

CHEMIST AND TECHNICIAN IN THE ANALYTICAL LABORATORY

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1.1 INTRODUCTION—THE ANALYTICAL CHEMIST AND TECHNICIAN

The analytical chemist and technician are an invaluable part of the chemistry laboratory. He/she is charged with performing analyses, updating records, taking inventory, documenting projects, keeping the laboratory clean, updating instrumentation and analyses, making sure the laboratory is safe, and giving support to the chief chemist. Choosing a career as a chemist or technician in any type of laboratory, such as environmental, petroleum, contract, medical, clinical, or biological to name a few, can be very rewarding. Some positions are more demanding than others, but all are challenging. New methods are often needed to be learned or developed. New instrumentation is needed to be set up and used. The chemist and technician also keep track of the testing that is being performed and documenting results.

1.2 TODAY'S LABORATORY CHEMIST AND TECHNICIAN

1.2.1 Computers in the Laboratory

The technicians and chemists in laboratories today are also routinely using computers on a daily basis. Computers are used

to control instrumentation and record the data that are being produced. The instrument vendors also often have their own software used for their instruments that the technician, chemist, or laboratory worker needs to learn and use on a routine, daily basis. The personal computer (e.g., tablet, notebook, laptop, etc.) is also used by the chemist and technician on a daily basis for entering laboratory data, processing data, and writing reports. Students today have now been introduced to the basic operations and use of the personal computer including the basics of coding.

1.2.2 Laboratory Information Management Systems (LIMS)

A very useful application found in most laboratories today is the Laboratory Information Management System (LIMS). LIMS is used to input data, track sample progress, and record sample data, such as company, type, and tests needed. In Chapter 12, we will be introduced to a LIMS example that we will use to log in samples, input data, search samples, approve samples, and print reports and certificates of analysis (C of A). Also becoming more common in laboratories are electronic laboratory notebooks, and we will take a brief look at using them.

1.3 ChemTech—THE CHEMIST AND TECHNICIAN TOOLKIT COMPANION

1.3.1 Introduction to ChemTech

This textbook is a comprehensive study of analytical chemistry as it pertains to the laboratory analyst and chemist. There are numerous chapters in the textbook devoted to the basics of analytical chemistry and introductions to the laboratory. This textbook includes an interactive application called ChemTech for chemists and laboratory technicians. The application acts as a learning aid as we move through the various aspects of analytical chemistry, laboratory work, and the needed skills to be learned. The ChemTech application has reference tables and an interactive periodic table. It has a link to a LIMS application that we will learn to use. The application also has a review of most of the chapters in this textbook. The combination of the ChemTech application and the chapters in this textbook can prepare the student for a rewarding career as a chemist or a laboratory technician.

1.3.2 Opening ChemTech

Please follow Appendix VI for installing all book-associated applications. Upon completion of downloading and installing the ChemTech application, open the application. The main introduction page should look like that found in Figure 1.1(a). Go down to the bottom right of the page and click on the button “Main Menu.” This will open up a page that includes many of the chapters present in the textbook. A link to each chapter is located in the box. Clicking on one of the buttons next to each chapter will open up pages associated with each chapter topic. Located to the right of the page are links to calculators, an interactive periodic table, and some reference tables (Figure 1.2).

1.3.3 Online ChemTech

An online version of the ChemTech program is available for use at the website www.chemistrysurvey.com. To open the ChemTech program, navigate to the link which designates opening the ChemTech program on the website.

1.3.3.1 Interactive Periodic Table Let us start by clicking the interactive periodic table button to open up the table. Once opened, the page should look like that in Figure 1.3. If you click on an element, a new page will open with facts about the element. Ten of the elements, listed at the bottom of the page, include a rotatable movie of the element. Click on the carbon “C” element to open its fact page. Different facts about the element are listed along with general information about the element. The page should look like the one in Figure 1.4. This is the same for each of the element pages in the periodic table. Also included are pictures of various representations of the elements. Take a few minutes to look at some of the other element information pages. Always remember that you can come back to this page for information about the elements. Click the “Return To Main Menu” button.

