ANALYTICAL CHEMISTRY

Analytical Chemistry

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TABLE OF CONTENTS

2: Analytical Chemistry 2.0 (Harvey)

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00: Front Matter

This page was auto-generated because a user created a sub-page to this page.

TABLE OF CONTENTS

00: Front Matter

1: Front Matter

2: Introduction to Analytical Chemistry

3: Basic Tools of Analytical Chemistry

4: The Vocabulary of Analytical Chemistry

5: Evaluating Analytical Data

10: Titrimetric Methods

11: Spectroscopic Methods

12: Electrochemical Methods

13: Chromatographic

14: Kinetic Methods

15: Developing a Standard Method

16: Quality Assurance

17: Additional Resources

18: Back Matter

CHAPTER OVERVIEW

1: Front Matter

DePauw University

Analytical Chemistry 2.1

David Harvey

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LibreTexts is the adaptable, user-friendly non-profit open education resource platform that educators trust for creating, customizing, and sharing accessible, interactive textbooks, adaptive homework, and ancillary materials. We collaborate with individuals and organizations to champion open education initiatives, support institutional publishing programs, drive curriculum development projects, and more.

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1.3: Table of Contents

Analytical chemistry spans nearly all areas of chemistry but involves the development of tools and methods to measure physical properties of substances and apply those techniques to the identification of their presence (qualitative analysis) and quantify the amount present (quantitative analysis) of species in a wide variety of settings.

In the chapters that follow we will explore many aspects of analytical chemistry. In the process we will consider important questions such as “How do we treat experimental data?”, “How do we ensure that our results are accurate?”, “How do we obtain a representative sample?”, and “How do we select an appropriate analytical technique?” Before we look more closely at these and other questions, we will first review some basic tools of importance to analytical chemists.

If you leaf through an issue of the journal Analytical Chemistry, you will soon discover that the authors and readers share a common vocabulary of analytical terms. You are probably familiar with some of these terms, such as accuracy and precision, but other terms, such as analyte and matrix may be less familiar to you. In order to participate in the community of analytical chemists, you must first understand its vocabulary.

When using an analytical method we make three separate evaluations of experimental error. First, before beginning an analysis we evaluate potential sources of errors to ensure that they will not adversely effect our results. Second, during the analysis we monitor our measurements to ensure that errors remain acceptable. Finally, at the end of the analysis we evaluate the quality of the measurements and results, comparing them to our original design criteria.

- 5: Standardizing Analytical Methods

  Standardization is the process of determining the relationship between the signal and the amount of analyte in a sample. Previously, we defined this relationship as Stotal=kACA+Sreag where Stotal is the signal, nA is the moles of analyte, CA is the analyte's concentration, kA is the method's sensitivity for the analyte, and Sreag is the contribution to Stotal from sources other than the sample. To standardize a method we must determine values for kA and Sreag, which is the subject of this chapt

- 6: Equilibrium Chemistry

  Regardless of the problem on which an analytical chemist is working, its solution requires a knowledge of chemistry and the ability to apply that knowledge. For example, an analytical chemist studying the effect of pollution on spruce trees needs to know the chemical differences between p-hydroxybenzoic acid and p-hydroxyacetophenone, two phenols found in the needles of spruce trees. Your ability to "think as a chemist" is a product of your experience in the classroom and in the laboratory.

- 7: Collecting and Preparing Samples

  When we use an analytical method to solve a problem, there is no guarantee that our results will be accurate or precise. In designing an analytical method we consider potential sources of determinate error and indeterminate error, and take appropriate steps to minimize their effect, such as including reagent blanks and calibrating instruments. Why might a carefully designed analytical method give poor results? One possibility is that we may have failed to account for errors with the sample.

  - 7.6: Classifying Separation Techniques
  - 7.7: Liquid–Liquid Extractions
  - 7.8: Separation Versus Preconcentration
  - 7.E: Collecting and Preparing Samples (Exercises)
  - 7.S: Collecting and Preparing Samples (Summary)

- 8: Gravimetric Methods
  Gravimetry includes all analytical methods in which the analytical signal is a measurement of mass or a change in mass. When you step on a scale after exercising you are making, in a sense, a gravimetric determination of your mass. Mass is the most fundamental of all analytical measurements, and gravimetry is unquestionably our oldest quantitative analytical technique.

