Green Chemistry

Laboratory Manual for General Chemistry

Sally A. Henrie

Kathleen Cooper
Kelsey Denny
Rachael Harris
Samantha Howard
Michael Jones
Patrick Jones
Kelsie Wood

CRC Press
Taylor & Francis Group

Green Chemistry

Laboratory Manual for General Chemistry

Sally A. Henrie
Union University
Jackson, Tennessee, USA

Kathleen Cooper
Kelsey Denny
Rachael Harris
Samantha Howard
Michael Jones
Patrick Jones
Kelsie Wood

CRC Press
Taylor & Francis Group
Boca Raton London New York

CRC Press is an imprint of the
Taylor & Francis Group, an **informa** business

CRC Press
Taylor & Francis Group
6000 Broken Sound Parkway NW, Suite 300
Boca Raton, FL 33487-2742

Printed on acid-free paper
Version Date: 20141218

International Standard Book Number-13: 978-1-4822-3020-8 (Paperback)

Library of Congress Cataloging-in-Publication Data

Henrie, Sally A., 1953-
Green chemistry laboratory manual for general chemistry / Sally A. Henrie.
pages cm
"A CRC title."
Includes bibliographical references and index.
ISBN 978-1-4822-3020-8 (alk. paper)
1. Green chemistry--Laboratory manuals. 2. Green chemistry--Study and teaching (Higher) 3. Chemistry--Laboratory manuals. 4. Chemistry--Study and teaching (Higher) I. Title.

TP155.2.E58H46 2015
540.78--dc23 2014038223

Visit the Taylor & Francis Web site at
http://www.taylorandfrancis.com

and the CRC Press Web site at
http://www.crcpress.com

Green Chemistry

Laboratory Manual for General Chemistry

Preface

I was impressed by **green chemistry** the very first time I was introduced to it in 2001 by Dr. Mary Kirchhoff at the American Chemical Society National Spring meeting. I was so captivated by it that right after this, I began working with undergraduate students on research to develop manuals that used greener chemicals and methods while still teaching the traditional material typically covered. As the research progressed, I gained a desire to not only develop greener experiments, but also introduce students to green chemistry in a tangible way that challenges them to embrace its vision. I hope that students will become captivated by it like I am as they realize green chemistry is a new way of actually doing chemistry. I want them to grasp that it requires creativity, innovation, and ingenuity to design novel ways to create and synthesize products and to implement processes that will eliminate or greatly reduce the environmental impact, and to be challenged by this. It was with these goals in mind that the *Green Chemistry Laboratory Manual for General Chemistry* was created.

To realize these goals, green chemistry principles are discussed in the introductory material and applied to the experiments that will be performed. After they have completed the procedure and analyzed their results, students are challenged in a **Think Green** inquiry section to consider what principles of green chemistry are positively impacted and to research particular relevant topics. Often in this section they are encouraged to develop a method based on what they learned and then to try their ideas. At the end of each chapter is the **Presidential Green Chemistry Challenge** section. From this, students are able to learn about how the green chemistry principles are actually applied in our world. Students are asked to look up a Presidential Green Chemistry Challenge award that relates to what was studied in the chapter and summarize what was accomplished to receive this award.

But why was the general chemistry laboratory chosen to introduce students to green chemistry principles in this way? The general chemistry laboratory is an excellent place to inspire students to learn to think green.

Many of these students will continue on in science or engineering professions and work in these areas to contribute to society. Teaching them to learn to apply the principles of green chemistry at the start of their college science education will allow them more time to firmly establish a foundation in the principles of green chemistry that they can later use in their future careers.

