

Limiting Reagent – Synthesis of Aspirin

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Aspirin (acetylsalicylic acid) is the most common medicinal drug in use today. Aspirin is an analgesic (pain reliever), an antipyretic (fever reducer), and anti-inflammatory medicine. While aspirin has many benefits, there are side effects, which include dizziness, nausea, upset stomach, and bleeding of the stomach.

Aspirin is produced by the esterification of salicylic acid. An ester is an organic functional group with the following structure:

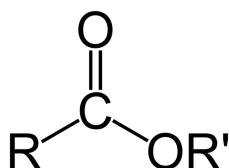


Figure 1 – Ester functional group¹

The R and R' represent different organic segments, they may be simple alkyl chains (carbon and hydrogen) or more complex structures. The reaction to produce aspirin occurs in a one to one ratio between salicylic acid and acetic anhydride according to the following reaction:

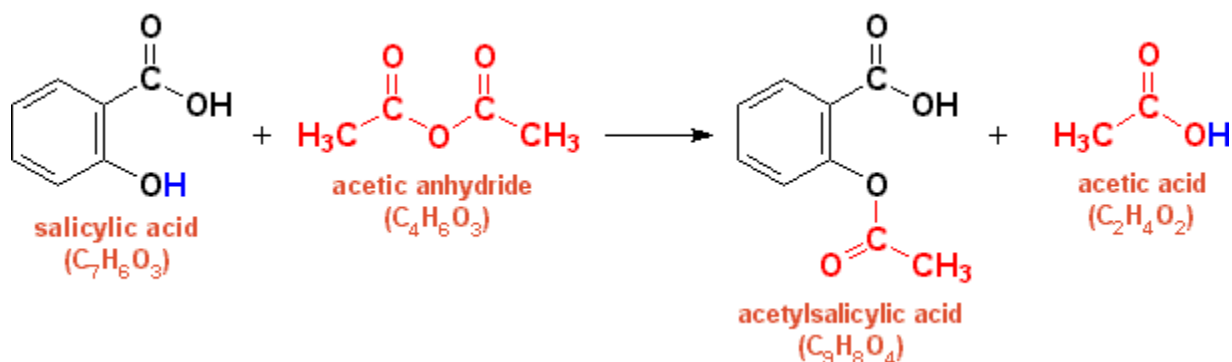


Figure 2 – Synthesis of aspirin²

In this experiment you will measure the amount of aspirin produced and calculate the percent yield.

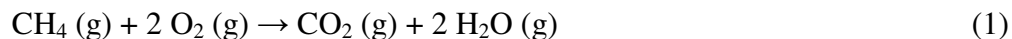
Chemical reactions will continue as long as there are reactants present. As soon as a reactant is consumed the reaction will stop, no matter how much of the other reactants remaining. The reactant that runs out first is called the limiting reagent. The term limiting reagent refers to the fact that the amount of product that can be produced is dependant upon that chemical, thus it

¹ Image source, <http://upload.wikimedia.org/wikipedia/commons/thumb/f/f6/Ester-general.png/781px-Ester-general.png>, accessed September 14, 2007.

² Image source, <http://www.chemheritage.org/educationalservices/pharm/asp/images/asp3102.gif>, accessed September 14, 2007.

limits the reaction. The reactants that are not entirely consumed are referred to as excess reagents.

Given the combustion reaction that occurs between methane and oxygen:



The above reaction can be read in two ways, either in terms of atoms/ molecules or in terms of moles. The more practical approach is to read the reaction in terms of moles, since we seldom deal with individual atoms or molecules in the lab. Thus the above reaction could be read as one mole of methane reacts with two moles of oxygen and forms one mole of carbon dioxide and two moles of water. In order to determine the limiting reagent it is necessary to determine the number of moles of all reactants present.

Example 1: If 12.0 g of methane reacts with 40.0 g of oxygen, which reactant is the limiting reagent? How much product would be formed? How much excess reagent would be left?

Step 1: Determine the moles present of each reactant

$$12.0 \text{ g CH}_4 \left(\frac{1 \text{ mol}}{16.0 \text{ g}} \right) = 0.750 \text{ mol CH}_4 \quad (2)$$

$$40.0 \text{ g O}_2 \left(\frac{1 \text{ mol}}{32.0 \text{ g}} \right) = 1.25 \text{ mol O}_2 \quad (3)$$

Step 2: Determine the amount of oxygen needed to completely react with 0.750 mol of methane. Multiply by the molar ratio, the values in the ratio come from the coefficients in the reactant.

$$0.750 \text{ mol CH}_4 \left(\frac{2 \text{ mol O}_2}{1 \text{ mol CH}_4} \right) = 1.50 \text{ mol O}_2 \text{ needed} \quad (4)$$

This calculation indicates that 1.50 moles of oxygen are needed to completely react with all of the methane. Comparing this amount to the actual amount of oxygen, shows that there isn't enough oxygen (we only have 1.25 mol). Thus the oxygen is the limiting reagent.