  - 8.1: Overview of Gravimetric Methods
  - 8.2: Precipitation Gravimetry
  - 8.3: Volatilization Gravimetry
  - 8.4: Particulate Gravimetry
  - 8.E: Gravimetric Methods (Exercises)
  - 8.S: Gravimetric Methods (Summary)

- 9: Titrimetric Methods
  Titrimetry, in which volume serves as the analytical signal, made its first appearance as an analytical method in the early eighteenth century. Titrimetric methods were not well received by the analytical chemists of that era because they could not duplicate the accuracy and precision of a gravimetric analysis. Not surprisingly, few standard texts from the 1700s and 1800s include titrimetric methods of analysis.

  - 9.1: Overview of Titrimetry
  - 9.2: Acid–Base Titrations
  - 9.3: Complexation Titrations
  - 9.4: Redox Titrations
  - 9.5: Precipitation Titrations
  - 9.E: Titrimetric Methods (Exercises)
  - 9.S: Titrimetric Methods (Summary)

- 10: Spectroscopic Methods
  "Colorimetry" is one example of a spectroscopic method of analysis. At the end of the nineteenth century, spectroscopy was limited to the absorption, emission, and scattering of visible, ultraviolet, and infrared electromagnetic radiation. Since its introduction, spectroscopy has expanded to include other forms of electromagnetic radiation—such as X-rays, microwaves, and radio waves—and other energetic particles—such as electrons and ions.

  - 10.1: Overview of Spectroscopy
  - 10.2: Spectroscopy Based on Absorption
  - 10.3: UV/Vis and IR Spectroscopy
  - 10.4: Atomic Absorption Spectroscopy
  - 10.5: Emission Spectroscopy
  - 10.6: Photoluminescence Spectroscopy
  - 10.7: Atomic Emission Spectroscopy
  - 10.8: Spectroscopy Based on Scattering
  - 10.E: Spectroscopic Methods (Exercises)
  - 10.S: Spectroscopic Methods (Summary)

11: Electrochemical Methods

In Chapter 10 we examined several spectroscopic techniques that take advantage of the interaction between electromagnetic radiation and matter. In this chapter we turn our attention to electrochemical techniques in which the potential, current, or charge in an electrochemical cell serves as the analytical signal. Although there are only three basic electrochemical signals, there are a many possible experimental designs—too many, in fact, to cover adequately in an introductory textbook.

- 11.1: Overview of Electrochemistry
- 11.2: Potentiometric Methods
- 11.3: Coulometric Methods
- 11.4: Voltammetric Methods
- 11.E: Electrochemical Methods (Exercises)
- 11.S: Electrochemical Methods (Summary)

12: Chromatographic & Electrophoretic Methods

For this reason, many analytical procedures include a step to separate the analyte from potential interferents. Although effective, each additional step in an analytical procedure increases the analysis time and introduces uncertainty. In this chapter we consider two analytical techniques that avoid these limitations by combining the separation and analysis: chromatography and electrophoresis.

- 12.1: Overview of Analytical Separations
- 12.2: General Theory of Column Chromatography
- 12.3: Optimizing Chromatographic Separations
- 12.4: Gas Chromatography
- 12.5: High-Performance Liquid Chromatography
- 12.6: Other Forms of Liquid Chromatography
- 12.7: Electrophoresis
- 12.E: Chromatographic and Electrophoretic Methods (Exercises)
- 12.S: Chromatographic and Electrophoretic Methods (Summary)

13: Kinetic Methods

One way to classify analytical techniques is by whether the analyte’s concentration is determined by an equilibrium reaction or by the kinetics of a chemical reaction or a physical process. Often analytical methods involve measurements made on systems in which the analyte is always at equilibrium. In this chapter we turn our attention to measurements made under non-equilibrium conditions.

- 13.1: Kinetic Methods Versus Equilibrium Methods
- 13.2: Chemical Kinetics
- 13.3: Radiochemistry
- 13.4: Flow Injection Analysis
- 13.E: Kinetic Methods (Exercises)
- 13.S: Kinetic Methods (Summary)

14: Developing a Standard Method

Among the goals of analytical chemistry are improving established methods of analysis, extending existing methods of analysis to new types of samples, and developing new analytical methods. In this chapter we discuss how we develop a standard method, including optimizing the experimental procedure, verifying that the method produces acceptable precision and accuracy in the hands of a single analyst, and validating the method for general use.

