

## Theoretical Yield and Percent Yield: The Synthesis of tris(2,4-pentanedionato)iron(III)

### EXPERIMENTAL TASK

Synthesize tris(2,4-pentanedionato)iron(III), and calculate both the theoretical yield and the percent yield of the product.

### Objectives

**After completing this experiment, the student will be able to:**

1. Synthesize a chemical compound, based on an experimental procedure such as would be published in a professional journal.
2. Calculate the theoretical yield of the product of a chemical reaction, based on the amounts of reactants used.
3. Calculate the percent yield of the product in a chemical reaction, based on its theoretical and actual yields.
4. Identify specific sources of error that lead to loss of product and/or impurities.

### Additional Reading

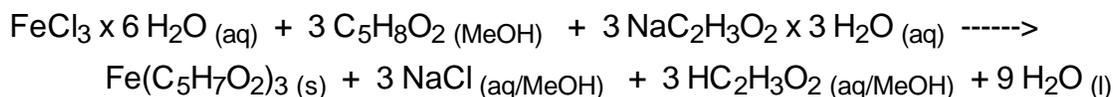
- General, Organic and Biological Chemistry, by Timberlake, chapter 7.
- Laboratory Handbook for General Chemistry, 2<sup>nd</sup> edition, by Griswold, et al, chapter 7 (Suction Filtration)

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### Background

One of the major branches of chemistry is that which deals with making new compounds. This area of chemistry is called **synthesis**, or **synthetic chemistry**. When a chemist carries out the synthesis of a new compound, he or she also determines the theoretical yield and percent yields of the product, based on the specific reaction or reactions used and the amounts of reactants used. This information helps the chemist to make the synthesis as efficient as possible, and to compare the efficiency of two or more different methods of synthesizing the same compound.

When the chemist has maximized the percent yield by improving the techniques and reactions involved, she or he will report the synthesis by publishing a paper in a journal. This paper must include the percent yield, which informs other chemists of the efficiency of the synthetic method involved. In this experiment you will carry out a synthetic reaction and determine the theoretical and percent yields. The balanced equation for the synthetic reaction you will carry out is:



Note that two of the reactants include water molecules in their formulas. The first is given as “FeCl<sub>3</sub> x 6 H<sub>2</sub>O” and is named iron(III) chloride hexahydrate. Both this compound and NaC<sub>2</sub>H<sub>3</sub>O<sub>2</sub> x 3 H<sub>2</sub>O are *hydrated ionic compounds*. When such a compound crystallizes from water solution, water molecules are trapped in the crystal structure as the solid forms (the water molecules are those that hydrated the positive ions while they were dissolved in the water). Because the water molecules can easily be removed by gentle heating of the compound, they are shown separately in the formula and are called *waters of hydration*. **The molar mass of a solid hydrated ionic compound includes the mass of the water molecules.** The molar mass of iron(III) chloride hexahydrate, then, is equal to the molar mass of FeCl<sub>3</sub> plus six times the molar mass of water.

In the procedure for synthesizing Fe(C<sub>5</sub>H<sub>7</sub>O<sub>2</sub>)<sub>3</sub> –tris(2,4-pentanedionato)iron(III)– two solvents, water and methanol, will be used instead of one. This is because two of the reactants, iron(III) chloride hexahydrate and sodium acetate trihydrate, are very soluble in water, while the third reactant, 2,4-pentanedione, is not very soluble in water, but is soluble in methanol. The 2,4-pentanedione will be dissolved in methanol first, and then added to an aqueous mixture of the other two reactants. As the reaction proceeds, the product, tris(2,4-pentanedionato)-iron(III), will precipitate out of the mixture. It is an orange-red solid at room temperature, and is soluble in methanol, but only slightly soluble in water. The product will be collected by suction filtration (see the Laboratory Handbook for General Chemistry), dried, and its actual yield measured.

In this reaction, sodium acetate trihydrate and 2,4-pentanedione are used in excess. You will use the mass of iron(III) chloride hexahydrate added to the reaction to calculate the theoretical yield of tris(2,4-pentanedionato)iron(III).