Step 3: Now that the limiting reagent has been determined we can calculate the amount of product that will be produced. Make sure that you start the calculation with the actual amount of limiting reagent.

$$1.25 \text{ mol O}_2 \left(\frac{1 \text{ mol CO}_2}{2 \text{ mol O}_2} \right) = 0.625 \text{ mol CO}_2 \quad (5)$$

This is the stoichiometric yield for carbon dioxide, which is the amount that would be produced if 100 % of the limiting reagent reacts. The stoichiometric yield of water could also be determined in the same manner. It is oftentimes more useful to express the stoichiometric yield in terms of mass, this can be done by simply multiplying by molar mass.

$$0.625 \text{ mol CO}_2 \left(\frac{44.0 \text{ g}}{1 \text{ mol}} \right) = 27.5 \text{ g CO}_2 \quad (6)$$

Step 4: In order to determine the amount of excess reagent that remains it is first necessary to determine the amount that reacted. Again you want to start this calculation with the limiting reagent.

$$1.25 \text{ mol O}_2 \left(\frac{1 \text{ mol CH}_4}{2 \text{ mol O}_2} \right) = 0.625 \text{ mol CH}_4 \text{ reacted} \quad (7)$$

Now the amount of methane that reacted can be subtracted from the initial amount methane present to determine the amount remaining.

$$0.750 \text{ mol} - 0.625 \text{ mol} = 0.125 \text{ mol CH}_4 \text{ left over} \quad (8)$$

Often times reactions do not proceed to completion, that is they do not reach the stoichiometric yield. There can be many reasons for a reaction to stop short of completion, including non-ideal conditions and competing reactions. A measure of a reaction's efficiency is percent yield:

$$\% \text{ yield} = \left(\frac{\text{actual yield}}{\text{stoichiometric yield}} \right) \times 100 \quad (9)$$

The actual yield is the amount of product formed during the experiment. By definition the actual yield cannot exceed the stoichiometric yield.

Example 2: Suppose that the actual yield of carbon dioxide from the reaction in example 1 was measured to be 21.2 g. The percent yield would then be:

$$\% \text{ yield} = \left(\frac{21.2 \text{ g}}{27.5 \text{ g}} \right) \times 100 = 77.1 \% \quad (10)$$

Procedure:

Caution: Some of the chemicals used in this experiment are dangerous, always follow proper safety procedures. The aspirin that you make will not be pure enough to take – **do not consume your aspirin!**

- 1.) Fill a 400 mL beaker with approximately 100 mL of water. Place the beaker on a hot plate and bring to a boil.
- 2.) Add between 4 and 6 g of salicylic acid to a flask. Record the exact mass in your notebook.
- 3.) In a hood, measure out between 5 and 7 mL of acetic anhydride ($d = 1.08 \text{ g/mL}$) and add this to the flask with the salicylic acid. Record the exact volume of acetic anhydride in your notebook. To the flask slowly add 7 to 10 drops of concentrated sulfuric acid. After every two or three drops swirl the flask for a few seconds.
- 4.) Place the flask in the water bath (no need to clamp). Heat the contents for 15 minutes. If any solid remains after this time consult your instructor.
- 5.) Remove the flask from the water bath and cool it by running it under cold water. Cool the flask further by adding about 30 mL of ice water, and placing the flask inside an ice bath. Aspirin crystals should start to form at this time. If crystals are slow to form, it may help to scratch the inside of the flask with a stirring rod. Leave the flask in the ice until it appears no more crystals are forming.
- 6.) Set up a Büchner funnel and filter flask according to your instructor's directions. Filter the crude aspirin. Use your stir rod and squirt bottle to transfer all of the aspirin from the flask to the funnel.
- 7.) When it appears that your aspirin is dry, weigh a watch glass. Scrape your aspirin from the funnel on to the watch glass.
- 8.) Store your aspirin for one week.
- 9.) At the beginning of the next lab, weigh the watch glass and aspirin.

Name: _____
Aspirin – Data Sheet

Section: _____
CHE 101

Data (show calculations in the space at the bottom):

Mass of Salicylic Acid: _____

Volume of Acetic Anhydride _____

Density of Acetic Anhydride _____

Mass of Acetic Anhydride _____

Moles of Salicylic Acid _____

Moles of Acetic Anhydride _____

Limiting Reagent _____

Theoretical Moles of Aspirin Produced _____

Theoretical Mass of Aspirin Produced _____

Mass of watch glass _____

Actual Mass of Aspirin _____

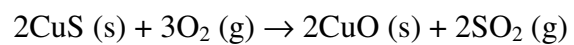
%-Yield _____

Calculations:

Name: _____
Aspirin – Questions

Section: _____
CHE 101

- 4.) If 75.0 g of copper (II) sulfide reacts with 40.0 g of oxygen and forms 54.0 g of copper (II) oxide, what is the percent yield? (*Hint: this is a limiting reagent problem.*)



Name: _____
Synthesis of Aspirin – Pre Lab Questions

Section: _____
CHE 101

- 1.) Calculate the molar masses for the following compounds:
 - a.) salicylic acid
 - b.) acetic anhydride
 - c.) aspirin

- 2.) Acetic acid is the active ingredient in what common food?

- 3.) Why do you have to store your aspirin for a week before weighing it?

- 4.) What are the common side effects associated with the use of aspirin?

- 5.) Define the following terms:

Antipyretic –

Analgesic –