- 14.1: Optimizing the Experimental Procedure
- 14.2: Verifying the Method
- 14.3: Validating the Method as a Standard Method
- 14.4: Using Excel and R for an Analysis of Variance
- 14.E: Developing a Standard Method (Exercises)
- 14.S: Developing a Standard Method (Summary)

- 15: Quality Assurance
  Knowing that a method meets suitable standards is important if we are to have confidence in our results. Even so, using a standard method does not guarantee that the result of an analysis is acceptable. In this chapter we introduce the quality assurance procedures used in industry and government labs for monitoring routine chemical analyses.

CHAPTER OVERVIEW

2: Introduction to Analytical Chemistry

Chemistry is the study of matter, including its composition and structure, its physical properties, and its reactivity. There are many ways to study chemistry, but, we traditionally divide it into five fields: organic chemistry, inorganic chemistry, biochemistry, physical chemistry, and analytical chemistry. Although this division is historical and, perhaps, arbitrary—as witnessed by current interest in interdisciplinary areas such as bioanalytical chemistry and organometallic chemistry—these five fields remain the simplest division spanning the discipline of chemistry.

Topic hierarchy

Thumbnail: Several graduated cylinders of various thickness and heights with white side markings in front of a large beaker. They are all filled about halfway with red or blue chemical compounds. The blue ink is showing signs of Brownian motion when dissolving into water. Image used with permission (CC BY-SA 3.0; Horia Varlan from Bucharest, Romania).

Contributors

- David Harvey (DePauw University)

2.1: What is Analytical Chemistry?

1.1: What is Analytical Chemistry?

2.2: The Analytical Perspective

1.2: The Analytical Perspective

2.3: Common Analytical Problems

1.3: Common Analytical Problems

2.4: Introduction to Analytical Chemistry (Exercises)

1.E: Introduction to Analytical Chemistry (Exercises)

2.5: Introduction to Analytical Chemistry (Summary)

1.S: Introduction to Analytical Chemistry (Summary)

CHAPTER OVERVIEW

3: Basic Tools of Analytical Chemistry

In the chapters that follow we will explore many aspects of analytical chemistry. In the process we will consider important questions such as “How do we treat experimental data?”, “How do we ensure that our results are accurate?”, “How do we obtain a representative sample?”, and “How do we select an appropriate analytical technique?” Before we look more closely at these and other questions, we will first review some basic tools of importance to analytical chemists.

Thumbnail: A Roberval balance. The pivots of the parallelogram understructure makes it insensitive to load positioning away from center, so improves its accuracy, and ease of use. Image used with permission (CC BY-SA 3.0; Nikodem Nijaki)

Contributors

- David Harvey (DePauw University)

3.1: Measurements in Analytical Chemistry

2.1: Measurements in Analytical Chemistry

3.2: Concentration

2.2: Concentration

3.3: Stoichiometric Calculations

2.3: Stoichiometric Calculations

3.4: Basic Equipment

2.4: Basic Equipment

3.5: Preparing Solutions

2.5: Preparing Solutions

3.6: Spreadsheets and Computational Software

2.6: Spreadsheets and Computational Software

3.7: The Laboratory Notebook

2.7: The Laboratory Notebook

3.8: Basic Tools of Analytical Chemistry (Exercises)

2.E: Basic Tools of Analytical Chemistry (Exercises)

3.9: Basic Tools of Analytical Chemistry (Summary)

2.S: Basic Tools of Analytical Chemistry (Summary)

CHAPTER OVERVIEW

4: The Vocabulary of Analytical Chemistry

If you leaf through an issue of the journal Analytical Chemistry, you will soon discover that the authors and readers share a common vocabulary of analytical terms. You are probably familiar with some of these terms, such as accuracy and precision, but other terms, such as analyte and matrix may be less familiar to you. In order to participate in the community of analytical chemists, you must first understand its vocabulary. The goal of this chapter, therefore, is to introduce you to some important analytical terms. Becoming comfortable with these terms will make the material in the chapters that follow easier to read and understand.

