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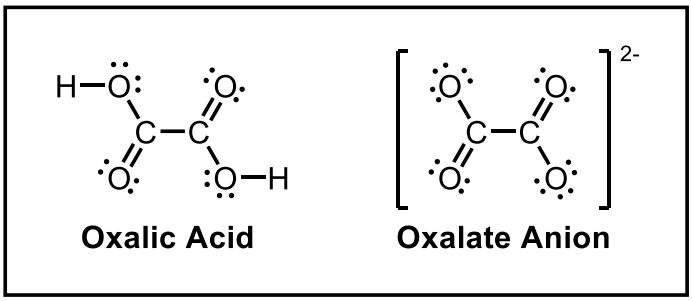
Experiment #4: Synthesis and Decomposition of an Oxalate Salt

Introduction:

Oxalic acid ($H_2C_2O_4$) is an organic acid that is widespread in biology. It is the simplest diacid, and forms compounds called oxalates when it loses two hydrogens.



The best-known oxalate salt is formed from calcium. These mineral deposits, kidney stones, are a common medical condition for not just humans, but also dogs, cats, and even iguanas. Kidney stones are formed by the precipitation of calcium ions with oxalate ions ($C_2O_4^{2-}$) in the urine. Surprisingly, increased consumption of calcium in the diet is not correlated with increased occurrence of kidney stones. However, eating high content oxalic acid foods, such as spinach, nuts, rhubarb, and sesame seeds, can lead to more kidney stones.

Today, we make an oxalate from either Iron or Manganese. Then, we will decompose the oxalate with heat. (This reaction is also called *pyrolysis*.)

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Procedure:

Part 1 – Synthesis of the Oxalate Salt

1. Weigh between 1.9 g and 2.1 g of YOUR starting sample (hydrated metal salt with either Fe or Mn) into a 100 ml beaker. Record the metal salt's exact mass in Table 1b, keeping all the significant numbers on the balance. Add 10 mL distilled water and stir to dissolve.

2. Use a graduated cylinder to measure 25.0 mL of 0.888 M oxalic acid solution.
   Solution contains 4.0 g oxalic acid ($H_2C_2O_4$) per 50.0 mL of solution.

3. Once the starting metal salt is completely dissolved, slowly pour the 25.0 ml of oxalic acid solution into the same beaker. Initially stir to mix and allow the solution to sit for 10 minutes while a precipitate forms. Try not to lose any precipitate.

4. Prepare a Büchner funnel vacuum filtration while the precipitate is forming. Before placing the filter paper in the Büchner funnel, weigh and record the filter paper mass in Table 1 *(f)*.

5. After the 10 minute waiting period, begin vacuum suction through the filter paper in the funnel, wet the paper with your wash bottle containing DI water. Then slowly transfer the precipitate solution from the beaker onto the center of the filter paper. Attempt to keep most of the precipitate in the center of the paper and not on the funnel walls. You are keeping the solid formed. The filtrate liquid that passes through will be waste that may go down a sink.

6. Once the entire solution has been emptied into the funnel, use your wash bottle to rinse out any remaining precipitate into the vacuum Büchner funnel. Transfer all of the precipitate from the beaker.

7. Because water dries too slowly, next wash the precipitate with ethanol. Do this while the vacuum is still running. Obtain an ethanol filled bottle from under the hood. Gently squirt the ethanol over the precipitate in the funnel to saturate. During this step, water on the precipitate particles will dissolve in the ethanol and facilitate the drying of your sample. Squirt a bit more ethanol over the solid one or two more times after pausing a minute.

8. Keep the vacuum on to draw air through the filter paper. The ethanol will evaporate from the solid. Keep the vacuum on until the solid is dry, (about 10-15 minutes). You can push around the precipitate with a glass rod to test for dryness. Once the solid is completely dry, turn off the vacuum.
   - **Be patient!** The drier your solid product, the more accurate your results.
   - This solid product must be **completely dry** before beginning Part II of the procedure.

9. Weigh the colored solid and record its mass in Table 1: *(g) experimental yield*

10. Determine *(h)* the identity of the limiting reactant, *(i)* theoretical yield of the hydrated metal oxalate, and *(j)* the percent yield of the hydrated metal oxalate product. Complete Table 1.

