An Iron One-Pot Reaction

Sodium Fluoride

Introduction

An iron one-pot reaction is actually a series of reactions that are performed in a single container. These reactions involve the formation of iron(III) complexes that have distinctive colors. Only colorless and light yellow solutions are added to the reaction pot to produce blue, red, yellow, and colorless solutions.

Concepts

• Complex ions

Equilibrium

• LeChâtelier's Principle

Materials

Iron(III) nitrate, Fe(NO ₃) ₃ ·9H ₂ O, 1.0 M, 2 mL	Sodium fluoride, NaF, 1.5 g
Nitric acid, HNO ₃ , 6 M, 2 mL	Water, distilled, 800 mL
Potassium thiocyanate, KSCN, 0.5 M, 1.5 mL	Beaker, 1-L
Oxalic acid, $H_2C_2O_4$ ·2 H_2O , 1.0 M, 4 mL	Magnetic stirrer
Potassium ferrocyanide, K ₄ Fe(CN) ₆ ·3H ₂ O, 0.1 M, 0.5 mL	Pipets, Beral-type, calibrated, 6

Safety Precautions

Iron(III) nitrate, oxalic acid, potassium thiocyanate, and sodium fluoride are toxic by ingestion and they may be irritating to the skin. Potassium ferrocyanide is moderately toxic by ingestion. Nitric acid is strongly corrosive and is also a very strong oxidizing agent. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Please review current Material Safety Data Sheets for additional safety, bandling, and disposal information.

Preparation

Prepare 1.0 molar iron(III) nitrate by adding 40.5 g of $Fe(NO_3)_3 \cdot 9H_2O$ to a 100-mL volumetric flask and filling with distilled water up to the line while mixing. Prepare 0.5 molar potassium thiocyanate by adding 4.9 g of KSCN to a 100-mL volumetric flask and filling with distilled water up to the line while mixing. Prepare 1.0 molar oxalic acid by adding 12.6 g of $H_2C_2O_4 \cdot 2H_2O$ to a 100-mL volumetric flask and filling with distilled water up to the line while mixing. Prepare 0.1 M potassium ferrocyanide by adding 4 g of $K_4Fe(CN)_6 \cdot 3H_2O$ to a 100-mL volumetric flask and filling with distilled water up to the line while mixing.

Procedure

To a 1-L beaker, add 800 mL of distilled water. Place it on a magnetic stirrer.

1. Add 2 mL of 1 M iron(III) nitrate solution with a 1-mL calibrated Beral-type pipet.

Add each of the following solutions, in order, allowing enough time between additions for each reaction to take place. Add only enough reagent to produce the desired color change. Avoid adding excess reagent.

- 2. 1–2 mL 6 M nitric acid solution.
- 3. 1.5 mL 0.5 M potassium thiocyanate solution.
- 4. 4 mL 1 M oxalic acid solution.
- 5. 1.5 g sodium fluoride.
- 6. 0.5–1 mL potassium ferrocyanide solution.

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Disposal

Please consult your current *Flinn Scientific Catalog/Reference Manual* for general guidelines and specific procedures governing the disposal of laboratory waste. The resulting solution may be flushed down the drain according to Flinn Suggested Disposal Method #26b.

Connecting to the National Standards

This laboratory activity relates to the following National Science Education Standards (1996):

Unifying Concepts and Processes: Grades K–12
 Evidence, models, and explanation
 Constancy, change, and measurement
 Evolution and equilibrium

 Content Standards: Grades 9–12
 Content Standard B: Physical Science, structure and properties of matter, chemical reactions

Discussion

The chemical equations of the reactions in this one-pot reaction are given below. Additional complexes of the hydrated ions may be present. In each reaction an equilibrium is established preventing the complete suppression of the original color.