Thumbnail: Colonies of fecal coliform bacteria from a water supply. Source: Susan Boyer. Photo courtesy of ARS–USDA (www.ars.usda.gov). Fecal coliform counts provide a general measure of the presence of pathogenic organisms in a water supply. For drinking water, the current maximum contaminant level (MCL) for total coliforms, including fecal coliforms is less than 1 colony/100 mL. Municipal water departments must regularly test the water supply and must take action if more than 5% of the samples in any month test positive for coliform bacteria.

Contributors

- David Harvey (DePauw University)

4.1: Analysis, Determination, and Measurement

3.1: Analysis, Determination, and Measurement

4.2: Techniques, Methods, Procedures, and Protocols

3.2: Techniques, Methods, Procedures, and Protocols

4.3: Classifying Analytical Techniques

4.4: Selecting an Analytical Method

4.5: Developing the Procedure

4.6: Protocols

3.6: Protocols

4.7: The Importance of Analytical Methodology

4.8: The Vocabulary of Analytical Chemistry (Exercises)

3.E: The Vocabulary of Analytical Chemistry (Exercises)

4.9: The Vocabulary of Analytical Chemistry (Summary)

3.S: The Vocabulary of Analytical Chemistry (Summary)

5: Evaluating Analytical Data

When using an analytical method we make three separate evaluations of experimental error. First, before beginning an analysis we evaluate potential sources of errors to ensure that they will not adversely effect our results. Second, during the analysis we monitor our measurements to ensure that errors remain acceptable. Finally, at the end of the analysis we evaluate the quality of the measurements and results, comparing them to our original design criteria. This chapter provides an introduction to sources of error, to evaluating errors in analytical measurements, and to the statistical analysis of data.

Thumbnail: The blue vertical line segments represent multiple realizations of a confidence interval for the population mean μ, represented as a red horizontal dashed line; note that some confidence intervals do not contain the population mean, as expected. Image used with permission (Public Domain; Tsyplakov) .

Contributors

- David Harvey (DePauw University)

5.1: Characterizing Measurements and Results

5.2: Characterizing Experimental Errors

4.2: Characterizing Experimental Errors

5.3: Propagation of Uncertainty

5.4: The Distribution of Measurements and Results

4.4: The Distribution of Measurements and Results

5.5: Statistical Analysis of Data

5.6: Statistical Methods for Normal Distributions

4.6: Statistical Methods for Normal Distributions

5.7: Detection Limits

4.7: Detection Limits

5.8: Using Excel and R to Analyze Data

5.9: Evaluating Analytical Data (Exercises)

5.10: Evaluating Analytical Data (Summary)

4.S: Evaluating Analytical Data (Summary)

CHAPTER OVERVIEW

6: Standardizing Analytical Methods

The American Chemical Society's Committee on Environmental Improvement defines standardization as the process of determining the relationship between the signal and the amount of analyte in a sample.[1] In Chapter 3 we defined this relationship as

$$S_{\text{total}} = k_{\text{A}} n_{\text{A}} + S_{\text{reag}} \quad \text{or} \quad S_{\text{total}} = k_{\text{A}} C_{\text{A}} + S_{\text{reag}} \tag{6.1}$$

where S_{total} is the signal, n_A is the moles of analyte, C_A is the analyte's concentration, k_A is the method's sensitivity for the analyte, and S_{reag} is the contribution to S_{total} from sources other than the sample. To standardize a method we must determine values for k_A and S_{reag}. Strategies for accomplishing this are the subject of this chapter.

Thumbnail: Illustration showing the evaluation of a linear regression in which we assume that all uncertainty is the result of indeterminate errors affecting y. The points in blue, y_i*, are the original data and the points in red,* $\hat{y}_i$*, are the predicted values from the regression equation,* $\hat{y} = b_0 + b_1 x$*. The smaller the total residual error (equation 5.16), the better the fit of the straight-line to the data*

Contributors

- David Harvey (DePauw University)

6.1: Analytical Standards

5.1: Analytical Standards

6.2: Calibrating the Signal

5.2: Calibrating the Signal

6.3: Determining the Sensitivity

5.3: Determining the Sensitivity

6.4: Linear Regression and Calibration Curves

5.4: Linear Regression and Calibration Curves

6.5: Blank Corrections

5.5: Blank Corrections

6.6: Using Excel and R for a Regression Analysis

6.7: Standardizing Analytical Methods (Exercises)

