40 LOW-WASTE, LOW-RISK CHEMISTRY LABS

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Dedication

This book is dedicated to my wife, Dawn,
my son, Beryl, my mother, Lila,
and to the hope and promise of the
Maine School of Science and Mathematics.

Acknowledgments

Experiments 3 and 39 were adapted from
Consumer Microscale Chemistry with permission
from Eric Henderson of Presque Isle High School, Presque Isle, Maine.

Disclaimer

Contact your state Department of Environmental Protection, your local wastewater treatment facility, and your school facilities manager before using any of the listed disposal procedures. The procedures outlined in this book represent currently accepted protocols for the disposal of small quantities of laboratory-generated wastes. State and local regulations may be more restrictive. Local wastewater treatment facilities may restrict wastewater discharge. Contact the facilities manager to determine local restrictions.

The author assumes the teacher has had training in laboratory safety, has read the General Information section of the teacher's guide and the specific guides for each experiment, and has conducted each experiment prior to having students conduct the experiment.

Contents

Experiment 13. Microscale: Oxidation States

Time: 40 minutes *Difficulty level:* 2 *Grouping:* single

Materials per student

Part 1

- 6 M sodium hydroxide
- potassium permanganate solution
- hydrogen peroxide solution
- 6-well plate
- 96-well plate
- toothpick
- beral pipet
- distilled water

Part 2

- 0.2 M iron(II) ammonium sulfate
- 0.1 M iron(III) chloride
- 0.1 M potassium ferricyanide
- 0.1 M potassium ferrocyanide
- 6-well plate
- 96-well plate
- toothpick
- beral pipet

Preparation time

Thirty minutes to one hour is required to prepare and test the hydrogen peroxide and potassium permanganate solutions. Approximately one hour is required to prepare the other solutions. Freshly prepared solutions produce the best results.

Preparation

Hydrogen peroxide solution Household hydrogen peroxide (3%) deteriorates with time. Dilute a small volume of H_2O_2 1 100 and test it in the experiment before having students do the experiment. If the results do not compare to the model data table, adjust the dilution to produce comparable results.

Potassium permanganate (3.8×10^{-2} M): The potassium permanganate solution can be prepared by adding 0.6 g of potassium permanganate to enough distilled water to make a 100 ml solution.

Background

This experiment relates color changes to the oxidation states of manganese and iron.

Safety

Six molar sodium hydroxide is corrosive. Any eye contact should be treated with a 15-minute eyewash in a plumbed eyewash station. Medical attention should be provided. Students should wear chemical splash goggles, gloves, and an apron.

Disposal

Add 1 M HCl to the waste NaOH solution from Part 1 until the pH is between 6 and 8; then flush the solution down the drain.

The mass of the waste potassium ferrocyanide is 81 mg per 24 students and the mass of the waste potassium ferricyanide is 63 mg per 24 students. Consult your state regulatory agency and facilities director to determine if this small volume can be disposed of in the wastewater discharge.

Alternatively, the total volume generated by Part 2 is 23 ml per 24 students. This can be labeled and stored as "potassium ferro/ferricyanide waste." Reduce the water volume tenfold by passive evaporation. The net volume for 100 students would be less than 10 ml of 1 M potassium ferro/ferricyanide solution. Label this properly for future disposal.

Pitfalls

Preparation of fresh solutions and use of clean well plates is essential for Part 1.

Typical results

Typical results are shown in the model data and calculations tables. The C1 well in Part 2 may be light blue if the solution is not fresh.

Model Data Table—Part 1

Well	A1	A2	A3	A4	A5
Color	purple	purple	green	brown	brown

Model Data Table—Part 2

Well	C1	C2	C3	C4
Color	(light blue) clear	dark blue	straw	dark blue

Model Calculations Table—Part 1

Well	A1	A2	A3	A4	A5
Ion present	MnO_4^-	MnO_4^-	MnO_4^{-2}	Mn^{-4}	Mn^{-4}
Oxidation state of Mn	+7	+7	+6	−4	−4

Model Calculations Table—Part 2

Well	C1	C2	C3	C4
Ion present	Fe^{++}	Fe^{++}	Fe^{+++}	Fe^{+++}
Oxidation state of Fe	+2	+2	+3	+3

EXPERIMENT 13

Microscale: Oxidation States

Background: Many transition elements can exhibit multiple oxidation states. Iron, for example, can have a +2 or +3 oxidation state. Frequently, the change in oxidation state results in a color change. In this lab the various oxidation states of manganese and iron will be investigated.

