Experiment 4: Gravimetry

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Purpose

The purpose of this experiment is to determine the percent yield of the reaction between barium nitrate ($Ba(NO_3)_2$) and sulfamic acid (NH_2SO_3H) that yields solid barium sulfate ($BaSO_4$).

Background

Sulfamic acid (NH_2SO_3H) is a very common chemical used to remove grout and mortar haze, as well as rust and mineral deposits [1]. It can be found in many home improvement retail stores [2]. The hydrolysis of sulfamic acid to give sulfates can be used to precipitate barium in the form of barium sulfate [3].

$$Ba(NO_3)_{2(aq)} + NH_2SO_3H_{(aq)} + H_2O_{(l)} \rightarrow BaSO_{4(s)} + NH_4NO_{3(aq)} + HNO_{3(aq)}$$

In this experiment, you will react sulfamic acid with barium nitrate in hot water to produce a precipitate of barium sulfate. The solid material will be isolated by gravity filtration. When the barium nitrate is the limiting reactant and the amount of barium nitrate used is known, that information, along with the mass of precipitate isolated, will allow for the percent yield to be calculated. The advantage of using sulfamic acid instead of sulfuric acid, is that sulfamic acid produces a coarse, crystalline precipitate with fewer impurities [4].

Reaction yield

Stoichiometric calculations can predict the amount of product that should be formed in a chemical reaction. However, chemical reactions are not perfect, and often times reactions do not proceed to full completion, and the amount of product predicted is not obtained in the experiment. The *yield* of a chemical reaction refers to the amount of product actually obtained in the experiment with respect to the expected quantity calculated with stoichiometric calculations.

%Yield = ((Actual amount obtained in lab) / (Expected amount from calculations))x100%

Example Calculation:

We mix 2.0 moles of $Ba(NO_3)_2$ with an excess of NH_2SO_3H to produce a $BaSO_4$ precipitate. We isolate only 1.0 mole of $BaSO_4$ in the lab.

$$Ba(NO_3)_{2(aq)} + NH_2SO_3H_{(aq)} + H_2O_{(l)} \rightarrow BaSO_{4(s)} + NH_4NO_{3(aq)} + HNO_{3(aq)}$$

Calculate the reaction yield.

Answer: According to stoichiometric calculations, 2 moles of $Ba(NO_3)_2$ should produce 2 moles of $BaSO_4$. However, the actual numbers of moles of $BaSO_4$ obtained in lab is 1.0 mole. Hence the yield is

%Yield = ((1.0 mole obtained in lab) / (2.0 moles expected from calculations))x100%

%Yield = 50%

Limiting Reactant

In a chemical reaction, the reaction stops when one of the reactants is completely used. This reactant is referred to as the limiting reactant, and this reactant limits the amount of product generated. After all limiting reactant has reacted, some of the other reactant will remain unused. Typically, one needs to do some basic calculations to identify the limiting reactant.

Filtration

Filtration is a technique employed in chemistry to separate a solid compound from the liquid. The mixture is poured onto a filter and gravity makes the liquid go through the filter, while the solid remains on the filter. When the mixture containing the solid is very hot, we call the filtration procedure "hot gravity filtration". Filter paper must be folded before proceeding with the filtration, forming a cone. In order to do this one needs to first fold the filter in half, and then in half again, as shown in Figure 1. Do not press the tip of the cone while folding, because it will weaken the paper.

Figure 1

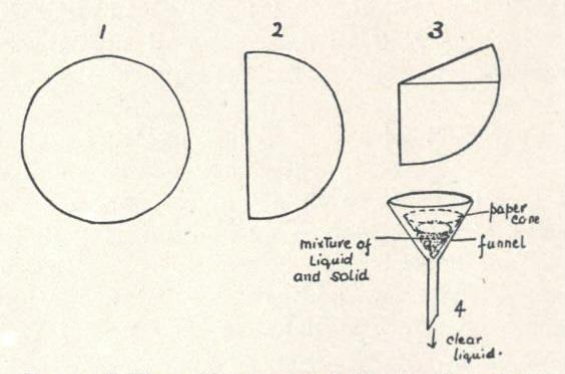

FIG. 4.—Folding a filter paper and filtration through it.

https://en.wikipedia.org/wiki/File:Paper_filter_folding_and_filtration.JPG

Chemicals

(*Barium compounds are toxic. Make sure you wear gloves and wash your hands after handling the barium solids and solution.)*

0.060 M $Ba(NO_3)_2$ solid NH_2SO_3H

Equipment

250 mL beaker
50 mL or 100 mL graduated cylinder
Stirring rod
Filter paper and funnel
250 mL Erlenmeyer flask (to support the funnel)
400 mL beaker (for the ice bath)
Tongs

Procedure
The Reaction
(1) You will need 2.3 g to 2.5 g NH_2SO_3H. This reactant is located near the lab balance.