5.E: Standardizing Analytical Methods (Exercises)

6.8: Standardizing Analytical Methods (Summary)

5.S: Standardizing Analytical Methods (Summary)

CHAPTER OVERVIEW

7: Equilibrium Chemistry

Regardless of the problem on which an analytical chemist is working, its solution requires a knowledge of chemistry and the ability to apply that knowledge. For example, an analytical chemist studying the effect of pollution on spruce trees needs to know, or know where to find, the chemical differences between *p*-hydroxybenzoic acid and *p*-hydroxyacetophenone, two common phenols found in the needles of spruce trees. Your ability to “think as a chemist” is a product of your experience in the classroom and in the laboratory. The material in this text assumes your familiarity with topics from earlier courses. Because of its importance to analytical chemistry, this chapter provides a review of equilibrium chemistry. Much of the material in this chapter should be familiar to you, although some topics—ladder diagrams and activity, for example—afford you with new ways to look at equilibrium chemistry.

Thumbnail: *The* $N_2O_{(g)} \rightleftharpoons 2NO_{2(g)}$ *system at Different Temperatures. Nitrogen dioxide* (NO_2) *gas converts to the colorless gas dinitrogen tetroxide* (N_2O_4) *at low temperatures, and converts back to* NO_2 *at higher temperatures. The bottles in this photograph contain equal amounts of gas at different temperatures. Figure used with permission from Wikipedia (CC BY-SA 3.0).*

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- David Harvey (DePauw University)

7.1: Reversible Reactions and Chemical Equilibria

6.01: Reversible Reactions and Chemical Equilibria

7.2: Thermodynamics and Equilibrium Chemistry

6.02: Thermodynamics and Equilibrium Chemistry

7.3: Manipulating Equilibrium Constants

6.03: Manipulating Equilibrium Constants

7.4: Equilibrium Constants for Chemical Reactions

6.04: Equilibrium Constants for Chemical Reactions

7.5: Le Châtelier’s Principle

6.05: Le Châtelier’s Principle

7.6: Ladder Diagrams

6.06: Ladder Diagrams

7.7: Solving Equilibrium Problems

6.07: Solving Equilibrium Problems

7.8: Buffer Solutions

6.08: Buffer Solutions

7.9: Activity Effects

6.09: Activity Effects

7.10: Using Excel and R to Solve Equilibrium Problems

6.10: Using Excel and R to Solve Equilibrium Problems

7.11: Some Final Thoughts on Equilibrium Calculations

6.11: Some Final Thoughts on Equilibrium Calculations

7.12: Equilibrium Chemistry (Exercises)

6.E: Equilibrium Chemistry (Exercises)

7.13: Equilibrium Chemistry (Summary)

6.S: Equilibrium Chemistry (Summary)

CHAPTER OVERVIEW

8: Collecting and Preparing Samples

When we use an analytical method to solve a problem, there is no guarantee that our results will be accurate or precise. In designing an analytical method we consider potential sources of determinate error and indeterminate error, and take appropriate steps to minimize their effect, such as including reagent blanks and calibrating instruments. Why might a carefully designed analytical method give poor results? One possibility is that we may have failed to account for errors associated with the sample. If we collect the wrong sample, or if we lose analyte while preparing the sample for analysis, then we introduce a determinate source of error. If we fail to collect enough samples, or if we collect samples of the wrong size, then our precision may suffer. In this chapter we consider how collecting samples and preparing them for analysis affects the accuracy and precision of our results.

Thumbnail: An example of pipettes and microplates manipulated by an anthropomorphic robot (Andrew Alliance). Image used with permission (Cc BY-SA 3.0; Pzucchel).