Potassium permanganate, $KMnO_4$, is a strong oxidizer and has a characteristic purple color. In solution the permanganate ion (MnO_4^{-}) is formed. The manganate ion (MnO_4^{-2}) is dark green, whereas the Mn^{-4} ion is brown. The hypomanganate ion (MnO_4^{-3}) exhibits a blue color in cold alkaline solutions. Hydrogen peroxide will be used to reduce the various manganese ions.

The two oxidation states of iron can be detected by use of the ferrocyanide ion $Fe(CN)_6^{-4}$ and the ferricyanide ion $Fe(CN)_6^{-3}$. The iron present in the ferrocyanide ion has a +2 oxidation state, whereas the iron present in the ferricyanide group has a +3 oxidation state. A blue precipitate forms when either ion combines with iron in a different oxidation state. For example, if $Fe(CN)_6^{-3}$ is mixed with a solution containing Fe^{+2}, a blue precipitate will form. If it is mixed with a solution containing Fe^{+3}, no precipitate will form. This reaction will be used to demonstrate the oxidation states of iron.

Objective: To demonstrate the color changes associated with the various oxidation states of manganese and iron.

Safety: Wear chemical splash goggles, gloves, and an apron. Sodium hydroxide is caustic. Report any skin or eye contact to your instructor immediately.

Disposal

Part 1· Using a beral pipet, transfer the well contents to the beaker labeled "waste NaOH solution."

Part 2: Using a beral pipet, transfer the well contents to the beaker labeled "waste ferri/ferrocyanides."

Materials

Part 1	Part 2
• 6 M sodium hydroxide	• 0.2 M iron(II) ammonium sulfate
• potassium permanganate solution	• 0.1 M iron(III) chloride
• hydrogen peroxide solution	• 0.1 M potassium ferricyanide
• 6-well plate	• 0.1 M potassium ferrocyanide
• 96-well plate	• 6-well plate
• toothpick	• 96-well plate
• beral pipet	• toothpick
• distilled water	• beral pipet

Procedure

Part 1

1. Obtain a 6-well plate and a 96-well plate from the supply table. Fill well 1 of the 6-well plate three-quarters full of 6 M sodium hydroxide and fill wells 2 and 3 half full with hydrogen peroxide and potassium permanganate solutions, respectively.
2. Using a modified pipet (standard configuration), transfer 10 drops of NaOH to wells A1–A5 of the 96-well plate.
3. Rinse the pipet several times with distilled water. Fill the pipet with potassium permanganate solution.
4. Add 1 drop of potassium permanganate solution to each of wells A1–A5.
5. Stir the solutions with your toothpick.
6. Rinse the pipet several times with distilled water. Fill the pipet with hydrogen peroxide solution. Add 1 drop of H_2O_2 to well A2. To each of wells A3–A5 add 1 drop more than you added to the preceding well. Stir each well after adding the H_2O_2 and record the color formed immediately after stirring.
7. Using a beral pipet, transfer the contents of the well plates to the beaker labeled "waste NaOH solution."

Part 2

1. Add 5 drops of 0.2 M iron(II) ammonium sulfate to wells C1 and C2 of the 96-well plate.
2. Add 5 drops of 0.1 M iron(III) chloride to wells C3 and C4.
3. Add 1 drop of 0.1 M potassium ferrocyanide to wells C1 and C4. Record the results.
4. Add 1 drop of 0.1 M potassium ferricyanide to wells C2 and C3 Record the results.
5. Using a beral pipet, transfer the contents of the well plates to the beaker labeled "waste ferri/ferrocyanides."

Analysis: Using the information contained in the Background section and the rules for assigning oxidation numbers, complete the Calculations Tables.

Data Table—Part 1

Well	A1	A2	A3	A4	A5
Color					

Data Table—Part 2

Well	C1	C2	C3	C4
Color				

Calculations Table—Part 1

Well	A1	A2	A3	A4	A5
Ion present					
Oxidation state of Mn					

Calculations Table—Part 2

Well	C1	C2	C3	C4
Ion present				
Oxidation state of Fe				