GROWING CRYSTALS

THE SYNTHESIS OF POTASSIUM TRISOXALATOFERRATE(III) TRIHYDRATE $K_3[Fe(C_2O_4)_3]\cdot 3\,H_2O$

The purpose of this experiment is to synthesize—that is, to prepare from other reagents —a chemical compound with the chemical formula $K_3[Fe(C_2O_4)_3]\bullet 3H_2O$ (**Figure 1**). The molecule contains an iron 3+ ion (Fe^{3+}) in the center with three $C_2O_4^{2-}$ ions (**Figure 2**) attached to it like the blades on a propeller (**Figure 3**). This unit, called a *complex ion*, has an electrical charge of 3-, so there are three potassium ions (K^+) in the solid to balance this negative charge. In addition, when the compound forms solid, green crystals, three water molecules are trapped in the solid for each unit of $K_3[Fe(C_2O_4)_3]$.

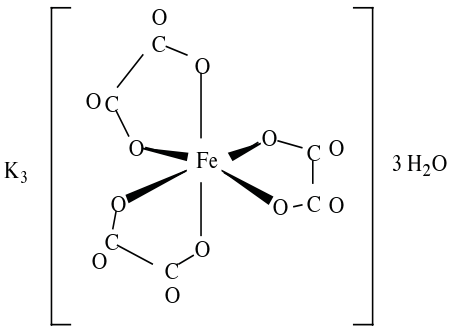


Figure 1 This iron-containing compound is a beautiful green, crystalline solid.

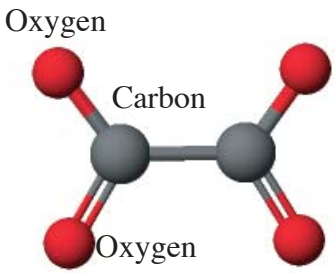


Figure 2 The oxalate ion, $C_2O_4^{2-}$, is a simple ion made up of two C atoms and 4 O atoms.

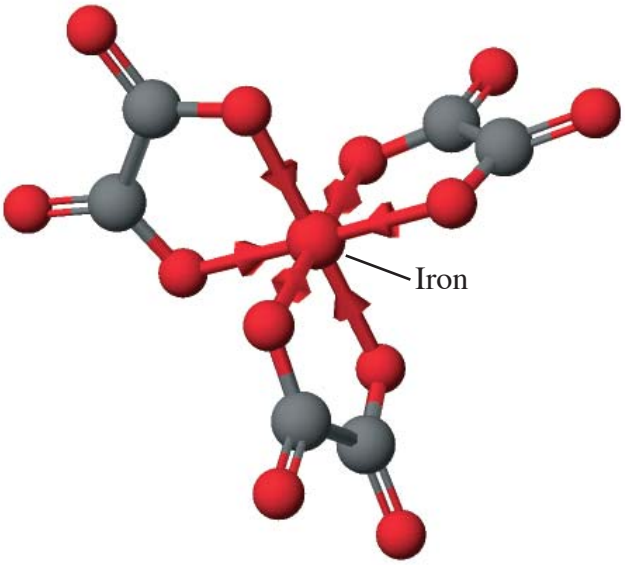


Figure 3 The iron-containing ion $Fe(C_2O_4)_3^{3-}$. At the center of the ion is a Fe^{3+} ion. Surrounding it are three $C_2O_4^{2-}$ ions, each attached through two O atoms to the Fe^{3+} ion. Notice that the large ion resembles a three-bladed propeller.

EXPERIMENTAL DIRECTIONS

SOLID REAGENTS that need to be weighed are generally next to the balances along the wall of the lab. LIQUIDS and SOLUTIONS are generally on the shelf near the stockroom or in a fume hood.

1. Add 5 drops of dilute sulfuric acid (H_2SO_4) to 15 mL of warm water in a 250 mL beaker. Next add 5.0 grams of ferrous ammonium sulfate hexahydrate, $Fe(NH_4)_2(SO_4)_2 \bullet 6H_2O$. When everything has been dissolved, add 25 mL of 1.0 molar oxalic acid ($H_2C_2O_4$), and then heat the solution to boiling; stir continuously.

Your instructor will demonstrate how you decant a solution from a solid precipitate.

2. Allow the yellow precipitate — the solid in the beaker — to settle, and then *decant* the liquid. Wash the precipitate with distilled water, and then decant the liquid after the precipitate has again settled. Throw away the liquid (wash it down the drain with lots of water) and keep the solid.

