CHEMISTRY

An Industry-Based Laboratory Manual

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Preface

Have you ever sat down and had an open discussion over coffee with an industrial chemist or chemical technician about his or her work? Have you ever "shadowed" a chemist or a chemical technician for a day as he or she went about the daily routine in an industrial or government laboratory? If you have done these things, you were likely surprised at how foreign the language seemed or startled at how unfamiliar the surroundings were, despite what you perceived as your thorough academic grounding in the principles of chemistry. Is there any talk of the quantum mechanical model of the atom? No. Is there any activity relating to the molecular orbital theory of bonding? No. Is there any use made of your knowledge of Lewis acids and bases? Probably not. Is the lab a large room with six 12-ft lab benches capable of accommodating up to two dozen chemists? No, not usually. The labs are typically small, with from one to six lab workers in each.

Do you hear anything about safety in the laboratory. Yes! In fact, if you visited a lab, you probably had to go through some sort of safety check, such as reading basic safety guidelines or the viewing of a safety video, prior to even entering the facility. You probably also had to "sign in" and be issued personal protective equipment, such as safety glasses and a lab coat. If you later followed a chemist into the production facility to take samples, you may have been issued a hard hat or perhaps a small white disposable stretch-cap to cover your hair.

If you toured a lab facility, you probably toured a "wet lab," a "quality control lab," or perhaps a "process development lab," or maybe all of the above and wondered what these terms meant. You probably saw a control chart and wondered what it was. You may have sat in on a meeting to prepare for the upcoming quality assurance audit and wondered what an audit actually was or what GLP, MSDS, and SOP meant. You may have heard someone talk about certified reference materials and wondered what that was. You may have encountered a formal means of disposing of chemical waste and said, "Wow!" Or you may have noticed an experiment or an instrument that wasn't working properly and, subsequently, observed chemists and technicians teaming together for troubleshooting.

Yes, unless your course in chemistry was "industry-based," you probably came away from this experience with a thought that went something like this: "Wow, why don't textbooks and lab manuals do a better job of communicating what these real-world chemists do?"

The intent of the author of the lab manual you hold in your hands is to do just that. Students who progress through this lab manual will get a heavy dose of laboratory safety issues as they exist for the real-life chemist. Students who use this lab manual will keep records much like an industrial chemist keeps records and learn how important communication is to the chemist. Students who use this lab manual will learn what a control

chart is, what certified reference material is, what is meant by GLP and MSDS, and what an SOP is all about. Students will read examples of what communications may occur between one industrial chemist and another and what is required to report the results of a laboratory analysis to a client. They will learn what an "out-of-spec" or "off-color" product is and how it may be studied. And, they will learn how to go about identifying unlabeled materials.

Students who use this lab manual will critically examine the labels on consumer products and commercial chemical products for safety information. They will attempt to solve problems for chemical companies and research proposed new industrial processes and laboratory methods. They will analyze materials and consumer products to provide third-party answers to industrial problems. They will participate in proficiency testing and identify a waste acid that is designated for disposal. Students also will learn how to think critically in order to apply chemistry principles to solve these various problems and to report to a client. Students even play the role of industrial chemists that become involved in National Chemistry Week activities.

Students who use this lab manual are laboratory technicians employed by a fictional consulting firm called the I.O.N.S. Corporation. The I.O.N.S. Corporation is directed by a chief executive officer by the name of Claire Hemistry, or C. Hemistry. The I.O.N.S. Corporation has a safety officer by the name of Ben Whell who issues a safety report for each project for which special safety considerations may be an issue. The instructor is the laboratory supervisor. Ms. Hemistry enters into contractual agreements with fictional clients that are faced with the problem or situation at hand. Industrial and academic consultants write memos giving the I.O.N.S. technicians the necessary background information. One of the consultants, the client, or Dr. Hemistry provides a standard operating procedure (SOP), or other procedure, that must be executed in order to solve the problem. The students then perform the lab work to solve the problem, keep a laboratory notebook according to company protocols, and write a report memo to the client on I.O.N.S. stationery reporting their results and recommendations, if appropriate.