Contributors

- David Harvey (DePauw University)

8.1: The Importance of Sampling

7.1: The Importance of Sampling

8.2: Designing a Sampling Plan

7.2: Designing a Sampling Plan

8.3: Implementing the Sampling Plan

7.3: Implementing the Sampling Plan

8.4: Separating the Analyte from Interferents

7.4: Separating the Analyte from Interferents

8.5: General Theory of Separation Efficiency

7.5: General Theory of Separation Efficiency

8.6: Classifying Separation Techniques

7.6: Classifying Separation Techniques

8.7: Liquid–Liquid Extractions

7.7: Liquid–Liquid Extractions

8.8: Separation Versus Preconcentration

7.8: Separation Versus Preconcentration

8.9: Collecting and Preparing Samples (Exercises)

8.10: Collecting and Preparing Samples (Summary)

7.S: Collecting and Preparing Samples (Summary)

CHAPTER OVERVIEW

9: Gravimetric Methods

Gravimetry includes all analytical methods in which the analytical signal is a measurement of mass or a change in mass. When you step on a scale after exercising you are making, in a sense, a gravimetric determination of your mass. Mass is the most fundamental of all analytical measurements, and gravimetry is unquestionably our oldest quantitative analytical technique. The publication in 1540 of Vannoccio Biringuccio's *Pirotechnia* is an early example of applying gravimetry—although not yet known by this name—to the analysis of metals and ores.[1] Although gravimetry no longer is the most important analytical method, it continues to find use in specialized applications.

Thumbnails: An analytical balance (Mettler ae-260) that is often used in gravimetric analysis Methods. Image used with permission (Public Domain; US DEA).

Contributors

- David Harvey (DePauw University)

9.1: Overview of Gravimetric Methods

8.1: Overview of Gravimetric Methods

9.2: Precipitation Gravimetry

8.2: Precipitation Gravimetry

9.3: Volatilization Gravimetry

8.3: Volatilization Gravimetry

9.4: Particulate Gravimetry

8.4: Particulate Gravimetry

9.5: Gravimetric Methods (Exercises)

9.6: Gravimetric Methods (Summary)

8.S: Gravimetric Methods (Summary)

CHAPTER OVERVIEW

10: Titrimetric Methods

Titrimetry, in which volume serves as the analytical signal, made its first appearance as an analytical method in the early eighteenth century. Titrimetric methods were not well received by the analytical chemists of that era because they could not duplicate the accuracy and precision of a gravimetric analysis. Not surprisingly, few standard texts from the 1700s and 1800s include titrimetric methods of analysis.

Precipitation gravimetry developed as an analytical method without a general theory of precipitation. An empirical relationship between a precipitate's mass and the mass of analyte—what analytical chemists call a gravimetric factor—was determined experimentally by taking a known mass of analyte through the procedure. Today, we recognize this as an early example of an external standardization. Gravimetric factors were not calculated using the stoichiometry of a precipitation reaction because chemical formulas and atomic weights were not yet available! Unlike gravimetry, the development and acceptance of titrimetry required a deeper understanding of stoichiometry, of thermodynamics, and of chemical equilibria. By the 1900s, the accuracy and precision of titrimetric methods were comparable to that of gravimetric methods, establishing titrimetry as an accepted analytical technique.

Thumbnail: A Winkler titration to determine the concentration of dissolved oxygen in a water sample. The dissolved oxygen has been converted to an equivalent amount of iodine, which is being titrated with thiosulfate using a starch indicator. The blue color in the flask will disappear when all the iodine has been converted to iodide. Image used with permission (CC BY-SA 3.0; Will Woodgate (Cornwall College, UK)).

Contributors

- David Harvey (DePauw University)

10.1: Overview of Titrimetry

9.1: Overview of Titrimetry

10.2: Acid–Base Titrations

9.2: Acid–Base Titrations

10.3: Complexation Titrations

9.3: Complexation Titrations

10.4: Redox Titrations

9.4: Redox Titrations

10.5: Precipitation Titrations

9.5: Precipitation Titrations

10.6: Titrimetric Methods (Exercises)

9.E: Titrimetric Methods (Exercises)

10.7: Titrimetric Methods (Summary)

9.S: Titrimetric Methods (Summary)

CHAPTER OVERVIEW

11: Spectroscopic Methods

An early example of a colorimetric analysis is Nessler’s method for ammonia, which was introduced in 1856. Nessler found that adding an alkaline solution of HgI_2 and KI to a dilute solution of ammonia produces a yellow to reddish brown colloid, with the colloid’s color depending on the concentration of ammonia. By visually comparing the color of a sample to the colors of a series of standards, Nessler was able to determine the concentration of ammonia.

