Using Greener Labs in High School and General Chemistry Courses

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Advantages of Green Chemistry Experiments

• Safer materials for large classroom size
• More sustainable and environmentally friendly
• Normally require only simple clean-up of waste
• No costly disposal of hazardous waste
• Often use materials students can easily relate to
Theoretical Annual Heavy Metal Waste Savings

- 3.767 million US public high school 10th grade students in 2014\(^1\)
- If an average of just 1 g heavy metal waste per student is saved, this would be 4.15 tons/year

Initial Research

• Development of the first Green Chemistry Laboratory Manual began in 2002
  › Undergraduate research project
  › Introductory high school chemistry

• Focus was to provide greener experiments that covered traditional topics.
  › It was not intended to teach students about green chemistry.

• Nineteen experiments were developed and printed in a student’s manual
  › Dissemination and field testing was started
Lab Criteria

• Require 45 minutes or less to perform
• Reduce or eliminate toxic organic solvents
• Reduce or eliminate hazardous heavy metals:
  › Pb, Ba, Ni, Ag, Co, Cu, Hg, Cr, and Mn
• Cost less than comparable labs
• Re-emphasize the material learned in class
• Incorporate relational/inquiry based learning appropriate for high school students
• Interest the typical high school student
• Use and teach basic lab techniques
Green Chemistry and Tennessee

• Tennessee Department of Environment and Conservation (TDEC) doing 2 fold project
  › Remove hazardous waste and materials from high schools across the region
  › Prevent future acquisition of hazardous materials

• Asked us to develop:
  › Supplementary Teacher’s Manual that included TN Education Standards
  › Permission to post 3 labs on their website
TDEC Green Chemistry Experiment’s Website

Green Chemistry Experiments

Green chemistry in the academic setting is the practice of using chemicals with less hazardous characteristics, thereby generating less hazardous waste. The benefits are a safer lab setting for students, less hazardous storage areas, and lower volumes of waste requiring special disposal. Green chemistry lab exercises often use materials found on grocery store shelves that can be flushed down the sink drain. In cases where acids or bases are left at the end of a lab exercise, directions are given to neutralize or dilute them and flush with running water.

Each set of three labs are part of a complete Green Chemistry Manual (Teacher’s Manual and Student’s Manual) developed and tested by Kimberly Lindsey, Kacey Fowler and Ruth Hall, undergraduate students at Union University’s Department of Chemistry in Jackson, Tennessee. The students worked under the guidance of Dr. Sally Hume, Professor Carol Leslie and Dr. Marilyn Newhouse also contributed to the project. The manual is intended to be used at the high school level. The lab exercises meet all chemistry standards required by the Tennessee Department of Education.

For more information about SC3 contact Ken Mafe, Linda Jordan, or Cynthia Rohrbach.
Teacher’s Suggestions

• **Student’s Laboratory Manual**
  ▶ Experiments covering additional concepts
  ▶ Pre-lab questions associated with the introduction and procedure
  ▶ Safety reminders included in the procedure text
  ▶ Helpful Hints and Cautions in the text
  ▶ Figures of the different apparatuses
Teacher’s Suggestions

• Teacher’s Manual
  › Answers to pre-lab and post questions in text (not appendix)
  › Unified materials section with supplies indexed
  › Large margins for note taking
Experimental Concepts

- Safety
- Significant Figures and Measurements
- Density
- Separation of a Mixture
- Physical and Chemical Changes
- Energy and Calorimetry
- Electron Configuration
- UV Spectrum
- Ions
- Molecular Shapes / Models
- Ionic and Covalent Bonds
- Polar and Nonpolar Bonding
- Types of Chemical Reactions
- Moles: Avogadro’s Number
- Periodic Trends and Uses
- Stoichiometry
- Properties of Acids and Bases
- Titration
- Oxidation-Reduction
- Rates of Reaction and Kinetics
- Thermodynamics
- Gas Laws
- Catalysts
Table of Contents