Make sure you pick up the correct solution bottles from the reagent shelf. One bottle has $K_2C_2O_4$ and the other has oxalic acid, $H_2C_2O_4$. Both are colorless solutions.

3. Add 10 mL of saturated, aqueous potassium oxalate ($K_2C_2O_4$) to the washed solid precipitate. Heat the solution to 40 °C, and then add 20 mL of 3% hydrogen peroxide (H_2O_2), a few milliliters at a time, stirring continuously. (Wait a few moments between additions and keep the temperature near 40 °C.)

4. Ignoring any red-brown precipitate [$Fe(OH)_3$] that may form, heat the solution to boiling, and add 5 mL of 1.0 molar oxalic acid. Add another 3 mL of the oxalic acid solution dropwise, keeping the solution near boiling.

5. Filter the hot solution into a 100 mL beaker as demonstrated by your instructor and illustrated on the next page.

6. Add 10 mL of ethanol (ethyl alcohol) and warm to dissolve any crystals that may form.

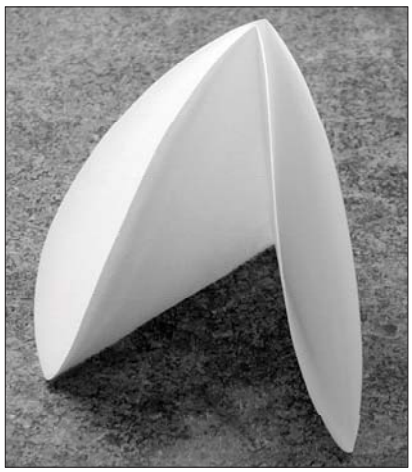
Your green crystals will form slowly so they will be ready when you return to the lab the next lab period.

7. Tie a short piece of thread to a splint and suspend the thread in the solution. Place a piece of paper over the beaker and place the beaker in your drawer until the next time you come to laboratory.

Make sure you weigh the crystals in the next lab period and turn them in to your instructor.

8. In the next laboratory period, remove the thread, now covered with crystals, from the beaker. Transfer the crystals to a paper towel and blot them dry with the towel. When the crystals are as dry as you can get them, weigh them and record the weight on the report form. Finally, place them in a test tube, label the tube with your name, and hand it in to your instructor. Turn the completed report form in to your instructor in your next laboratory period.

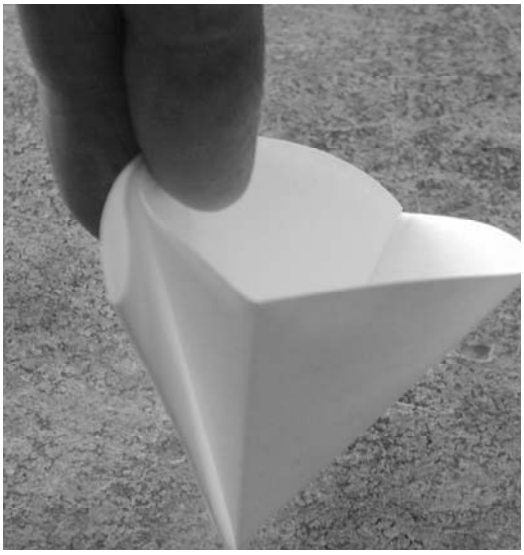
Figure This figure illustrates the way to fold a piece of analytical filter paper and place it in a funnel. The funnel should be placed in a metal ring on your ring stand or in an Erlenmeyer flask.



Step 1: Fold the filter paper in half and then in half again.

Step 2: It helps the filter work more effectively if you tear off a small corner. If you are going to weigh the paper, make sure you do so *AFTER* you tear off the corner.

Step 3: Separate the top edges of the paper so that three leaves go one way and the fourth leaf in the other direction.



Step 4: Place the filter paper in the funnel. Moisten the paper with a few drops of water from your squeeze bottle.

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LABORATORY REPORT

Name_____ Section _____ Grade _____

1. In your own words, describe what you saw as you added hydrogen peroxide in Step 3.

2. Describe the crystals of $K_3[Fe(C_2O_4)_3]\bullet 3H_2O$. Include the color, relative size, crystal shape, and any other observations that allow you to give a complete description.

3. Mass of $K_3[Fe(C_2O_4)_3]\bullet 3\ H_2O$ = _____ grams