THE CASE OF THE GREEN CRYSTALS

4-Part Mystery Lab
Synthesis, Purification, Analysis, Identification and Yield

STARRY STARRY NIGHT

The moon lights the star-filled sky
Suddenly a brilliant green fireball appears, streaking through the clear night sky
A muted boom is heard as the fireball lands in the distance
The next day, all the vegetation in the vicinity of the impact crater are struggling for life and a scattering of mysterious green crystals are found
The crystals have been found to contain iron, potassium, oxalate and water, but little else is known
You must synthesize some of these crystals in the lab and determine the exact composition to save the world!
Back in the Lab

Michael and Ron have been able to produce some green crystals by mixing solutions of iron (III) chloride and potassium oxalate, but they need more to save the world. You can help! Keep reading for the top secret procedure.

We will react FeCl₃ with K₂C₂O₄, yielding a solid precipitate

\[ xK^+_{(aq)} + yFe^{3+}_{(aq)} + zC_2O_4^{2-}_{(aq)} + nH_2O \rightarrow K_xFe_y(C_2O_4)_z \cdot nH_2O_{(s)} \]

After synthesizing our rough crystals, we will re-crystallize from clean water and try to grow bigger purer crystals

- When a precipitate falls out of solution, other impurities may be carried with the solid & trapped in the crystal matrix
- We can re-dissolve the crystals in clean hot water and allow the crystals to re-form as the solution slowly cools
- The resulting crystals will be bigger and purer
Synthesis of the Green Crystals

- Preparation of iron (III) chloride solution
  - Use a dry 250 mL beaker
  - Weigh 3-4 g of iron (III) chloride hexahydrate into beaker using top loading balance (tare balance)
  - Add 15 mL deionized water and warm slightly to dissolve (set-up ring stand, ring and wire guaze)

- Preparation of potassium oxalate solution
  - Use a second dry 250 mL beaker
  - Weigh 8-9 g potassium oxalate monohydrate in beaker
  - Add 30 mL deionized water and heat gently to boiling.

The Reactants Come Together

- Once the $\text{K}_2\text{C}_2\text{O}_4$ is dissolved, add the $\text{FeCl}_3$ solution to the beaker. Rinse beaker with a tiny amount of deionized water to make sure all the solution is transferred.
- Heat the reaction to almost boiling
- Remove from heat using beaker tongs and place on non-asbestos pad or asbestos cloth to cool for 5 min
- After 5 min, transfer beaker to tray w/ ice water for 15 min.
- Check for crystals and if none, I will seed the solution.
Re-crystallization

• After ~30 min, carefully decant the liquid from the crystals into a beaker
• Discard the solution (not the crystals!!) into the waste container in the hood
• Add 25 mL deionized water to the crystals and heat gently until all the crystals dissolve

Place the beaker with the hot solution into your lab drawer and cover with a watch glass.
• Clean up and close your drawer gently
• By the next lab period your purer crystals should appear! You’re on your way to saving the world!

LOOKING AHEAD

• Today we synthesized our crystals and prepared the solution for re-crystallization. We still don’t know the formula of our crystals
• Next Tuesday, we will first wash the crystals using filtration and then determine the mass percent of oxalate ion in the crystals by a redox reaction titration
• On Thursday, we will prepare standard solutions and do an analysis to determine the mass percent of iron in the crystals using absorption spectrophotometry. We will also find mass percent of water in our crystals by weighing a sample and then heating the sample in the drying oven to remove the hydrated water.
DIRE WARNINGS

• Iron (III) chloride and potassium oxalate can be irritating – handle with care – clean up all crystals around balances

• Use caution with the bunsen burners – be aware of all the burners around you.

• Use BEAKER TONGS to handle your hot beakers.
  – In a container on the lab bench at the back of lab

• Pay attention to each step in the lab so you don’t accidently destroy or discard your crystals or the liquid that contains them. This is a 4 part lab and you need YOUR crystals each session.