, g	55.8463 g
Mass of sample, g	0.0594 g

Limiting Reactant (highlight one): $Pb(NO_3)_2$ <mark>KI</mark>

Theoretical Yield, g	0.0508 g
% Yield	116.8%

Part H. Weigh Dried Filter Paper from Day 1

Mass of dry filter paper + weight boat + "dry" sample, g	3.1128 g
Mass of "dry" sample, g	0.0284 g

Limiting Reactant (highlight one): $Pb(NO_3)_2$ <mark>KI</mark>

Actual yield	0.0284 g
Theoretical Yield	0.050
% Yield	57%

Sample Calculations

Show an example for all types of calculations; the following are graded for correctness: moles of each reagent, actual yield of product, theoretical yield of product, percent yield of product.

$$0.0370 \, \cancel{g \, Pb(NO_3)_2} \times \frac{1 \, mol \, Pb(NO_3)_2}{331.22 \, \cancel{g \, Pb(NO_3)_2}} = 0.000112 \, mol \, Pb(NO_3)_2$$

$$0.0360 \, \cancel{g \, KI} \times \frac{1 \, mol \, KI}{166.00 \, \cancel{g \, KI}} = 0.000217 \, mol \, KI$$

Y_a = (mass of casserole dish and collected sample,g – mass of casserole dish,g)

+ (mass of watch glass and collected sample,g – mass of watch glass,g)

$$Y_a = (55.0293g - 54.9714g) + (55.8463g - 55.8448g)$$

$$Y_a = 0.0594 \, g \, PbI_2$$

$$Y_T = 0.0366 \, \cancel{g \, KI} \times \frac{1 \, \cancel{mol \, KI}}{166.00 \, \cancel{g \, KI}} \times \frac{1 \, \cancel{mol \, PbI_2}}{2 \, \cancel{mol \, KI}} \times \frac{461.00 \, g \, PbI_2}{1 \, \cancel{mol \, PbI_2}} = 0.0508 \, g \, PbI_2$$

$$percent \, yield \, (\%) = \frac{actual \, yield \, (Y_a)}{theoretical \, yield \, (Y_T)} \times 100$$

$$percent \, yield \, (\%) = \frac{0.0594 \, \cancel{g \, PbI_2}}{0.0508 \, \cancel{g \, PbI_2}} \times 100 = 116.8\%$$

Discussion:

The lab procedure occurred through two days, each day performing different methods of synthesizing and purifying lead iodide. The first day began with mixing the samples of potassium iodide and lead (ii) nitrate with distilled water, and heating that solution followed by cooling it. This caused lead iodide to collect at the bottom of the solution as a precipitate and the potassium nitrate to dissociate. In order to collect the lead iodide precipitate, a vacuum filtration apparatus was created, and the solution was poured onto the filter paper, in the Büchner funnel, placed on top of a flask. This allowed for the collection of lead iodide. By weighing the filter paper with the leftover sample on it we were able to calculate the amount of lead iodide in the solution and calculate the limiting reactant (potassium iodide) as well as percent yield.

The second day, the potassium iodide and lead (ii) nitrate were mixed together again with distilled water, and placed in a casserole dish. This casserole dish was put over a Bunsen burner with a watch glass over it, to protect the precipitate from evaporating out. Through this process the water evaporated out, but the potassium nitrate was not separated from the lead iodide. By weighing the casserole dish and watch glass beforehand, we were able to find the weight of the resulting sample, allowing the identification of the limiting reactant (potassium iodide) and the ability to calculate the percent yield.

From finding the actual and theoretical yields, derived by the measured mass of potassium iodide and the mass of lead iodide samples, the percent yields were calculated. The percent yield of the vacuum filtration is 57%, and the percent yield of the evaporative collection is 116.8%. The percent yield of the vacuum filtration is smaller than the percent yield of the evaporative collection because the sample collected by the evaporative collection was not pure lead iodide, it was lead iodide and potassium nitrate. While the vacuum filtration's sample had only pure lead iodide. It was also found that the limiting reactant in both reactions was potassium iodide.

In a reaction the limiting reactant is the reactant that limits more product from forming. In both of my reactions the limiting reactant was found to be potassium iodide. Although I had the same limiting reactants for both methods, it is possible for my reactions to have different limiting reactants in each. This could happen by human error such as by measuring the reactants to the incorrect masses.

In the evaporative collection method the sample collected was not pure lead iodide. Only the water evaporated in the solution, leaving the lead iodide and the potassium nitrate behind in the end. On the other hand, since in the vacuum filtration the potassium nitrate dissociated allowing it to be aqueous. Consequently it was able to leak through the filter paper, leaving the only solid lead iodide behind. In comparison to this in the evaporative cooling there possibly was some water left behind, potassium nitrate, and lead iodide. In both of these procedures, only in the evaporative cooling were we able to achieve a over 75% yield. In evaporative cooling we were able to achieve this goal because there was much more than lead iodide in the sample, such as

potassium nitrate, and even possibly some water particles. On the other hand, in the vacuum filtration the solution was transferred and mixed with a stir rod, leaving the chance for some on the sample to be lost on glassware. This could contribute to the smaller percent yield.