Part 2 – Pyrolysis of the Metal Oxalate

You will pyrolyze the metal oxalate you synthesized in Part I. You will use stoichiometric considerations to determine which pyrolysis reaction (of those given in prelab Question #5) your metal oxalate undergoes.

1. Record the mass of the Al dish in Table 2 *(a)*.

2. Measure between 0.990 g and 1.010 g of your dry, hydrated metal oxalate into the aluminum dish. Record precise masses in Table 2 *(b)*. Don't use the entire sample from the first part if you have more than 1.0 g product! If you have less than one gram, you may be able to find another group with excess of your same sample to share with you.
    - To enhance heat transfer, spread out the solid over the bottom of the dish.
    - It is important to use between 0.990 g and 1.010 g because your prelab calculations were based upon pyrolysis of 1.0 g of hydrated metal oxalate.

3. Turn the dial on a hot plate up to about 400°C or level 7. Place the aluminum dish on the hot plate. The hydrated metal oxalate will undergo dehydration (loss of water) followed by a pyrolysis reaction. You will see a visible change in the product.

4. Wait until the metal oxalate has completely changed color. Remove the aluminum dish from the hot plate using a pair of metal tongs and place it on the laboratory bench to cool to room temperature. The aluminum dish should cool quickly but the precipitate may take longer.
    - Caution: the aluminum dish will be VERY hot – Use tongs!!
    - Be patient! The cooler the precipitate, the better your results.
    - Never weigh hot objects. It's bad for the balance's electronics.

5. Record the mass of the pyrolyzed product in Table 2 *(c)*.

6. Complete Table 2. The pyrolysis reaction occurs with near 100% yield, so the actual yield of the pyrolysis reaction should be very close to the theoretical yield you calculated for the correct reaction equation (as calculated in Question #5 of the prelab).

7. Dispose of all chemical waste properly.

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Option Fe: Pre-lab Calculations for the Iron Based Experiment:

1. Calculate the molar mass of the starting hydrated salt material, $FeSO_4 \cdot 7H_2O$.

2. Your oxalate synthesis product will be $FeC_2O_4 \cdot 2H_2O$. Calculate the molar mass of $FeC_2O_4 \cdot 2H_2O$.

3. Write a balanced molecular equation that shows your $FeSO_4 \cdot 7H_2O$, reacting with oxalic acid ($H_2C_2O_4$) to form $FeC_2O_4 \cdot 2H_2O$. What will be the other product(s) of the double displacement reaction? Water will need to be added somewhere to balance correctly.

4. Pyrolysis of $FeC_2O_4 \cdot 2H_2O$ will follow one of the following molecular equations. Balance each molecular equation. *(It's OK to keep a fraction when balancing the O_2 molecule in* A *or* B*)*

   A. $FeC_2O_4 \cdot 2H_2O(s) + O_2(g) \rightarrow FeO(s) + H_2O(g) + CO_2(g)$

   B. $FeC_2O_4 \cdot 2H_2O(s) + O_2(g) \rightarrow Fe_2O_3(s) + H_2O(g) + CO_2(g)$

   C. $FeC_2O_4 \cdot 2H_2O(s) \rightarrow FeCO_3(s) + H_2O(g) + CO(g)$

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5. For each of the possible reactions, calculate the theoretical yield (in grams) of the solid product, assuming that you use 1.0 g $FeC_2O_4 \cdot 2H_2O$ and that oxygen is the excess reactant. You will need to write in coefficients from question 4 to balance the molecular equations.

   A. $FeC_2O_4 \cdot 2H_2O(s) + O_2(g) \rightarrow FeO(s) + H_2O(g) + CO_2(g)$

   B. $FeC_2O_4 \cdot 2H_2O(s) + O_2(g) \rightarrow Fe_2O_3(s) + H_2O(g) + CO_2(g)$

   C. $FeC_2O_4 \cdot 2H_2O(s) \rightarrow FeCO_3(s) + H_2O(g) + CO(g)$

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Option Mn: Pre-lab calculations for the Manganese based experiment:

1. Calculate the molar mass of the starting hydrated salt material, $MnSO_4 \cdot H_2O$.

2. Your oxalate synthesis product will be $MnC_2O_4 \cdot 3H_2O$. Calculate the molar mass of $MnC_2O_4 \cdot 3H_2O$

3. Write a balanced molecular equation that shows $MnSO_4 \cdot H_2O$, reacting with oxalic acid ($H_2C_2O_4$) to form $MnC_2O_4 \cdot 3H_2O$. What will be the other product(s) of the double displacement reaction? Water will need to be added somewhere to balance correctly.