1.4 CHAPTER LAYOUT

1.4.1 Glassware, Chemicals, and Safety

Now that we have been introduced to the ChemTech application, we can begin to look at the other chapters. The textbook is designed to take the reader through many of the basic aspects of working in a laboratory. It also teaches the fundamental skills needed of a chemist and technician in science, including basic mathematics. An introduction to laboratory glassware, the layout of laboratories, and instruments used in the laboratory are covered in early chapters. Safety in the laboratory is also covered in an early chapter (see Chapter 3) to give the reader an overview of important aspects of safety in the laboratory.

1.4.2 Basic Math and Statistics

An overview of mathematics used in the laboratory is presented in Chapter 4. The chapter takes the reader through fundamental aspects such as the metric system, conversions such as pounds to grams, and significant figures. The scientific hand calculator is next presented to introduce and prepare the student for its use in the laboratory. The math next moves to statistics in Chapter 5. Statistics is a common and very useful tool in the analytical laboratory. The student is taught the basics of statistics while using ChemTech as a learning aid to help with understanding the associated calculations and concepts. Plotting and graphing are techniques that are used every day in the analytical laboratory.

1.4.3 Graphing and Plotting

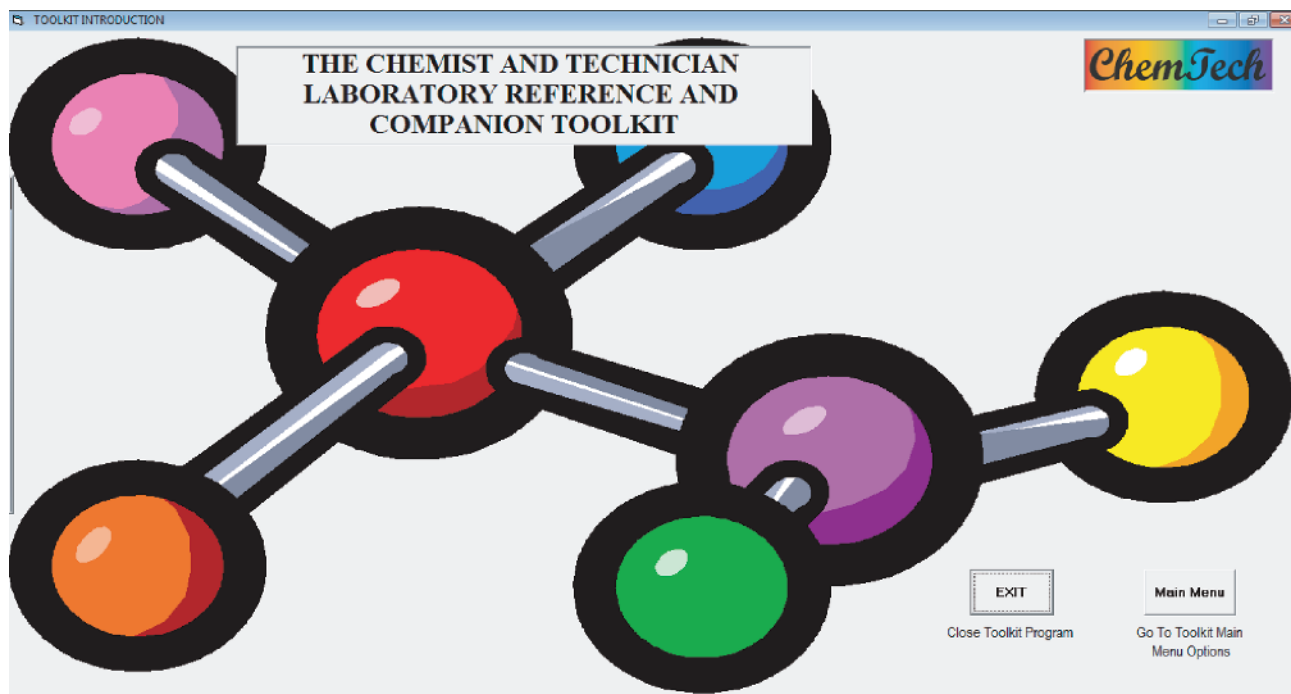
Chapter 6 introduces the reader to the basic construction and use of graphs in the laboratory. To aid in the plotting of data, the ChemTech program is used to construct the graphs. Finally, for the math portion of the textbook, Microsoft Excel[®] is introduced in Chapter 7 as a valuable lab tool for spreadsheet calculation and graphing.