Acknowledgments

I would like to thank all of the people who worked to create this laboratory manual, and wish I could mention everyone. It could not have been completed without the expertise, contributions, help, and encouragement of many individuals. Most of all, I would like to thank my husband, Ray, and my family for their patience, support, and encouragement. I am especially grateful to all of the research students who have worked diligently with me to develop greener chemistry manuals. Those who worked on this particular manual are Kathleen Cooper, Kelsey Denny, Rachael Harris, Samantha Howard, Michael Jones, Patrick Jones, and Kelsie Wood. I am also appreciative for the help, expertise, and encouragement from my colleagues and students at Union University, especially Dr. Randy Johnston, Dr. Marlyn Newhouse, and Giley Wright. I am most grateful to all of the dedicated and talented people who helped in the production of this manual at CRC Press, Taylor & Francis Group. I am especially appreciative of Hilary Rowe, acquisitions editor, chemistry, for making a way and encouraging me to publish this manual; to Judith Simon, project editor; and Jill Jurgensen, senior project coordinator, for all their efforts to bring this manual to print.

Introduction: Why green chemistry?

A laboratory procedure you are working on requires you to measure 20.0 ml of concentrated sulfuric acid. Someone bumps you while you are doing this and the acid goes everywhere, including on your skin and clothes. You rush to the safety shower but still end up with some chemical burns, a trip to the emergency room, and totally ruined clothes!

This lab manual has been designed with "greener" procedures so that an accident like the one described above will not happen. Suppose this experiment had been conducted using vinegar instead. You would just rinse off your skin, attempt to dry your clothes with a paper towel, and then spend the rest of the day joking with your friends about how you smelled like Easter egg dye. Or better yet—what if instead the procedure could be modified to use a reusable acid catalyst anchored on a solid?

This green chemistry lab manual was developed not just to prevent accidents and make lab settings safer for you, but *also* to teach you about green chemistry. There are many exciting innovations that have happened because of green chemistry, and even more being developed. While working through the experiments in this manual you will learn about some of them, and hopefully begin to grasp the vision of what thinking as a green chemist really means.

But what exactly is green chemistry? The Environmental Protection Agency defines green chemistry as "the design, development, and implementation of chemical products and processes to reduce or eliminate the use and generation of substances hazardous to human health and the environment." This means green chemistry is not about just trying to find ways to treat hazardous materials used or produced or to develop better protective equipment. Instead, it seeks innovative ways to *reduce* or even eliminate hazards from the start. The word *reduce* is in italics to make the point that just because a process is greener, it does not mean that all hazards have been eliminated. But it does mean that they have been significantly reduced in some way.

There was not really a description for green chemistry until the mid-1990s, when Paul Anastas and John Warner created a list of 12 criteria or principles that can be used when designing chemical processes (*Green Chemistry: Theory and Practice*, 1998, Oxford University Press, NY, p. 30). These are known as the *12 principles of green chemistry*. These 12 principles and a brief explanation of each are listed below.

1. **Prevention**: It is always better to prevent hazardous waste than to have to clean it up once it has already been created. It is also better to design a process to be safe instead of having to figure out ways to protect people from toxic chemicals being used or dangerous processes.
2. **Atom economy**: If you build a bookcase, you want to use as much of the wood purchased as possible and not have anything wasted. You may consider different plausible designs and see which one has the least waste and will work the best. Methods of synthesis should be designed in a similar manner—you want as many atoms as possible from the starting materials to be incorporated into the final product.
3. **Less hazardous chemical synthesis**: When you think about chemistry, you may imagine someone in a yellow hazmat suit working with some kind of chemical that has a skull and crossbones on the bottle. Green procedures attempt to eliminate dangerous chemicals like these and use materials that have little or no danger to human health and the environment.
4. **Designing safer chemicals**: If you could design two products that did the same thing—one that could cause cancer and one that would not—which one would you make? The answer to that is obvious—the one that is less toxic! Products of chemical reactions should do what they are designed to, while having minimal toxicity.
5. **Safer solvents and auxiliaries**: Using solvents (liquids used to dissolve other materials), separating agents (chemicals that help two other things separate completely), or other auxiliaries should be cut out of a procedure as much as possible. But, if they are really needed, they need to be as harmless as possible.
6. **Design for energy efficiency**: You're probably very familiar with this principle if you have ever gone shopping for a TV or refrigerator. Most appliances advertise how energy efficient they are, or how much energy you can save by using them. But, you probably have not thought about energy efficiency in the chemistry lab. A chemical reaction should be designed to occur at room temperature and normal pressure, if possible, to save energy. If heating is necessary, it should be designed to be done as efficiently as possible. This could even involve using microwaves for heating!