4. Pyrolysis of $MnC_2O_4 \cdot 3H_2O$ will follow one of the following molecular equations. Balance each molecular equation. *(It's OK to keep a fraction when balancing the O_2 molecule in* A *or* B.*)*

   A. $MnC_2O_4 \cdot 3H_2O(s) + O_2(g) \rightarrow MnO(s) + H_2O(g) + CO_2(g)$

   B. $MnC_2O_4 \cdot 3H_2O(s) + O_2(g) \rightarrow Mn_3O_4(s) + H_2O(g) + CO_2(g)$

   C. $MnC_2O_4 \cdot 3H_2O(s) \rightarrow MnCO_3(s) + H_2O(g) + CO(g)$

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5. For each of the possible reactions, calculate the theoretical yield (in grams) of the solid product, assuming that you use 1.0 g $MnC_2O_4 \cdot 3H_2O$ and that oxygen is the excess reactant. You will need to write in coefficients from question 4 to balance the molecular equations.

   A. $MnC_2O_4 \cdot 3H_2O(s) + O_2(g) \rightarrow MnO(s) + H_2O(g) + CO_2(g)$

   B. $MnC_2O_4 \cdot 3H_2O(s) + O_2(g) \rightarrow Mn_3O_4(s) + H_2O(g) + CO_2(g)$

   C. $MnC_2O_4 \cdot 3H_2O(s) \rightarrow MnCO_3(s) + H_2O(g) + CO(g)$

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Data and Calculations

Part 1: Synthesis of Metal Oxalate

Determine the identity of the limiting reactant, theoretical yield of the hydrated metal oxalate, and the percent yield of the hydrated metal oxalate product. Complete Table 1.

Table 1. Synthesis of Metal Oxalate

a) complete chemical formula of your starting material: (formula found on question 1)	
b) mass of hydrated metal salt used (g) *tare your 100 ml beaker and add 1.9-2.1g sample*	
c) moles of hydrated metal salt used convert grams from (b) using molar mass in question 1	
d) volume of 0.888M oxalic acid solution used *should be close to 25.0mL*	
e) moles of oxalic acid used *calculate from volume and molarity*	
f) mass of filter paper	
g) experimental yield: total mass of hydrated metal oxalate precipitate (grams) after drying by vacuum filtration. filter paper + salt mass: _____ *subtract the filter paper*	
h) identify the limiting reactant: either your starting formula in part (a) or oxalic acid *Refer to the balanced chemical equation in pre-lab quest. #3 and your moles of each reactant calculated in c and e*	
i) theoretical yield of hydrated metal oxalate product (g)	
j) percent yield of hydrated metal oxalate product	

Calculations: Show your calculations. Watch your units and report all answers with the correct number of significant figures.

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Part 2: Decomposition (Pyrolysis) of Metal Oxalate

Complete Table 2. The pyrolysis reaction occurs with near 100% yield, so the actual yield of the pyrolysis reaction should be very close to the theoretical yield you calculated for the correct reaction equation (as calculated in Question #5 of the prelab).

Table 2. Pyrolysis of Metal Oxalate

a) Weigh the empty Aluminum dish before adding the filtered product.	
b) Before pyrolysis: mass of hydrated metal oxalate (grams) *Ideally between 0.99 to 1.01 gram* Al + oxalate salt mass: _____ *subtract the Al dish*	
c) After pyrolysis: final mass of pyrolysis product (grams) Al + pyrolyzed salt mass: _____ *subtract the Al dish*	
d) theoretical mass of product in reaction **A** From prelab (quest. #5A):	
e) theoretical mass of product in reaction **B** From prelab (quest. #5B):	
f) theoretical mass of product in reaction **C** From prelab (quest. #5C):	
g) Using the above information, which pyrolysis reactions actually occurred, **A**, **B** or **C**?	
h) What is the expected chemical formula of the pyrolyzed metal salt formed?	

Calculations: Refer to your work from prelab Question #5 from your option. Watch your units and report all answers with the correct number of significant figures.