1.4.4 Making Laboratory Solutions

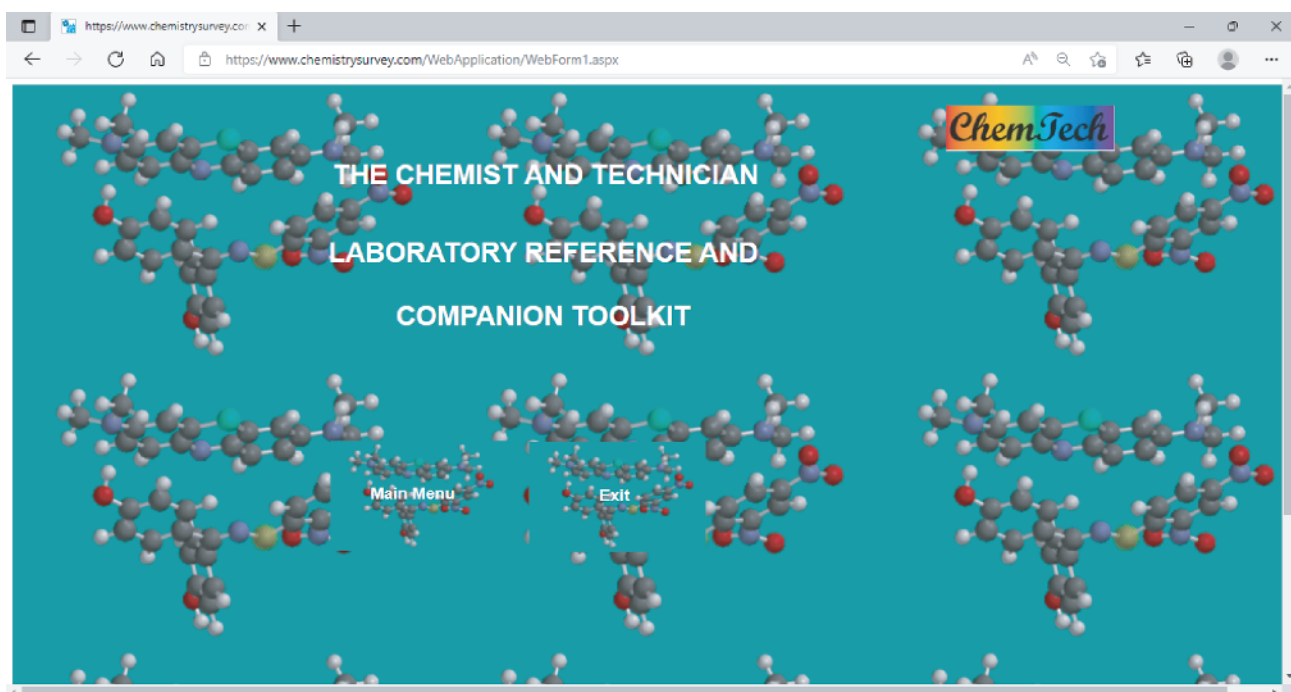
Making solutions in the laboratory is a necessary skill for the chemist and laboratory analyst. There are in fact full-time positions that are devoted to making laboratory solutions. We will learn to make solutions of different concentrations and representations such as molarity (M), normality (N), and parts per million (ppm). The program ChemTech will be used to calculate a variety of solutes and solvents needed to make laboratory solutions. In Chapter 9, we will look at acid–base theory, and how to measure and calculate the pH of solutions. Also covered in this chapter is how to make buffer solutions. The ChemTech program is a useful tool in the laboratory for calculating the amount of solutions needed to make some of the most common buffers used in the laboratory.

1.4.5 Titrimetric Analysis

Making buffers and solutions leads us to the analytical technique of titrations. A very useful and widely employed technique in



(a)



(b)

FIGURE 1.1 (a) The standalone (desktop) ChemTech application introduction page when it is first opened. The buttons to the bottom right will exit the application or take the user to the main menu of ChemTech. (b) The client-server (web based) application introduction page, including links to the Main Menu, and exiting.

the analytical laboratory is the titrimetric (volumetric) method of analysis. Titrimetric analysis is the process of measuring a substance of unknown concentration in a solution of interest via reaction with a standard that we have made that contains a known substance concentration. If we take a known weight or volume

of our solid or solution of interest, we can calculate the concentration of the unknown from the measured use of our known concentration solution. The ChemTech application will be used for a multitude of titrations using various solutions and analyte measurements.