• Lab 1  Getting Acquainted
• Lab 2  Introduction to Significant Figures and Measurements
• Lab 3  Properties of Matter: Density
• Lab 4  Separation of a Mixture
• Lab 5  Chemical and Physical Changes of Caffeine
• Lab 6  The Calorimetry of Junk Food
• Lab 7  Thermodynamics: Cold Packs vs. Hand Warmers
• Lab 8  Electron Configuration: Chemistry of Fireworks
• Lab 9  Finding a Laser Pointer’s Wavelength of Light
• Lab 10 Magnets, Marshmallows, and Molecular Models
• Lab 11 Sugar and Salt? Ionic and Covalent Bonds
• Lab 12 Chemical Bonding: Polarity of Slime and Silly Putty
Table of Contents

- Lab 13 Qualitative Tests for Fluoride Ions in Mouth Rinses
- Lab 14 Types of Chemical Reactions
- Lab 15 ReDox Reaction: Can Aluminum Become Magnetic?
- Lab 16 The Mole: Avogadro’s Number
- Lab 17 The Periodic Table: Its Trends and Uses
- Lab 18 Stoichiometry: Synthesis of Garden Lime
- Lab 19 Ideal Gas Law: Finding % H₂O₂ with Carrot Juice
- Lab 20 Rates of Reactions
- Lab 21 Acceleration of Reactions by a Catalyst
- Lab 22 Properties of Acids and Bases
- Lab 23 Titration of Acidic Candy
- Lab 24 Who Done It?
Helpful Hints and Cautions

Procedure

**SAFETY FEATURES**
- Safety Gloves
- Safety Goggles
- Corrosive

**MATERIALS NEEDED**
- 0.8 gram of caffeine (C₈H₈N₄O₂)
- Stir rod
- 5 mL 3M hydrochloric acid (HCl)
- 6-7 mL 5M sodium hydroxide (NaOH)
- 1 medium sized test tube
- 1 Petri dish (both top and bottom)
- 10 mL graduated cylinder
- Hot plate

1. After the caffeine crystals obtained from the "Separation of Mixtures" lab have been weighed and recorded in the previous lab data section, divide them in half. Or if they are not available, obtain ~0.8 g of caffeine. You will now use half of the mixture to show a chemical change and half of the mixture to show a physical change.

**Part 1: Physical Change**

2. Place half of the mixture or ~0.4 g of caffeine into the bottom part of a Petri dish and spread it out evenly.

3. Place the top on the Petri dish and place the Petri dish on a hot plate. Gradually increase the heat until the caffeine starts to sublime. **HINT:** Slowly increase the heat setting. If heating occurs too fast, the caffeine will turn brown or only burn and not sublime.

4. Allow the caffeine to sublime until either no more crystals are forming on the top of the Petri dish or the top of the Petri dish can no longer hold all of the crystals. Turn off the hot plate and allow the Petri dish to cool to room temperature. **CAUTION:** Do not try to remove the top of the Petri dish immediately. It is extremely hot and can cause severe injury or release caffeine vapor into the air. **HINT:** To save time you can now go to the Chemical Changes section. After you have completed these steps, come back to step 5 of Part 1.
Teacher’s Manual
Pre-lab Questions

\[ 4\text{Fe} + 3\text{O}_2 \rightarrow 2\text{Fe}_2\text{O}_3 \]

a) Elemental iron (Fe) \(0\)

b) Elemental oxygen (O) \(0\)

c) One iron atom in \(\text{Fe}_3\text{O}_4\) \(+3\)

d) One oxygen atom in \(\text{Fe}_3\text{O}_4\) \(-2\)

2. Which element was oxidized and which element was reduced in the above reaction equation?

The iron (Fe) was oxidized, while the oxygen (O) was reduced.

3. From the Activity Series of Metals, determine the order of reactivity of the following metals: Ni, Au, Fe, Ca, Zn, and Al.

Most Reactive → Least Reactive

\(\text{Ca} \rightarrow \text{Al} \rightarrow \text{Zn} \rightarrow \text{Fe} \rightarrow \text{Ni} \rightarrow \text{Au}\)
<table>
<thead>
<tr>
<th>Preparation Materials Table</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>100 mL HCl (per group)</strong></td>
</tr>
<tr>
<td><strong>Hydrochloric acid</strong></td>
</tr>
<tr>
<td><strong>Prep:</strong> 8.3 mL concentrated HCl (37%) per 100 mL H₂O</td>
</tr>
</tbody>
</table>
Green Chemistry
Labs’ Website

"Green Chemistry is the design of chemical products and processes that reduce or eliminate the use and generation of hazardous substances" as defined by the American Chemical Society.