In the end in looking at these two different procedures, I would recommend the vacuum filtration to the head engineer. I would do this because in the end they were looking for the synthesis and purification of lead iodide, and although some of the lead iodide was lost in the vacuum filtration, the end sample was pure lead iodide unlike the evaporative cooling sample that had different particles in it. I would also want to notify the engineer of potential error in the experiment. Systematic errors in the vacuum filtration are related to loss of the sample, such as some of the sample being left on the glass stir rod or the inner edges of the Büchner funnel. As a result, these parts of the sample will never end up on the filter paper, causing a lower percent yield. Systematic errors to improve on in the evaporative cooling could be related to the small water droplets forming on the top of the watch glass, causing the sample collected below to burn while the droplet sat on the top. This causes not all the water to be evaporated and puts the sample at risk. Although both procedures have their systematic errors to be improved on, I would still recommend the vacuum filtration to the engineer, especially since those errors can be fixed with careful attention.

Conclusion:

The different methods of product purification each have their own benefits and costs, but each allowed the identification the limiting reactants in the chemical reaction. Purification of a product can be reached by methods of vacuum filtration or by evaporative cooling. Although the end sample of vacuum filtration may be smaller by error, the sample is more pure lead iodide than the sample collected by evaporative cooling. As a result the synthesis and purification of a product is more precise by vacuum filtration than by evaporative cooling.

Report Questions: Observation Tables

Record your observations for Day 1 below:

Part A.

Table 3.1 Observations.

Solid Pb(NO₃)₂	Solid KI
- Solution turned completely yellow with mixing. - Precipitate is at bottom of solution.	- Solution turned completely yellow with mixing - Precipitate is at bottom of solution.

Part B.

Table 3.2 Observations.

Observations During Heating	Observations During Cooling
- Solution is no longer completely yellow. - Precipitate has collected at the bottom of the solution, and is yellow.	- Precipitate is completely dissolved. - In ice bath precipitate is beginning to form on the bottom of the test tube.

Table 3.3 Observations.

Observations of Filter Paper
- Sample is a sparkly/yellow/gold color. - There are small grains of the precipitate.

Record your observations for Day 2 below:

Part F.

Table 3.4 Observations.

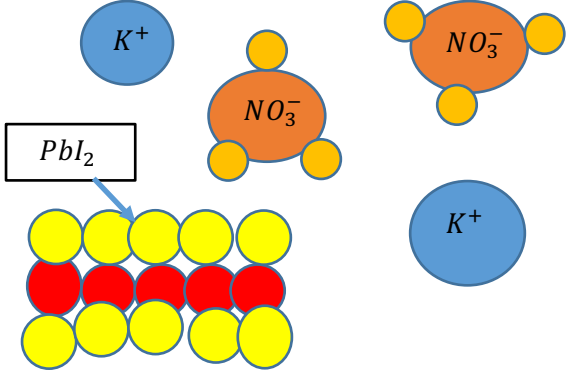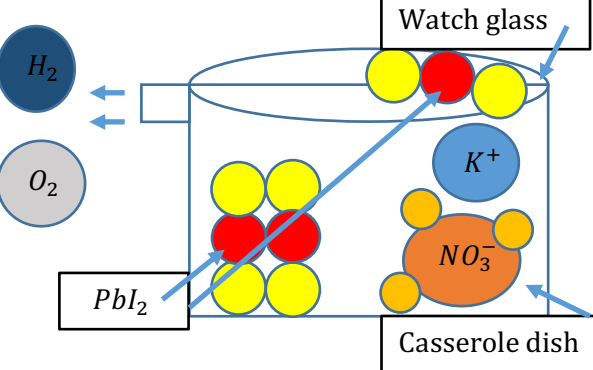| Observations of Casserole Dish |
|---|
| - Sample has a bright yellow color.
- The precipitate is visible. |

Part G.

Table 3.5 Observations.

Observations During Heating	Observations During Cooling
- Color of sample is gone, no longer yellow. - The precipitate is collecting on the watch glass.	- There is solid precipitate on the watch glass and on sides of the casserole dish. - The precipitate is a sparkly orange/yellow color.

Report Questions: Particle Diagrams

A particle diagram is a symbolic representation of atoms and molecules at the microscopic level. Particle diagrams for all listed scenarios are required. TAs are looking for an accurate representation of the state and identity of particles present.

Draw particle diagrams to represent what the following stages in your procedure would look like on a particle level:

In your Test Tube before purification	In the casserole dish after purification (evaporative collection)

In your Buchner funnel after purification (vacuum filtration)	In your side-arm flask after purification (vacuum filtration)