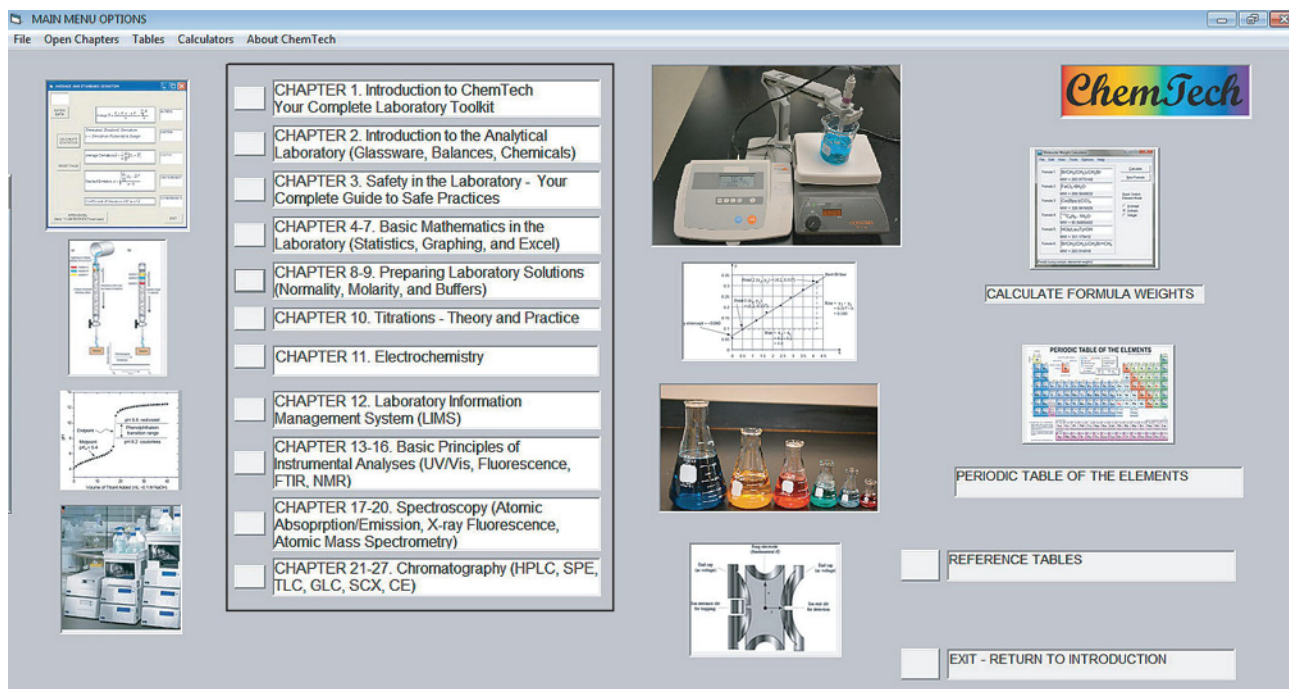


FIGURE 1.2 ChemTech Main Menu Page. The boxed-in area contains links to the various chapters covered in the textbook. To the right are links to calculators, reference tables, and an interactive periodic table.

CLICK ON ELEMENT TO SEE
INFORMATION SUCH AS: ELEMENT'S
NAME, OXIDATION STATE, FACTS,
TRIVIA, ETC.

Atomic
Number
→
Atomic
Weight
→
Symbol

Cobalt
27 Co
58.9332

Non Metals

Alkali metals

Alkaline earth metals

Transition Elements

Halogens

Noble gases

Return to Main Menu

Exit - Return to Chapter 8 - Close Table

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FIGURE 1.3 Interactive periodic table of the elements. Click on an element link to open a page that includes facts, pictures, and movies about the elements.

