EXPERIMENT 15

Percentage Yield of Lead (II) Iodide in a Gravimetric Analysis

INTRODUCTION

In a gravimetric analysis, a substance is treated so that the component of interest is separated either in its pure form or as a constituent in a compound of known composition, which can be weighed. The separation is often accomplished by first dissolving a sample of the substance in a suitable solvent and then adding a reagent, which reacts with the desired component to form an insoluble solid.

A gravimetric precipitation method must fulfill certain basic requirements in order to provide an accurate analysis.

1. The precipitation reaction should be quantitative so that only a negligible fraction of the component of interest remains in solution after the precipitate is isolated. A moderate excess of the precipitating agent is commonly employed to help achieve this condition.

2. Other constituents in the sample solution should not interfere with the precipitation of the component of interest.

3. The precipitation should be as free of contaminants as possible.

4. The particles of precipitate should be large enough to filter easily and to facilitate the removal of soluble impurities during washing operations.

5. The precipitate should easily be converted to a form suitable for weighing. The conversion may require only a drying operation to remove residual moisture, or, if the precipitate has an indefinite chemical composition, an ignition to produce a solid with a definite composition.

6. In addition to a definite composition, the weighing form should have: stability at the relatively high temperatures required for the drying or ignition operations, little or no tendency to react with substances in the air, and a molar mass significantly larger than that of the compound of interest.

A gravimetric analysis for potassium iodide features the use of the reagent lead (II) nitrate to bring about the precipitation of lead (II) iodide from an aqueous solution containing a known mass of sample. The precipitate reaction is

\[ 2KI (aq) + Pb(NO_3)_2 (aq) \rightarrow PbI_2 (s) + 2KNO_3 (aq) \]

This reaction satisfies the important criteria for a satisfactory gravimetric method: namely, a quantitative reaction yielding a product of definite composition, essentially free from impurities. The lead (II) iodide is isolated by filtration, washed and dried to a constant mass.
The following are the major techniques that are used in a gravimetric analysis.

1. **PRECIPITATION.** The heart of any gravimetric analysis is the precipitation reaction. In most cases the reaction involves the formation of an ionic solid due to cation-anion combination during the mixing of precipitating agent and sample solutions.

2. **DIGESTION.** A precipitate is seldom ready for filtration immediately after it is formed, because its particles are generally small and retain an unnecessarily large amount of impurities. In order to minimize both these problems, the precipitate is allowed to remain in contact with the liquid phase for a period of time prior to being filtered. The process is known as digestion. An increase in particle size is observed during a digestion, and is attributed to two processes: the dissolution of the very small, more soluble particles to yield ions, which then reprecipitate on the surface of the larger crystals, and the coagulation of small particles to form aggregates. As digestion takes place, impurities adsorbed on the surface of the precipitate particles tend to dissolve. Although the impurities may eventually be readsorbed, the decrease in precipitate surface area that goes along with larger particles lessens the chance of adsorption. Thus, digestion yields purer as well as larger, more easily filtered particles.

3. **FILTRATION.** The liquid that is in contact with the precipitate is called the supernatant. To separate the precipitate from the supernatant liquid, the mixture is passed through filter paper in a funnel. The precipitate is trapped by the filter paper, and the liquid passes through. The liquid that has passed through the filter paper is called the filtrate.

4. **WASHING THE PRECIPITATE.** After the removal of the supernatant liquid, the small amount of solution that remains along with a precipitate may serve as a source of contamination in later operations. In addition, foreign ions are likely to be absorbed by a precipitate as it forms. To remove these contaminants, the precipitate is washed. Although the wash liquid is often deionized water, a very dilute solution of the original precipitating agent can also be used. A precipitate can also be washed with acetone to remove water from the precipitate in order to decrease the drying time.

5. **DRYING.** Solid materials must be dried to remove moisture before they are weighed. This operation is usually performed in an oven set at 110°C.

**PROCEDURE**

1. Except for the laboratory handout, remove all books, purses, and such items from the laboratory bench top, and placed them in the storage area by the front door. For laboratory experiments you should be wearing closed-toe shoes. Tie back long hair, and do not wear long, dangling jewelry or clothes with loose and baggy sleeves. Open you lab locker. Put on your safety goggles, your lab coat, and gloves.

2. Bring a 250-mL beaker and your microspatula to a milligram balance. Place the beaker on the milligram balance. “Tare” the beaker by pressing one of the keys with the symbol "\(\rightarrow T/0\)". This will zero out the mass of the beaker.

3. Transfer a small amount of potassium iodide from its reagent bottle to a weighing cup or a glass or porcelain container by pouring.
   **CAUTION:** Never place your microspatula or scoopula into a reagent bottle.
4. Remove the beaker from the balance chamber. Add between 0.4 and 0.6 grams of potassium iodide to the beaker, place it back on the balance, read the exact mass, and record it in your Data Table.

**CAUTION:** Never pour solid or liquid reagents back into stock bottles. Any excess chemicals can be given to another student or should be properly discarded in the waste bottle in fume hood A.

