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Experiment #7 – Preparation of an Alum

Laboratory Overview

CHEM 1361

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Learning Objectives

• Learn the technique of gravity filtration

• Learn the technique of vacuum filtration

• Convert between grams and moles

• Convert between molarity and volume of solution and moles

• Identify the limiting reactant in a chemical reaction

• Calculate theoretical yield in a chemical reaction

• Calculate % yield from an experiment
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• Basics of Stoichiometry

• Synthesis of an Alum

• Percent Yield in a Chemical Reaction

• Technique Notes
Basics of Stoichiometry

Stoichiometry refers to the study and application of the quantitative relationships between reactants and products in chemical reactions.

The stoichiometric coefficients in a balanced chemical equation give the mole relationships between the reactants and products. For example, consider the reaction:

$$\text{N}_2 (g) + 3 \text{H}_2 (g) \rightarrow 2 \text{NH}_3 (g)$$

This equation says that 1 mol of N\textsubscript{2} and 3 mol of H\textsubscript{2} will react to form 2 mol of NH\textsubscript{3} which provides the stoichiometric relationship between reactants and products.

Based on this relationship, one can predict the number of mol of NH\textsubscript{3} formed from different numbers mol of N\textsubscript{2}. For example, if 5 mol of N\textsubscript{2} were completely reacted $5 \times 2$ mol = 10 mol NH\textsubscript{3} would be formed.
Basics of Stoichiometry (continued)

Direct measurement of the number of mol of a substance is virtually impossible unless you want to count atoms and molecules. Relationships have been developed between moles and several laboratory measuring techniques. Examples of methods for finding the number of mol of a substance include, but are not limited to:

\[
\text{\#mol} = \frac{\text{\#g of substance}}{\text{molar mass}}
\]

\[
\text{\#mol} = \text{Molarity} \times \text{\#Liters solution}
\]

where molar mass is the sum of the atomic weights in the formula for an element or compound and molarity is an expression for solution concentration that is defined as the \# mol of solute/\# L solution.
Synthesis of Alum

In this experiment you will synthesize an alum, a double salt of the general formula:

\[ M^{1+}M^{3+}(SO_4)_2 \cdot nH_2O \]

where the letter M represents metal cations and the n may be any integral number of water molecules. Such a molecule is called a hydrate due to the water molecules associated with the structure.

In this experiment you will synthesize the alum:

\[ KAl(SO_4)_2 \cdot 12H_2O \]
Synthesis of Alum (continued)

The laboratory book carefully outlines the detailed steps in the synthesis. The overall reaction may be written as (Equation 7.6 in the lab book):

\[
2 \text{Al(s)} + 2 \text{KOH(aq)} + 4 \text{H}_2\text{SO}_4(aq) + 22 \text{H}_2\text{O}(\ell) \rightarrow 2 \text{KAl(SO}_4)_2 \cdot 12\text{H}_2\text{O(s)} + 3 \text{H}_2(\text{g})
\]

Though the equation may appear to be a little daunting, it has reactants and products just like any other chemical equation. The coefficients give the mole relationships between reactants and products. For example, 2 mol of Al will lead to 2 mol of alum, 0.5 mol of Al will lead to 0.5 mol of alum, etc., if there is sufficient of all of the other reactants.

Since you will mix several reactants together to form the alum, it is important to identify the limiting reactant to determine the theoretical yield of alum.
Identification of Limiting Reactant

In class you will certainly cover different approaches for addressing limiting reactant problems. The following is more conceptual than calculational.

\[2 \text{Al}(s) + 2 \text{KOH(aq)} + 4 \text{H}_2\text{SO}_4(aq) + 22 \text{H}_2\text{O}(\ell) \rightarrow 2 \text{KAl(SO}_4)_2\cdot12\text{H}_2\text{O}(s) + 3 \text{H}_2(g)\]

Consider the following possible scenarios:

<table>
<thead>
<tr>
<th>If you had this many mol of Al</th>
<th>You would need this many mol of each reactant</th>
<th>And would form this many mol of each product</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>KOH</td>
<td>H$_2$SO$_4$</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>100</td>
<td>100</td>
<td>200</td>
</tr>
</tbody>
</table>

If you don’t have sufficient mol of any of the reactants indicated above, Al will not be the limiting reactant. In this experiment, you will need to decide the limiting reactant based on your starting materials.
Identification of Limiting Reactant (continued)

Water is by far in excess in this reaction so we don’t have to worry about running out of it. The question then becomes which of the other three reactants will be the one that limits the reaction?

\[
2 \text{Al(s)} + 2 \text{KOH(aq)} + 4 \text{H}_2\text{SO}_4(\text{aq}) + 22 \text{H}_2\text{O}(\ell) \rightarrow 2 \text{KAl(SO}_4)_2 \cdot 12\text{H}_2\text{O(s)} + 3 \text{H}_2(\text{g})
\]

Two of the substances, Al and KOH, will be weighed out as solids so the number of moles of each may be determined by dividing the number of grams used by the respective molar masses. Sulfuric acid, \(\text{H}_2\text{SO}_4(\text{aq})\), is acquired as an aqueous solution. The number of moles of sulfuric acid may be determined by multiplying the molarity of the solution by the number of liters used.
You will use two types of filtration in this experiment:

Gravity filtration – Mixture is passed through filter paper through the influence of gravity. Used to separate solids and suspended material from the mixture.

Vacuum filtration – Mixture is drawn through filter paper by pulling a vacuum in the receiving flask. In this experiment used to help dry your final product.

A brief overview of these techniques may be found on the following pages.
Gravity Filtration

The trickiest part of a gravity filtration is folding the filter paper before placing it in the funnel. The sequence below shows the folding process.

Step 1. Fold a piece of filter paper in half.

Step 2. Fold the semicircle not quite in half again.

This poorly drawn black line represents the edge of the folded-over paper.
Folding Filter Paper - continued

Step 3. Tear off a small piece of the corner that has been folded short.

Step 4. Place the folded filter paper in the funnel with the three thicknesses including the torn edge on one side and only one thickness of paper on the other. Wet with water to secure it in the funnel.
Vacuum Filtration

The filter paper here is not folded at all. It is simply placed whole in the bottom of a Büchner funnel. The sink faucets have devices called aspirators on them as a source of vacuum. Simply hook a hose up between the aspirator and the suction flask and it will provide a vacuum. Refer to the figure below.

The reality is you may have to leave your alum air drying in your drawer until the next week – this likely won’t dry it completely.