CARBON (C) ELEMENTAL FACTS

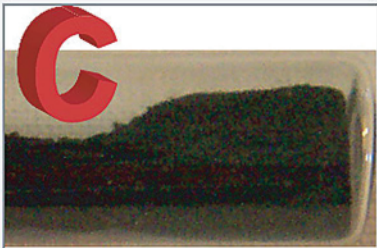
Carbon

Atomic number: 6

Atomic weight: 12.011

Symbol: C

Oxidation states: +2, +4, and -4



Carbon (from Latin: carbo "coal") is a chemical element with symbol C and atomic number 6. As a member of group 14 on the periodic table, it is nonmetallic and tetravalent — making four electrons available to form covalent chemical bonds. There are three naturally occurring isotopes, with ^{12}C and ^{13}C being stable, while ^{14}C is radioactive, decaying with a half-life of about 5,730 years.[11] Carbon is one of the few elements known since antiquity.[12]

There are several allotropes of carbon of which the best known are graphite, diamond, and amorphous carbon.[13] The physical properties of carbon vary widely with the allotropic form. For example, diamond is highly transparent, while graphite is opaque and black. Diamond is the hardest naturally-occurring material known, while graphite is soft enough to form a streak on paper (hence its name, from the Greek word "γράφω" which means "to write"). Diamond has a very low electrical conductivity, while graphite is a very good conductor. Under normal conditions, diamond, carbon nanotubes, and graphene have the highest thermal conductivities of all known materials.

All carbon allotropes are solids under normal conditions, with graphite being the most thermodynamically stable form. They are chemically resistant and require high temperature to react even with oxygen. The most common oxidation state of carbon in inorganic compounds is +4, while +2 is found in carbon monoxide and other transition metal carbonyl complexes. The largest sources of inorganic carbon are limestones, dolomites and carbon dioxide, but significant quantities occur in organic deposits of coal, peat, oil and methane clathrates. Carbon forms a vast number of compounds, more than any other element, with almost ten million compounds described to date,[14] which in turn are a tiny fraction of such compounds that are theoretically possible under standard conditions.

Carbon is the 15th most abundant element in the Earth's crust, and the fourth most abundant element in the

EXIT

FIGURE 1.4 Carbon element information page. The page contains facts about the elements and pictures.

1.4.6 Electrochemistry

Chapter 11 covers the area of electrochemistry in the analytical laboratory including oxidation–reduction reactions and the electrochemical cell. Working with redox equations is covered along with the important Nernst equation. The fundamentals of electrochemistry lead us to redox titrations such as potentiometric titrations.

1.4.7 Laboratory Information Management System (or Software) LIMS

Chapter 12 covers an application that is utilized in most laboratories today, the LIMS. LIMS is used to input data, track sample progress, and record sample data such as company, type, and tests needed. In Chapter 12, we will be introduced to a LIMS example that we will use to log in samples, input data, search samples, approve samples, and print reports and certificates of analysis (C of A). Also becoming more common in laboratories are electronic laboratory notebooks that are often coupled with LIMS, and we will take a brief look at using them.

1.4.8 Instrumental Analyses—Spectroscopy

Chapters 13–20 cover a wide range of instrumental analyses that the analyst needs to be introduced to. The analytical laboratory

utilizes the phenomenon of the electromagnetic spectrum for an untold number of analyses. Chemists and technicians in the analytical laboratory often make use of the special interaction of molecules with electromagnetic radiation, with the assistance of analytical instrumentation, such as ultraviolet/visible (UV/Vis) spectrophotometers, fluorometers, and Fourier transform infrared spectrometers (FTIR) to measure, identify, and even quantitate compounds of interest. Analysis of metals is also covered in these chapters.

1.4.9 Instrumental Analyses—Chromatography

Chapters 21–27 are dedicated to the area of chromatography. Chromatography is the separation of analyte species using a combination of a mobile phase and a stationary phase. The instrumental techniques covered in these chapters involve different types of chromatography, an extremely useful and quite common technique found in most analytical laboratories that the technician may find himself working in. The chromatographic techniques covered include column liquid chromatography (CLC), high-performance liquid chromatography (HPLC), solid-phase extraction (SPE), thin-layer chromatography (TLC), and gas-liquid chromatography (GC). Chapter 21 starts with the basic theory behind chromatography and then looks in detail at the aforementioned instrumental techniques that include the

important components of the chromatography instrumentation. There are illustrative examples throughout the chapters to help the technician in mastering each section followed by a set of problems to be worked at the end of the chapter.

1.4.10 Instrumental Analyses—Mass Spectrometry

Chapters 28–30 cover the more advanced topic of mass spectrometry. We previously looked at the analytical technique of gas chromatography coupled to a single quadrupole mass spectrometer. This is a robust, stable, and well-characterized instrumental analysis that has mostly been automated where the analyst is not called upon for advanced interpretation. The spectra are searched against a library if needed for identifications.