7. **Use of renewable feedstocks**: We place a higher value on something if it is limited, and try to protect it. This is the same way we should treat our natural resources. Whenever possible, a *renewable* raw material should be used rather than depleting a limited stock of another material.
8. **Reduce derivatives**: Sometimes in chemistry, certain chemicals are used to protect certain parts of a molecule from reacting. Doing this or temporarily modifying a product should be done as little as possible because it generates additional waste.
9. **Catalysis**: A catalyst is something that speeds up the rate of a reaction, but is not used up in the reaction. You can get it back after the reaction has occurred and use it again. A selective catalyst should be used when possible.
10. **Design for degradation**: Some chemicals stick around and build up in the environment, which can cause damage to the ecosystem. The end products of a chemical reaction should break down instead of persisting in the environment.
11. **Real-time analysis for pollution prevention**: Thermostats constantly monitor the temperature of an area to ensure that it stays at just the right temperature. If the room starts to become just a little too hot or too cold, it sends a signal for the heat or air unit to come on. Once the set temperature is reached, it signals it to stop. In the same way, methods involving chemicals should be constantly monitored to prevent the formation of hazardous products, optimize yields, and decrease chemical waste.
12. **Inherently safer chemistry for accident prevention**: Even though safety measures are put in place to prevent accidents, they still happen in the lab—you drop a beaker, a bottle rolls off of the counter, you spill an acid, etc. When an accident occurs, you want it to cause as little damage as possible. This is desired not just for a lab setting, but also for manufacturing that is going on every day that uses chemistry. The chemicals used, all products of the reactions, and the processes should be designed to be as safe as possible and to prevent accidents. This way, even if an accident occurs, it is not as serious as it could have been since the process was designed to make it as safe as possible.

The authors of this manual tried to make labs that would be more interesting to you while incorporating as many of the above principles into each experiment as possible. With all of these steps taken to make the labs safer, they should be completely safe, right? You should be able to do a chemical experiment on the counter, lay a sandwich down on it, and be able to eat it a few minutes later, shouldn't you? Your lab partner can drink what is in a beaker, and nothing should happen to him, right? Wrong!

Many have the misconception that green chemistry experiments are completely without risk. A procedure can be described as green if it makes an improvement in any of the 12 principles. So, conducting procedures in a green way does not make safety precautions irrelevant. Rules such as not eating or drinking in the lab, wearing safety goggles, and avoiding horseplay are still crucial. Once again, even though these experiments are designed to be safer, precautions still need to be observed. Vinegar may be much safer than sulfuric acid, but that does not mean you should be careless when using it!

Green chemistry has often been referred to as preventive medicine for the environment. Heavy metals and other toxic materials have to be put in special containers, sealed up, and taken to special facilities carefully designed to prevent them from entering the environment. If they are not disposed of properly, they can cause illness and environmental disasters. An advantage for you, your instructor, and the environment is that the products of each of these labs normally do not have to be disposed of in this special way. Often it is safe to pour solutions down the drain (with plenty of running water), and solid waste can often be thrown directly in the trash can. Even if the waste does need to be disposed of in a special manner, it will be one used for less hazardous chemicals. This may not seem like such a big deal to you, but if schools across the nation began doing procedures in a green way, it could eliminate tons of hazardous waste and reduce the environmental cost of chemistry education.

Laboratory safety, equipment, and procedures

A green chemistry laboratory manual means you can't get hurt, right? Wrong!

While this laboratory manual has been designed with greener procedures to cut down on harmful reagents and waste, there are still hazards and risks. Accidents may occur, even using greener procedures. However, if you use appropriate safety practices, a laboratory can be a reasonably safe place. Some common safety procedures you should follow are listed below.