(2) Bring your lab notebook and a clean, dry 250 mL beaker to the balance.

(3) Tare a piece of weighing paper and weigh out 2.5 _ _ g of NH_2SO_3H. Record the exact mass in your notebook, and then transfer all of this sulfamic acid into the 250 mL beaker.

Return to your lab bench to do the following steps.

(4) Use a 100 mL or 50 mL graduated cylinder to measure 100.0 mL of the 0.060 M barium nitrate solution. Transfer all of this barium solution into the 250 mL beaker that contains the sulfamic acid.

(5) Add 25 mL of deionized water to the graduated cylinder as a rinse, and add this to the beaker. Mix with a glass stirring rod.

(6) **IN THE HOOD**, use a hotplate to heat the solution to boiling. Allow the solution to boil GENTLY for approximately 30 minutes. Cover the beaker with a watch-glass, and add small amounts of deionized water to make sure the amount of liquid does not change much. Add small amounts of the water so you don't stop the boiling. Stir the solution occasionally. Do not lose sample on the glass stir rod.

(7) Use the 250 Erlenmeyer flask to support the funnel for the filtration.

(8) After 30 minutes of boiling, turn off the hot plate. Use several layers of paper towel to make potholders so you can remove the beaker from the hotplate and onto your lab bench. When the beaker has cooled a bit, place the very warm beaker into an ice bath. Be careful not to contaminate your reaction mixture with the ice water. Cool the beaker to room temperature.

(9) Obtain a piece of filter paper, write you name on it with pencil, and then record its mass.

(10) When the reaction mixture containing the precipitate is cooled, proceed to filter the mixture. Make sure no trace of precipitate remains in the beaker. Use a wash bottle to rinse any remaining product out of the beaker and onto the filter paper.

(11) Transfer the filter paper and its contents to a watch glass. Make sure the precipitate does not fall off of the filter paper.

(12) Place the watch glass with filter paper in an oven at 105°C for 15 minutes.

(13) Remove the dry filter paper with dry precipitate from the oven after the 15 minutes. Be careful not to burn yourself. Put the hot watch glass on the lab bench, and slide the filter paper and precipitate onto the lab bench to cool. Once the filter paper and precipitate are cool, weigh them and record the mass into your notebook. Then return the filter paper with precipitate back to the watch glass.

(14) Return the filter paper/precipitate and watch glass to the hot over for anther 15 minutes. Then repeat step 13 to find the mass of the filter paper and precipitate. Record this mass in your notebook. Use this second mass for your calculations. We are assuming this second mass is the dry precipitate. Was this second mass much less than the first 'dry' mass?

(15) Calculate the % Yield.

a) Convert the liters of barium nitrate used to moles of barium nitrate. (M x L=moles)
b) Convert the mass of sulfamic acid to moles of sulfamic acid. (g / g/mole = mole)
c) Confirm that barium nitrate is the limiting reactant. Determine the amount of excess sulfamic acid remaining after the reaction stopped, assume all of the barium nitrate was used. (moles start – moles used = moles left over)
d) Base your calculations on the number of moles of limiting reactant, barium nitrate, that was used. How many moles of barium sulfate should have been made, based on the moles of barium nitrate used? (1:1 ratio)
e) Convert the moles of barium sulfate you should have made to grams. (mole x g/mole = g)
f) Calculate the % Yield, using the grams of barium sulfate precipitate obtained in lab and the grams of barium sulfate you expected to make *(calculation step e)*.

References

(1) pubchem., https://pubchem.ncbi.nlm.nih.gov, Accessed: 2017-04-22.
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(3) Wagner,W. F.;Wuellner, J. A. *Analytical Chemistry* **1952**, *6*, 1031–1032.
(4) Notley, J.M. *Journal of Applied Chemistry and Biotechnology* **1973**, *10*, 717–723.

Procedure