Students do not know the outcome of the work before they start. In that sense, they are "discovery" experiments in which they do the work in order to "discover the answer" for the client. I hope both instructors and students will have as much fun working their way through this manual as I have had preparing it.

John Kenkel
Southeast Community College

Author

John Kenkel is a chemistry instructor at Southeast Community College in Lincoln, NE. He has a Master's Degree in chemistry from the University of Texas at Austin (1972) and a Bachelor's Degree in chemistry (1970) from Iowa State University.

Throughout his 23-year career at SCC, Kenkel has been directly involved in the education of chemistry-based laboratory technicians in a vocational program called Environmental Laboratory Technology. He also has been heavily involved in chemistry-based laboratory technician education on a national scale, having served on a number of American Chemical Society committees, including the Committee on Technician Activities and the Coordinating Committee for the Voluntary Industry Standards project. Besides these, he has served a 5-year term on the ACS Committee on Chemistry in the Two-Year College, the committee that organizes the Two-Year College Chemistry Consortium ($2YC_3$) conferences. He chaired this committee in 1996.

Kenkel has authored several popular textbooks for chemistry-based technician education. *Analytical Chemistry for Technicians* was first published in 1988. A second edition of this book was published in 1994. In addition, he has authored two other books, one entitled *Analytical Chemistry Refresher Manual* in 1992 and another entitled *A Primer on Quality in the Analytical Laboratory*, which was published in 2000. All were published through CRC Press/Lewis Publishers.

Kenkel has been the Principal Investigator for a series of curriculum development project grants funded by the National Science Foundation's Advanced Technological Education Program. A textbook (*Chemistry: An Industry-Based Introduction*) and this manual (*Chemistry: An Industry-Based Laboratory Manual*) were produced under these grants. He also has authored or coauthored four articles on the curriculum work in recent issues of the *Journal of Chemical Education* and has presented this work at more than a dozen conferences since 1994.

In 1996, Kenkel won the prestigious National Responsible Care Catalyst Award for excellence in chemistry teaching sponsored by the Chemical Manufacturer's Association.

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The National Science Foundation support allowed a number of people to become involved in many different ways. The following attest to this.

Susan Rutledge, adjunct faculty member at Southeast Community College, very ably served as project assistant throughout. In this role, she researched and field tested experiments, developed and maintained a directory of chemical technology faculty members nationwide, created a valuable index for the American Chemical Society's (ACS) Voluntary Industry Standards document, and cheerfully accepted and accomplished many a menial task for the good of the project. She deserves much credit.

Don Mumm of Southeast Community College contributed two of the experiments and Karen Wosczyna-Birch of Tunxis Community–Technical College contributed one.

There were four individuals who were members of an advisory board for the project. It was at a meeting of this board that the concept of the consulting firm, later to be named the Innovative Options and New Solutions Corporation, or the I.O.N.S. Corporation, was hatched. The advisory board members included the following individuals (listed in alphabetical order):

John Amend, *Montana State University*
Onofrio Gaglione, *New York City Technical College (retired)*
Paul Kelter, *University of North Carolina/Greensboro*
Kathleen Schulz, *Sandia National Laboratory*

There were six very faithful colleagues who graciously field tested many of the experiments over a period of 1 or 2 years. These folks (listed below in alphabetical order) were very free with both criticism and encouragement and contributed greatly to the manuscript's quality.

Dale Buck, *Cape Fear Community College*
Paul Grutsch, *Athens Area Technical Institute*
Aniruddh Hathi, *Texas State Technical College*

Leslie Hersh, *Delta College*

Joseph Rosen, *New York City Technical College*

Karen Wosczyna-Birch, *Tunxis Community–Technical College*

There were several colleagues who agreed to perform a last-minute detailed review of the experiments and their input also was very important. These include Kelter, Gaglione, Buck, Grutsch, Hersh, and Wosczyna-Birch, who are listed above, and also the following:

Ken Chapman, *American Chemical Society (retired)*

Susan Marine, *Miami University/Middletown*

The grant from the E.I. DuPont DeNemours Company funded a conference, later to be dubbed "The DuPont Conference," at which colleagues from the chemical industry presented their thoughts and ideas as to what elements should be included in an industry-based laboratory manual. The participants' enthusiasm for the project and their advice were extremely important. Following is a list of the participants in alphabetical order.