Green Chemistry Labs is a manual containing twenty-four (24) experiments targeted at the high school level of education. It was developed by Dr. Sally Henrie, Kacey Fowler, Ruth Hall, and Kimberly Lindsey at Union University over multiple years. Dr. Marilyn Newhouse and Prof. Carol Leslie also contributed to the project.

Take a look around this site to get a feel for what our labs are all about and find out if they're right for your classroom.

Student’s Laboratory Manual

- Pre-lab questions associated with the introduction and procedure
- Safety reminders included in the procedure text
- Helpful Hints in the text
- Figures of the different apparatuses
- Experiments covering additional concepts

Teacher’s Manual

- Answers to pre-lab and post questions in text (not appendix)
- Unified materials section with supplies indexed
- Large margins for note taking

Lab Manual Features

Homeschool and Web-based Introductory and General Chemistry Laboratory Courses

- Manuals and accompanying kits for home school students and web-based college introductory and general chemistry courses are now available at eScience Labs.
- eScience Labs will design Custom Chemistry Kits to meet your specific needs.

Green General College Laboratory Manual

- The Green Chemistry Laboratory Manual for General Chemistry is available for college and AP chemistry courses from CRC Press Taylor and Francis Group.
- This manual teaches students about green chemistry and how to use it.

Workshops

- Day-long hands-on inservices/workshops available upon request.
Experiment Example: Sugar or Salt? Ionic and Covalent Bonding

Objectives

- Define ionic and covalent bonding and determine the differences between each
- Observe and link the physical properties of sugar and salt to ionic and covalent bonding
- Properly characterize the type of bonding for sugar and salt
Experiment Example: Sugar or Salt? Ionic and Covalent Bonding

- Two part procedure:
  - Part 1. Simple anodizing apparatus to observe ionic and covalent bonding differences in conducting of electricity
  - Part 2. Melting Temperature Differences

Figure 1. Apparatus for Part 1
Experiment Example: The Periodic Table: It’s Trends and Uses

• Objectives
  › To understand the Periodic Table and its uses.
  › To relate the characteristics within a group on the Periodic Table.
  › To introduce using moles in a reaction
Experiment Example:
The Periodic Table: It’s Trends and Uses

• Overview

› Equal molar amounts of Li$_2$CO$_3$, Na$_2$CO$_3$, and K$_2$CO$_3$ (Group I) are placed into balloons and attached to flasks containing 1M HCl.

› Equal gram amounts of Li$_2$CO$_3$ and K$_2$CO$_3$ are also compared.

› As each reaction proceeds, CO$_2$ gas is given off.
Exploring Web-based and Homeschool

- Administration looked at contingency plans if major disaster caused campus shut down
- Dean’s office considered how labs could be accommodated
- Researched how to revise lab manual for web-based course and later for homeschool
- Worked mentioned by Linda Wang in 4-16-07 C&EN article “Science of Homeschooling”
- Led to eScience Labs contacting us

• eScience Labs wanted to create a chemistry kit that used green experiments
• Worked with them to develop:
  › Initially: Introductory Chemistry kit
  › Later: General Chemistry kit

12 Principles of Green Chemistry

- Prevention
- Atom Economy
- Less Hazardous Chemical Syntheses
- Designing Safer Chemicals
- Safer Solvents and Auxiliaries
- Design for Energy Efficiency
- Use of Renewable Feedstocks
- Reduce Derivatives
- Catalysis
- Design for Degradation
- Real-Time Analysis for Pollution Prevention
- Inherently Safer Chemistry for Accident Prevention
The Next Step

• Educational materials are needed that teach students how to utilize green chemistry.¹

• Teaching students how to apply green chemistry within a traditional course appears to work best in a laboratory setting.

• In 2011, an undergraduate research project was begun to develop a Green Chemistry Laboratory Manual for the AP/General chemistry level.

• It is published by CRC Press, Taylor & Francis Group

Why Target General/AP Chemistry?

• General/AP chemistry is taken by most science and engineering students.
• It is their foundational chemistry course.
• This provides an excellent opportunity for students to learn to Think green which they can continue to build on.
Goals

• Previously established goals
  › Reduce or eliminate hazardous chemicals
  › Teach traditional material
  › Use and teach basic laboratory techniques
  › Incorporate relational/inquiry based learning
  › Interest typical general/AP chemistry student

• Added goals
  › Educate students on how to utilize green chemistry
  › Impart a vision to students to Think green
How?

