**CH 412 LA: INORGANIC CHEMISTRY LABORATORY (Spring 2021)**

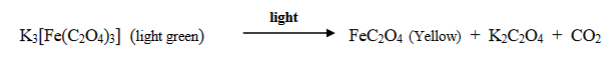
**Title:** Synthesis of light sensitive compound potassium iron (III) oxalate

1. **Purpose: (1 point)**

**The purpose of this experiment is to synthesize iron (III) oxalate and potassium iron (III) oxalate and isolate potassium iron (III) oxalate using solvent exchange crystallization. Another purpose is to show the stability of coordination complexes and the light sensitivity of iron oxalate.**

1. **Drawing of structure of the main compound or balanced chemical equation if synthesis is performed: (1 point)**

(light sensitive reaction)



**3. Reagents and the major product (up to 5 points)**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Name** | **M.W.**  (0.5 pts) | **Density**  (0.5 pts) | **Amount (grams/mL)**  (0.5 pts) | **Moles**  (0.5 pts) | **Hazards/Precautions**  **(MSDS data) and melting point or boiling point** (2 pts) | **Role of the reagent** (1 pts)\* |
| Iron (II) sulfate heptahydrate | 278.01 | 2.84 g/cm3 | 3.6 grams | 0.0129 | Harmful if swallowed. Causes eye and skin irritation. May cause respiratory tract irritation.  MP: 64°C  BP: 300°C | Reactant |
| Oxalic acid dihydrate | 126.04 | 1.9 g/cm3 | 1.7 grams + 6 mL 0.5 M + excess | 0.0165+ | Causes burns by all exposure routes. Harmful if swallowed, inhaled, or absorbed through skin.  MP: 101°C | Reactant |
| Iron (II) oxalate | 179.90 | 2.28 g/cm3 | N/A | N/A | Harmful if swallowed or in contact with skin.  MP: 190°C | Product |
| Sulfuric acid | 98.07 | 1.83 g/cm3 | Several drops 3M | N/A | Causes eye and skin burns. Causes digestive and respiratory tract burns. Strong inorganic acid. Corrosive.  MP: 10°C BP: 290-338°C | Catalyst |
| Hydrogen peroxide | 34.0147 | 1.450 g/mL  (pure) | 8 mL 6% | N/A | May cause fire or explosion, strong oxidizer. Harmful if swallowed, causes skin and eye burns.  MP: -0.43°C  BP: 150°C | Reactant |
| Potassium oxalate monohydrate | 184.24 | 2.00 g/cm3 | N/A | N/A | May cause severe digestive tract irritation. Harmful in contact with skin and eyes.  MP: 356°C | Product |
| Potassium iron (III) oxalate trihydrate | 491.26 | 2.13 g/cm3 | N/A | N/A | Harmful if swallowed or in contact with skin.  MP: 230°C | Product |
| Iron (III) hydroxide | 106.87 | N/A | N/A | N/A | May cause skin or eye irritation. | Product |

**\*** Mention role as either reactant, solvent, catalyst or product

**4. Calculations: (1 point) (ignore it)**

Show each calculation for moles of reagents and for theoretical and actual yield. Fill in the box with the limiting reagent and theoretical yield:

The limiting reagent is

The theoretical yield is

**5. Procedure (up to 2 points)**

|  |  |
| --- | --- |
| **Procedure** | **Observations and Lab Data** |
| * Create solution A by weighing 3.6g FeSO4 · 7 H2O into a beaker and adding a few drops of 3M H2SO4. Add water into the beaker until FeSO4 ·7 H2O is completely dissolved. * Create solution B by weighing 1.7 g H2C2O4 · 2 H2O into a beaker and adding water until sample is dissolved. * Remove any insolubles from both solutions. * Slowly mix solution A and B. Boil the solution for 4 minutes with constant stirring. Get rid of the supernatant, and was the FeC2O4 · 2 H2O precipitation with hot water to remove leftover sulfate ion. * Next, weigh 3.5 g K2C2O4 · H2O and add 10 mL H2O to dissolve sample completely. Heat if necessary. * Add potassium oxalate solution to prepared iron (II) oxalate crystals and then place solution in 40°C water bath. * Slowly add 8 mL 6% hydrogen peroxide with continued stirring. Solution will become turbid when all hydrogen peroxide is added. * Place one drop of reaction solution into a well on assay plate, and add another drop of K3Fe(CN)6. Note development of blue color, indicating presence of Fe2+ ion. * Add 6 mL 0.5M H2C2O4 into the solution. Keep adding until solution is clear. Record added H2C2O4 volume. * Add 10 mL alcohol into the transparent K3[Fe(C2O4)3] solution. * Place one end of a cotton string into the solution and tie another end of the string to a glass rod placed over the mouth of the beaker. * Cover the beaker with a paper and leave it at a dark place overnight. * Collect the crystals through filtration. Wash crystals with alcohol and dry the samples. Weigh and calculate yield. * Prepare 0.5 mL K3[Fe(C2O4)3] saturated solution. Use the solution as ink to write words or draw on paper. Expose paper to light and observe appearance of words/drawings. | Color changes, exothermic or endothermic reactions, gas generation, etc.; tare weights for flasks, etc. |

**6.** Results; include actual yield in grams and % yield.

**Results (need to get signed by instructor or TA):**