Deb Butterfield, *Eastman Kodak*

Ed Cox, *Procter and Gamble*

Sue Dudek, *Monsanto*

Ruth Fint, *DuPont*

Charlie Focht, *Nebraska Agriculture Laboratory*

Dennis Marshall, *Eastman Chemical Company*

Dan Martin, *LABSAF Consulting*

Ellen Mesaros, *DuPont*

Jerry Miller, *Eastman Kodak*

Connie Murphy, *The Dow Chemical Company*

Karen Potter, *University of Nebraska*

Richard Sunberg, *Procter and Gamble*

Fran Waller, *Air Products and Chemicals*

Gwynn Warner, *Union Carbide*

The project is indebted to Dan Martin (listed above) who graciously provided the basis for the Safety Manual used in this book. Another important safety resource that proved helpful is the Laboratory Safety Institute, Natick, MA (Web site: http://www.lab-safety.org/).

Others who assisted by reviewing various drafts of experiments along the way include the following:

Clarita Bhat, *Shoreline Community College*

Robert Hofstader, *Exxon Corporation (retired)*

Lynn Melton, *University of Texas at Dallas*

Carol White, *Athens Area Technical Institute*

The author would like to acknowledge Mickey Sarquis and Amy Stander of the Partnership for the Advancement of Chemical Technology (PACT) for allowing me to link two of the experiments to the PACT publication, *Building Student Safety Habits for the Workplace*. I would also like to acknowledge the American Chemical Society's Committee on Chemical Safety. Its *Guidelines for Authors of Laboratory Manuals* provided a framework for communicating the safety issues of each experiment. Many thanks go to the Molecular Arts Corporation for granting permission to use some of their ChemClipArt 1000 images in this book.

Finally, the author is indebted to the program officers in the Advanced Technological Education (ATE) program at the National Science Foundation, especially Dr. Frank Settle, who was a program officer when the project began in 1997, and Dr. Elizabeth Teles, who has kept all of us on our toes since ATE was created in 1994.

Dedication

To all the students who have passed through my laboratory at Southeast Community College since 1977. Without them, I would not have the job I love nor the incentive to write books. May God bless all of you with many precious gifts.

"Happy are those who dwell in your house. They never cease to praise you." ***(Ps 84 5)***

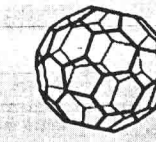
To the Student...

The I.O.N.S Corporation is a fictitious consulting firm created to serve as your employer for some simulated real-world laboratory work to be performed in the college laboratory. You are a laboratory technician for this company and will be working as part of a team, performing laboratory analyses, keeping a notebook, solving problems, making decisions, and writing reports much like a laboratory technician employed by the chemical industry. You will be expected to always exercise good laboratory practices (GLPs) and good management practices (GMPs) as you perform the standard operating procedures (SOPs) necessary to obtain the required results.

The Chief Executive Officer (CEO) of the I.O.N.S. Corporation is Claire Hemistry. Besides serving as CEO, Claire secures all the contracts for the company and always brings a new problem to you via a written memo. She arranges for individuals to serve as industrial and university consultants. You will be in contact with these experts to obtain relevant facts about the work which will help in solving the problem. These consultants will be giving you some initial direction for each problem you undertake. Following is an introductory memo from Ms. Hemistry.

I.O.N.S.

Innovative Options and New Solutions



Memo to: New Employees of I.O.N.S.

From: Claire Hemistry, CEO

Welcome to the I.O.N.S. Corporation! We are very pleased that you have joined us in our efforts to provide quality laboratory and consultation work for our clients. While specific aspects of your job will be communicated to you by your immediate supervisor, there are some very important general expectations to which I would like to call your attention. These relate to our safety policies and procedures and our expectations regarding laboratory notebooks and reporting memos.