• **Start with the basics**
  - Educate students:
    - “Green Chemistry is the design of chemical products and processes that **reduce or eliminate** the use and generation of hazardous substances.”
  - Introduce the 12 Principles of Green Chemistry
  - Emphasize safety and include equipment and common laboratory procedures

Experiments

• Consistent Format
  › Introduction
  › Objectives
  › Pre-lab questions
  › Procedure
  › Data
  › Observations
  › Calculations
  › Analysis
  › Think green
  › Presidential green chemistry challenge
Experimental Concepts

- Percent of water in a hydrate
- Formula of a compound
- Mass & mole relationship
- Molar mass by vapor density
- Molar volume of a gas
- Enthalpy
- UV-visible spectroscopy
- Chromatography
- Freezing point depression
- Standardization of a solution
- Acid-base titrations
- Acid-base titration indicators
- Buffer solutions
- Reaction rate and its order
- Equilibrium constant
- Oxidation-reduction titration
- Qualitative analysis
- Electrochemical series
- Voltaic cells
- Coordination compound synthesis
- Organic synthesis
Educate Students on How to Utilize Green Chemistry

• This goal is incorporated throughout each experiment.
  › The introductory material integrates green chemistry concepts and applications.
  › Prelab, at times, includes green topics such as atom economy; last question always asks students to determine hazards.
  › In *Think Green*, students often compare the procedure they used to traditional ones or try to develop greener methods and test their ideas.
## Educate Students on How to Utilize Green Chemistry

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Green chemistry principles</th>
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</thead>
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<tr>
<td>Determining the percent water in Epsom salt</td>
<td>Design for energy efficiency</td>
</tr>
<tr>
<td>Determination of the formula of a copper supplement</td>
<td>Prevention, catalysis</td>
</tr>
<tr>
<td>Determination of mass and mole relationships in a chemical reaction</td>
<td>Atom economy</td>
</tr>
<tr>
<td>Finding ethanol’s molar mass using vapor density</td>
<td>Prevention, Designing safer chemicals, Use of renewable feedstocks, Design for degradation</td>
</tr>
<tr>
<td>Determining molar volume of a gas at STP</td>
<td>Prevention</td>
</tr>
<tr>
<td>Determining the enthalpy change for solvation reactions</td>
<td>Prevention, Inherently safer chemicals for accident prevention</td>
</tr>
</tbody>
</table>
## Educate Students on How to Utilize Green Chemistry

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Green chemistry principles</th>
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<tbody>
<tr>
<td>Synthesis and chemical analysis of sodium iron (III) EDTA</td>
<td>Prevention, Atom economy, Safer solvents and auxiliaries, Design for energy efficiency, Reduce derivatives, Real-time analysis for pollution prevention, Inherently safer chemistry for accident prevention</td>
</tr>
<tr>
<td>Analysis of Biodiesel synthesized from canola oil</td>
<td>Design for energy efficiency, Use of renewable feedstock, Catalysis, Design for degradation, Real-time analysis</td>
</tr>
</tbody>
</table>
Imparting Importance of a Vision to “Think Green”

- Presidential Green Chemistry Challenge
Instructor’s Manual

- Format for each experiment includes:
  - Green chemistry principle(s) investigated
  - Introduction - relates green chemistry to chapter
  - Experimental considerations
  - Unified materials section with supplies, preparation and alternatives
  - Notes area
  - Example answers to questions and data for student hand in pages in a text format, not an appendix
  - Think green information
Experiment Example: Determination of the Formula of a Copper Supplement

Objectives

- Introduce determining formula of a compound
  - Student’s calculate the chemical formula of copper (II) gluconate
- Incorporate green chemistry principles:
  - Prevention
  - Catalysis

Experiment Example: Determination of the Formula of a Copper Supplement

- Procedure Overview:
  - Dissolve 0.500 g copper gluconate in 10 mL 1% NaCl
  - Plate copper onto three Al washers
  - Recover isolated copper from washers
  - Dry recovered copper
  - Calculate mass and moles of recovered Cu
  - Determine the formula of the compound
Experiment Example:
Determination of the Formula of a Copper Supplement
Experiment Example:
Determination of the Formula of a Copper Supplement

• Analysis Example

1. Create a pie chart showing the percentage composition for each element in the compound copper gluconate; clearly label each element and the percentage.