**NOTE:** If any potassium iodide is spilled on the balance or on the lab bench, clean them up immediately, and dispose of it in the waste bottle in fume hood A. If there are any crystals left on the balance or the lab bench at the end of the lab period, the instructor will deduct one point from everyone’s lab score as a charge for cleaning up after you.

5. Using a 150-mL beaker obtain 50 mL of lead (II) nitrate from the pump bottle on the lab bench in front of the instructor’s desk. Hold the beaker under the spout of the pump bottle, pull the top of the pump up as high as it will go, and gently push it down as far as it will go. If it is set at 50.0 mL, it will deliver 50.0 mL to your beaker. Using your 100-mL graduated cylinder measure out 25 mL of the lead (II) nitrate solution, then slowly and while stirring, add the solution to your 250-mL beaker containing the potassium iodide solution.

6. In order to prepare the precipitate for filtration, it must be digested. Place the beaker on a ceramic-centered wire gauze, supported by an iron ring attached to a ring stand. Heat the mixture, with frequent stirring, for about 5 minutes. Keep the liquid just under is boiling point. Do not allow any of the precipitate to leave the beaker on your stirring rod. Rinse any precipitate adhering to your stirring rod back into the beaker with a stream of deionized water from your wash bottle before setting the stirring rod down.

**CAUTION:** Do not let the liquid boil, boiling may cause the mixture to “bump”.

At the end of the 5 minutes, turn off the burner and cease the frequent stirring. Set the beaker on a hot pad and allow the mixture to air cool for at least 5 minutes.

7. Obtain a clean 600-mL beaker and add about 200 mL of ice to create an ice bath. Set the 600-mL beaker on your lab bench, place the 250-mL beaker with your solution and precipitate on top of the ice in the 600-mL beaker, and let it cool for 10 minutes. This will allow the precipitate to digest. During the digestion, the precipitate coagulates appreciably and should settle to the bottom of the container, leaving a nearly clear supernatant solution.

8. While the solution is cooling, obtain a 12.5-cm disk of Whatman Grade No. 1 filter paper (medium porosity, medium flow rate, 11 μm particle retention) from the back counter or drawer 014. Fold the disk of filter paper as shown below, and determine its mass on the same milligram balance.
9. Support a glass funnel in a clay triangle on an iron ring, attached to a ring stand. Place a clean 400-mL beaker below the funnel such that the stem of the funnel is touching the inside of the 400-mL beaker. Insert the filter paper cone into the glass funnel and moisten the filter paper with water so it adheres to the funnel.

10. Without disturbing the precipitate, carefully pour off the supernatant liquid into the filter paper. Use a stirring rod to guide the flow. Do not let the supernatant liquid level come within 1 cm of the top of the filter paper, otherwise precipitate particles may pass through into the filtrate. Rinse the remainder of the precipitate into the filter paper with a stream of water projected from your deionized water wash bottle. Scrape the beaker with the rubber policeman and rinse the residue it has collected into the filter paper with your wash bottle.

11. The filtrate collected in the 400-mL beaker should be clear. If the filtrate contains solid particles, place another beaker below the glass funnel, and pour the filtrate through the filter paper again. Once the filtrate is clear, rinse the collected precipitate and filter paper with 3 10-mL portions of acetone from an acetone bottle found in Fume Hood B.

12. Remove filter paper from the funnel, set it in a beaker labeled with your name, and place it in the drying oven for 75 minutes. After this time, remove the beaker from the oven with beaker tongs, allow the filter paper to cool, and determine the mass of the filter paper and precipitate on the same milligram balance.

13. Pour all of the filtrate into the bottle for Liquid Waste in Fume Hood A. After the filter paper and precipitate have been weighed, place them in the bag for Solid Waste in Fume Hood A.

14. Clean and wipe dry your laboratory work area and all apparatus. When you have completed your lab report have the instructor inspect your working area. Once your working area has been checked your lab report can then be turned in to the instructor.
DATA TABLE

<table>
<thead>
<tr>
<th>Mass of Potassium Iodide</th>
<th>.</th>
<th>g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of Filter Paper</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>Mass of Filter Paper and Lead (II) Iodide</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>1 Mass of Lead (II) Iodide Collected</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>2 Theoretical Yield of Lead (II) Iodide</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>3 Percent Yield of Lead (II) Iodide</td>
<td>.</td>
<td>%</td>
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</tbody>
</table>

OBSERVATIONS

CALCULATIONS

1.
POSTLAB QUESTIONS

1. If a 0.625 g sample of magnesium chloride is dissolved in water and then treated with a silver nitrate solution, how many grams of silver chloride precipitate should be collected? *Box your answer.*
2. A 1.103 g sample of sodium fluoride is dissolved in water, and then a precipitate of calcium fluoride is produced by adding a calcium nitrate solution. If the dried calcium fluoride precipitate has a mass of 0.947 g, what is the percent yield? *Box your answer.*