1.4.10.1 Mass Analyzers In these chapters, we will look further at mass spectrometers that are increasingly being used in laboratories today. These include an electric and magnetic sector mass analyzer, a time-of-flight mass spectrometer (TOF/MS), a time-of-flight/time-of-flight mass spectrometer (TOF–TOF/MS), the hybrid (hybrids are mass analyzers that couple together two separate types of mass analyzers) quadrupole time-of-flight mass spectrometer (Q-TOF/MS), a triple quadrupole or linear ion trap mass spectrometer (QQQ/MS or LIT/MS), a three-dimensional quadrupole ion trap mass spectrometer (QIT/MS), a Fourier transform ion cyclotron mass spectrometer (FTICR/MS), and finally the linear ion trap-Orbitrap mass spectrometer (IT-Orbitrap/MS). Also included are discussions of the two more recently introduced hybrid mass analyzers in use in laboratories today: the linear quadrupole ion trap Fourier transform mass spectrometer (LTQ-FT/MS) and the linear quadrupole ion trap Orbitrap mass spectrometer (LTQ-Orbitrap/MS).

1.4.10.2 Mass Ionization Also covered are the ionization sources used with the mass spectrometers. Source/ionization systems include electron ionization (EI), electrospray ionization (ESI), chemical ionization (CI), atmospheric pressure chemical ionization (APCI), atmospheric pressure photoionization (APPI), and matrix-assisted laser desorption ionization (MALDI). These ionization techniques produce ions of analyte molecules (often designated as “M” for molecule), which includes molecular ions $M^{+\bullet}$ (from EI), protonated molecules ($[M+H]^+$), deprotonated molecules ($[M-H]^-$), and metal ($[M+metal]^+$, e.g., $[M+Na]^+$) or halide ($[M+halide]^-$, e.g., $[M+Cl]^-$) adduct (all possible from ESI, CI, APCI, APPI, and MALDI).

1.4.11 Small Molecule and Macromolecule Analysis

Chapters 31–33 cover the advanced topics of small molecule analysis, macromolecule analysis, and proteomics. The study

of a biological system’s compliment of proteins (e.g., from cell, tissue, or a whole organism) at any given state in time has become a major area of focus for research and study in many different fields and applications. In proteomic studies, mass spectrometry can be employed to analyze both the intact, whole protein and the resultant peptides obtained from enzyme-digested proteins. The area of proteomics has been applied to a wide spectrum of physiological samples often based on comparative studies where a specific biological system’s protein expression is compared to either another system or the same system under stress. Often in the past, the comparison is made using two-dimensional electrophoresis where the gel maps for the two systems are compared looking for changes such as the presence or absence of proteins and the up or downregulation of proteins. Proteins of interest are cut from the gel and identified by mass spectrometry. More recently, proteomics is performed using the advanced instrumentation nano-HPLC/nano-electrospray mass spectrometry in conjunction with bioinformatics software. All will be covered in this chapter.

1.5 USERS OF ChemTech

The ChemTech program is a teaching tool designed to equip the chemist, the laboratory analyst, and the technician for a career in the analytical laboratory whether it is a clinical laboratory, an industrial, petrochemical, petroleum, environmental, college or university, or contract laboratory. This textbook covers the basics of the laboratory, including safety, glassware, and balances. Most of the fundamental aspects of laboratory analysis are also covered including titrations, chromatography, and instrumental analyses. Because the ChemTech application contains key points of most of the chapters, it can be used as a reference and toolkit for the chemist and technician throughout his/her career. ChemTech progresses from the basics of laboratory fundamentals in safety and glassware through advanced laboratory techniques and analyses. The ChemTech application and this textbook are a valuable source of reference material also for the industrial or academic researchers in the laboratory. Both are in a convenient format for looking up techniques and information in one place for numerous aspects of the laboratory and of samples whether it is the analyst, chemist, or researcher using them. They can also be useful as a supplemental learning source for the college student studying the sciences, such as the chemistry, biology, or premed student. The combination of this textbook with the ChemTech application would be a perfect approach for a community college that is designing a program that would teach and prepare laboratory technicians for an associate degree.