![Pie Chart]

Percent Composition

- Oxygen: 49.35%
- Carbon: 31.76%
- Copper: 14.00%
- Hydrogen: 4.89%
Think Green
Determination of the Formula of a Copper Supplement

• In Lab Inquiry:
  › Students develop a method to determine chemical formula for Cu(II)SO$_4$·5H$_2$O
  › Test procedure if time and resources permit
  › Evaluate impact on 12 Principle’s of Green Chemistry

• Research:
  › Compare % copper in copper gluconate to Cu(II)Cl$_2$ and discuss this method’s advantages
Presidential Challenge
Determination of the Formula of a Copper Supplement

• Presidential Green Chemistry Award:
  › 2009 in Academics Category
  • Professor Krzysztof Matyjaszewski
  • Developed copper catalyst with lower % copper

www2.epa.gov/green-chemistry; challenge award winners
Experiment Example:
Synthesis of Sodium Iron (III) EDTA

- Objectives
  - Introduce coordination chemistry
  - Incorporate green chemistry principles:
    - Real time analysis
    - Solventless reaction
    - Atom economy and E-factor
    - Microwave for energy efficiency

Jones, M. T. and Henrie, S. A., Incorporating green chemistry principles into general chemistry labs for organic synthesis, coordination compounds and determination of an equilibrium constant, ASC National Spring meeting, 2014.
Experiment Example: Synthesis of Sodium Iron (III) EDTA

• Ferric ammonium sulfate was chosen as the iron source.

Dark blue: Stock sodium ferric EDTA;
Tested iron sources: Ferric ammonium sulfate (light blue);
Ferric chloride (pink); Ferric sulfate (red)
Experiment Example:
Synthesis of Sodium Iron (III) EDTA

• Procedure uses two reactions:
  1. Titration:
     › Uses EDTA disodium titration
     › Synthesis completion correlates to pH increase
     › Real time analysis
  2. Solventless:
     › Involves ferric acetate intermediate
     › Microwave

• Students compare atom economies, E-factors, % yields and energy efficiency
Experiment Example:
Synthesis of Sodium Iron (III) EDTA

1. Titration (real time analysis):
   › First see color change correlates to pH increase
   › Next synthesize larger quantity using only pH meter
Experiment Example:
Synthesis of Sodium Iron (III) EDTA

2. Solventless:
   › Uses ferric acetate intermediate
Experiment Example:
Synthesis of Sodium Iron (III) EDTA

- Results:

<table>
<thead>
<tr>
<th>Method</th>
<th>% Yield</th>
<th>Atom economy</th>
<th>E-factor</th>
<th>Time heated/method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titration</td>
<td>58.26%</td>
<td>49.28%</td>
<td>23.15</td>
<td>27 minutes/hot plate</td>
</tr>
<tr>
<td>Solventless</td>
<td>69.48%</td>
<td>38.79%</td>
<td>17.48</td>
<td>1.5 minutes/microwave</td>
</tr>
</tbody>
</table>
Think Green

Synthesis of Sodium Iron (III) EDTA

• Research:
  › A coordination compound
  › Solvent purification or disposal
  › Green chemistry metrics
  › Microwave reactors
Presidential Green Chemistry Challenge

- Presidential Green Chemistry Award:
  - 2009 in Green Synthetic Pathways
    - Eastman Chemical Company
    - For their solvent-free process to produce esters used in cosmetics and personal care products

www2.epa.gov/green-chemistry; challenge award winners
Conclusions

• Completed:
  › The Green Chemistry Laboratory Manual for High School and Introductory College Chemistry
  › Instructor’s Manual
  › eScience Lab kits

• Manual Available through:
  › www.greenchemistrylabs.com
Conclusions

• Completed:
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• Manual Available through:
  › CRC Press
    • Taylor & Francis Group
  › Amazon
Future Goals

• Incorporate green chemistry education into introductory manuals
• Complete and make available introductory college student and instructor’s manuals
• Monitor results and make indicated improvements
• Develop additional green labs and demonstrations
• Continue to promote the use of green chemistry labs through presentations, in-services, and workshops
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