1. The iron(III) (Fe³⁺) from the iron(III) nitrate exists as an equilibrium mixture yielding a light yellow color.

$$Fe(H_2O)_6^{3+}(aq) \rightleftharpoons [Fe(H_2O)_5OH]^{2+}(aq) + H^+(aq) \qquad K_{eq} = 1.23 \times 10^{-3}$$

colorless yellow-orange colorless

2. The addition of the nitric acid forces the equilibrium back towards the colorless aqueous iron(III) ions.

$$\begin{array}{rcl} \operatorname{Fe}(\operatorname{H}_2\operatorname{O})_6^{3*}(\operatorname{aq}) &\rightleftharpoons & [\operatorname{Fe}(\operatorname{H}_2\operatorname{O})_5\operatorname{OH}]^{2*}(\operatorname{aq}) + \operatorname{H}^*(\operatorname{aq}) \\ & colorless & yellow-orange \end{array}$$

3. The aqueous iron(III) ions react with the thiocyanate from potassium thiocyanate to form a blood-red thiocyanto complex.

$$\begin{array}{rll} \operatorname{Fe}(\operatorname{H_2O})_6^{3+}(\operatorname{aq}) + \operatorname{SCN}^-(\operatorname{aq}) &\rightleftharpoons & [\operatorname{Fe}(\operatorname{H_2O})_5 \operatorname{SCN}]^{2+}(\operatorname{aq}) + \operatorname{H_2O}(\operatorname{l}) & \operatorname{K_{eq}} = 1.3 \times 10^{-2} \\ & colorless & colorless & blood-red \end{array}$$

4. The oxalate ion in the oxalic acid complexes with the iron(III) ion preferentially over the thiocyanate, reflecting the large equilibrium constant.

$$\begin{array}{cccc} [\mathrm{Fe}(\mathrm{H}_{2}\mathrm{O})_{5}\mathrm{SCN}]^{2+}(\mathrm{aq}) &+ & \mathrm{C}_{2}\mathrm{O}_{4}^{-2-}(\mathrm{aq}) & \rightarrow & [\mathrm{Fe}(\mathrm{H}_{2}\mathrm{O})_{4}\mathrm{C}_{2}\mathrm{O}_{4}]^{+}(\mathrm{aq}) &+ & \mathrm{SCN}^{-}(\mathrm{aq}) &+ & \mathrm{H}_{2}\mathrm{O}(\mathrm{l})\mathrm{K}_{\mathrm{eq}} &= 2.5 \times 10^{6} \\ \hline & blood-red & colorless & yellow-green & colorless \end{array}$$

5. Excess fluoride ion from sodium fluoride is needed to cause the iron(III) oxalate complex to decompose forming the color-less fluoro complex.

$$[Fe(H_2O)_4C_2O_4]^+(aq) + F^-(aq) + H_2O(l) \rightleftharpoons [Fe(H_2O)_5F]^{2+}(aq) + C_2O_4^{2-}(aq) \qquad K_{eq} = 6.0 \times 10^{-5}$$

yellow-green colorless colorless colorless

6. The ferrocyanide ion, [Fe(CN)₆]⁴⁻, from potassium ferrocyanide reacts with the fluoro complex producing the more stable, dark blue complex, iron(III) ferrocyanide.

$$\begin{array}{rcl} 4[Fe(H_2O)_5F]^{2+}(aq) &+ & 3Fe(CN)_6^{4-}(aq) & \rightarrow & Fe_4[Fe(CN)_6]_3(s) &+ & 4F^-(aq) &+ & 20H_2O(l) & K_{eq} &= & 1 \times & 10^{16} \\ \hline colorless & pale \ yellow & blue & colorless \\ \end{array}$$

Acknowledgment

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References

Dean, J. A., Ed., *Lange's Handbook of Chemistry*, 13th ed.; McGraw-Hill: New York, 1985; pp 5–72 to 5–91. Shakhashiri, B. Z. *Chemical Demonstrations: A Handbook for Teachers of Chemistry*, Vol. 1; The University of Wisconsin: Madison, 1983: pp 338–343.

Materials for An Iron One-Pot Reaction are available from Flinn Scientific, Inc.

Catalog No.	Description
F0008	Iron(III) Nitrate, 100 g
N0048	Nitric Acid, 6 M, 500 mL
P0178	Potassium Thiocyanate, 0.1 M, 500 mL
S0316	Sodium Fluoride, 100 g
P0220	Potassium Ferrocyanide, 0.1 M, 500 mL
W0001	Distilled Water, 4 L
Q0005	Oxalic Acid, 100 g

Consult the Flinn Scientific website for current prices.

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