Using glassware

- *Do not use glassware that is cracked.* The glassware may break upon heating or using.
- Clean all broken glassware immediately and dispose of it in the appropriate receptacle.
- *Keep all glassware clean.* Do not store dirty equipment.

Handling laboratory reagents

- *Always be aware of what you are doing.* Study the experiment and know the chemical and other hazards before entering the lab. A good way to find out about a chemical's hazards is to look up and study its (material) safety data sheet ((M)SDS).
- Report hazardous chemical spills to the instructor when they occur. Chemical spills need to be cleaned up immediately and properly.
- Use a fume hood for all chemicals that may produce hazardous or irritating gases or vapors.

- *Keep flammable liquids away from flames or exposed wiring.* If a small fire in a beaker occurs, turn off the source that caused the fire and cover the beaker with a watch glass. Be sure to tell your instructor about it. If a larger fire starts, tell your instructor immediately. Depending on the size of the fire and what is burning, it may be necessary to evacuate immediately.
- Always pour concentrated acids into water, never water into acid. Heat caused by the reaction may cause splattering.
- *Clean up messes when they occur.* If you are unsure of how to properly handle a chemical spill, ask your instructor.
- *Keep reagents pure by not contaminating them through using dirty utensils or glassware.* Not only will your reagents become contaminated, but undesirable reactions, even explosions, may occur.
- *Dispose of waste properly.* Follow the instructions given to you on how to dispose of your chemical waste by your instructor. These vary according to how your lab has been designed to accommodate chemical wastes and your location's regulations.
- If a corrosive liquid or any laboratory chemical used in this manual gets on your skin, immediately rinse the area with water and alert your instructor.
- Do not inhale large quantities of the reagents when checking the odor. Gently waft vapors toward your nose.
- Never eat or drink anything that has been in the lab, including reagents or samples. Eating, drinking, and smoking are not permitted in any laboratory setting.

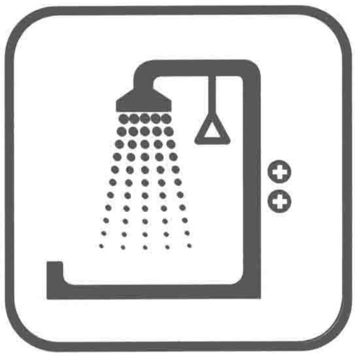
Laboratory attire

- *Wear safety goggles at all times.* It is possible there may be flying debris from glassware breaking and spilled chemicals. If you wear contacts, you should remove them. Splashed chemicals and noxious gases could cause more damage if you are wearing them.
- Long, loose hair should be tied back.
- Wear closed-toe shoes at all times.
- *Wear clothes that give appropriate coverage for protection.* Try to avoid wearing loose-fitting or very flammable clothing.

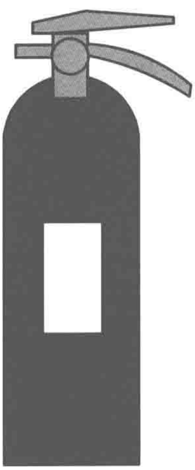
Safety equipment

- Know when to use and how to use all safety equipment.
- Know the location and purpose for all safety equipment, including the eye wash station, safety shower, and fire extinguisher. Illustrations of some common safety equipment are shown below:

Eye wash station

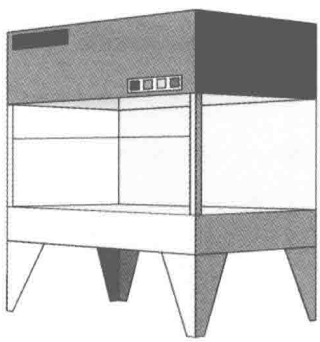


Safety shower with pull chain

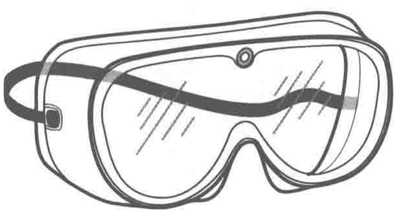


Fire extinguisher

Fire blanket



Laboratory fume hood



Safety goggles