First, regarding safety, the I.O.N.S. safety manual is included herein. We are very proud of the fact that only minor safety violations have occurred since our company was founded in 1978. We expect that all safety policies and procedures outlined in the manual will be in effect at all times. For this reason, it is imperative that you diligently study and familiarize yourself with the material contained in this manual. Before you are permitted to do any laboratory work, you must satisfactorily demonstrate your knowledge of safety to your supervisor. He or she will go over the safety manual with you and point out important aspects of your specific workspace. In addition, Ben Whell, the I.O.N.S. Safety Coordinator, provides a safety report for all projects that require special attention with respect to safety.

Second, the lifeline of our company rests solely on our ability to acquire and maintain the confidence of our clients. Accordingly, we expect that all personnel will abide by all GLP procedures set forth by the federal agencies that regulate a given project that you will undertake, such as the Food and Drug Administration (FDA) and the Environmental Protection Agency (EPA). We are a fully certified GLP laboratory and are audited periodically by these agencies. A critical aspect of GLP is good recordkeeping. We cannot emphasize enough the importance of keeping good records as you perform your laboratory work. Accordingly, we have adopted some very basic rules regarding the keeping of notebooks and journals in the laboratory. These are also included herein. We expect you to abide by these rules at all times in all of your work.

Finally, I.O.N.S. workers are asked to write report memos to clients for all projects completed. For each client project you undertake, I will provide the names of the person to whom you should address the memo. Specific instructions and an example reporting

memo also are included herein. Both your supervisor and I will evaluate your memos before they are sent.

Again, welcome to the company. If you have any questions at any time, please see your supervisor, or call and set up an appointment with me.

Sincerely,

Claire

Claire Hemistry
CEO, I.O.N.S. Corporation

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Section 1

I.O.N.S. Safety Manual

Innovative Options and New Solutions

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I. Safety Practices

A. Chemical Hygiene Plan

1. All workers must familiarize themselves with and know the location of the I.O.N.S. Chemical Hygiene Plan for the particular laboratories in which they work. The effect of this plan is to define the safety training needed for the laboratory, the circumstances requiring special approval, standard operating procedures for working with hazardous chemicals, criteria for control measures, measures to ensure proper operation of safety equipment, provisions for special hazards, provisions for medical consultants, and the designation of the I.O.N.S. safety hygiene officer.

B. General Practices

1. Horseplay in the laboratory is strictly prohibited.
2. Never climb onto or stand on chairs and stools.
3. Never place bottles, beakers, flasks, etc., containing solutions or chemicals precariously close to the edge of a bench, etc., where they can be knocked off easily.
4. Read and take seriously all signs placed in the area by supervisors or plant personnel, such as "Emergency Exit Only," "No Smoking," "Wet Floor," etc. (Figure 1.1). If you see any potential safety hazard, such as a wet floor, contact your supervisor immediately so that signs may be erected and/or the problem rectified.

FIGURE 1.1
All signs should be taken seriously.

5. If toxic, dangerous, or unpleasant gases become present in the lab at unsafe levels, all personnel should vacate the lab immediately and the plant supervisor notified. Examples include natural gas leaks, an experiment gone awry, etc.
6. Avoid raising chemicals or solutions above eye level.

C. Laboratory Safe Practices

1. All laboratory workers will adopt a safety-first policy. This means you must have adequate **knowledge** and a safe **attitude**.
2. Be cautious and careful, not sloppy and reckless.
3. Smoking, eating, or drinking in the laboratory is not allowed at any time (Figure 1.2).
4. Wear the appropriate personal protection equipment and adopt common sense policies regarding personal safety.
   a. Safety glasses with side shields must be worn at all times (Figure 1.3). (See also Section IIB.)
   b. Latex (or other appropriate material) gloves must be worn if your hands will possibly contact potentially toxic or hazardous materials (Figure 1.4).
   c. Tongs or hand insulators must be used when handling hot materials and equipment.
   d. Shorts, skirts, and open-toed shoes are discouraged.
   e. Tie back long hair, especially when working with flames.