***The final results will be:***

- A) evidence the desired product was synthesized, in the form of observations of the properties of the reactants and products**
- B) the theoretical yield of the product for the reaction as you carried it out**
- C) the actual yield of the product in your reaction**
- D) the percent yield of product in your reaction.**

### Pre-lab Questions

1. How many moles of mercury are contained in 46.9 mL of Hg? The density of mercury is 13.546 g/mL.
2. How many moles of cobalt(II) chloride hexahydrate are there in 3.091 g of  $\text{CoCl}_2 \times 6 \text{H}_2\text{O}$ ?
3. What places in the procedure given on the next page need more information about what to do, or what equipment to use, in order for you to perform the experiment? Where can you get this information?

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### BEFORE STARTING THE EXPERIMENT

The procedure given here is in a format similar to what might be presented in a professional chemistry journal. It is assumed that the person reading it (you, the student, in this case) has enough experience in the chemistry laboratory to know how to “fill in the blanks” – that is, to figure out what equipment, materials, etc., will be needed in addition to what the procedure specifically mentions. Therefore, **you must read through the procedure and find places where the instructions are incomplete in some way**, and determine what other equipment or information you will need when you reach that part of the experiment, using both your own knowledge from this course and the information in the **Laboratory Handbook for General Chemistry**. The procedure is given in the past tense, as a report of what the chemists did in their own laboratory.

It will also be your responsibility to record all observations of reactants and products, all measurements, and a description of the procedure as you performed it. When the procedure calls for “1.3 g of iron(III) chloride hexahydrate” it means “about 1.3 g,” but that you will need to record the amount you use as precisely as the measuring instrument allows.

### Safety

Always keep in mind the rules presented in both the "MCC Laboratory Safety Rules" and the Laboratory Handbook for General Chemistry. It is your responsibility to make sure that you follow all safety rules at all times, and to graciously help everybody else in the laboratory (including the instructor) to do the same.

### Hazardous Materials

In this experiment you will be using **methanol**, which is flammable, so there will be no flames in the lab during this experiment. **2,4-pentanedione** is corrosive, so it will be stored in the fume hood to reduce the hazard of any spills. **Iron(III) chloride hexahydrate** is an irritant. You should wear gloves when handling any of these compounds. **Dispose of all wastes in appropriate containers—separate metal pieces from liquids before disposing of them.**

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### EXPERIMENT PROCEDURE

1.3 g of iron(III) chloride hexahydrate ( $\text{FeCl}_3 \times 6 \text{H}_2\text{O}$ ) was dissolved in a minimum amount of water (4 mL maximum). 2.5 g of sodium acetate trihydrate ( $\text{NaC}_2\text{H}_3\text{O}_2 \times 3 \text{H}_2\text{O}$ ) was dissolved in a minimum of water (5 mL maximum). The aqueous sodium acetate solution was slowly added to the aqueous iron(III) chloride solution, and the mixture was stirred during the addition. (Note that the mass of the iron(III) chloride hexahydrate must be measured carefully and precisely to the significant figure limit given by the balance--usually 0.001 g--because it is this mass that will be used to calculate the theoretical yield).

2.00 mL of 2,4-pentanedione ( $\text{C}_5\text{H}_8\text{O}_2$ ) was dissolved in about 5 mL of methanol.

The 2,4-pentanedione/methanol solution was added to the aqueous iron(III) chloride/sodium acetate solution. The mixture was stirred during the addition, and for another 5 minutes afterward.

The resulting mixture was suction filtered and the solid product was rinsed three times with 25 mL of water each time. The product was allowed to air dry with the suction on. The product was then collected in a sample container and dried in a desiccator before the actual yield was measured.

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### RESULTS

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Calculate the molar mass of both tris(2,4-pentanedionato)iron(III) and iron(III) chloride hexahydrate. Use these molar masses, the measured mass of iron(III) chloride hexahydrate and the mole-to-mole ratio of these two substances from the balanced reaction equation to calculate the theoretical yield of the product, tris(2,4-pentanedionato)iron(III), for your synthesis.

Use the actual yield (as measured in the synthesis) and theoretical yield of tris(2,4-pentanedionato)iron(III) to calculate the percent yield of the synthesis, as you performed it.