Colorimetry, in which a sample absorbs visible light, is one example of a spectroscopic method of analysis. At the end of the nineteenth century, spectroscopy was limited to the absorption, emission, and scattering of visible, ultraviolet, and infrared electromagnetic radiation. Since its introduction, spectroscopy has expanded to include other forms of electromagnetic radiation—such as X-rays, microwaves, and radio waves—and other energetic particles—such as electrons and ions.

Contributors

- David Harvey (DePauw University)

11.1: Overview of Spectroscopy

10.1: Overview of Spectroscopy

11.2: Spectroscopy Based on Absorption

10.2: Spectroscopy Based on Absorption

11.03: UV

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11.3: UV/Vis and IR Spectroscopy

11.4: Atomic Absorption Spectroscopy

10.4: Atomic Absorption Spectroscopy

11.5: Emission Spectroscopy

11.6: Photoluminescence Spectroscopy

10.6: Photoluminescence Spectroscopy

11.7: Atomic Emission Spectroscopy

10.7: Atomic Emission Spectroscopy

11.8: Spectroscopy Based on Scattering

10.8: Spectroscopy Based on Scattering

11.9: Spectroscopic Methods (Exercises)

11.10: Spectroscopic Methods (Summary)

CHAPTER OVERVIEW

12: Electrochemical Methods

In Chapter 10 we examined several spectroscopic techniques that take advantage of the interaction between electromagnetic radiation and matter. In this chapter we turn our attention to electrochemical techniques in which the potential, current, or charge in an electrochemical cell serves as the analytical signal.

Although there are only three basic electrochemical signals, there are a many possible experimental designs—too many, in fact, to cover adequately in an introductory textbook. The simplest division of electrochemical techniques is between bulk techniques, in which we measure a property of the solution in the electrochemical cell, and interfacial techniques, in which the potential, charge, or current depends on the species present at the interface between an electrode and the solution in which it sits. The measurement of a solution's conductivity, which is proportional to the total concentration of dissolved ions, is one example of a bulk electrochemical technique. A determination of pH using a pH electrode is an example of an interfacial electrochemical technique. Only interfacial electrochemical methods receive further consideration in this chapter.

Thumbnail:Comparison of the current response of a platinum disc electrode in 1 M sulfuric acid given by linear sweep voltammetry and staircase voltammetry methods. Image used with permission (CC BY-SA 3.0; Earth-Rare).

Contributors

- David Harvey (DePauw University)

12.1: Overview of Electrochemistry

11.1: Overview of Electrochemistry

12.2: Potentiometric Methods

11.2: Potentiometric Methods

12.3: Coulometric Methods

12.4: Voltammetric Methods

12.5: Electrochemical Methods (Exercises)

11.E: Electrochemical Methods (Exercises)

12.6: Electrochemical Methods (Summary)

11.S: Electrochemical Methods (Summary)

CHAPTER OVERVIEW

13: Chromatographic

Drawing from an arsenal of analytical techniques—many of which were the subject of the preceding four chapters—analytical chemists design methods for the analysis of analytes at increasingly lower concentrations and in increasingly more complex matrices. Despite the power of these analytical techniques, they often suffer from a lack of selectivity. For this reason, many analytical procedures include a step to separate the analyte from potential interferents. Although effective, each additional step in an analytical procedure increases the analysis time and introduces uncertainty. In this chapter we consider two analytical techniques that avoid these limitations by combining the separation and analysis: chromatography and electrophoresis.

Thumbnail: Separation of black ink on a thin layer chromatography plate. Image used with permission (CC BY-SA 3.0; Natrij)}

Contributors

- David Harvey (DePauw University)

13.1: Overview of Analytical Separations

12.1: Overview of Analytical Separations

13.2: General Theory of Column Chromatography

13.3: Optimizing Chromatographic Separations

13.4: Gas Chromatography

13.5: High-Performance Liquid Chromatography

12.5: High-Performance Liquid Chromatography

13.6: Other Forms of Liquid Chromatography

12.6: Other Forms of Liquid Chromatography

13.7: Electrophoresis

12.7: Electrophoresis

13.8: Chromatographic and Electrophoretic Methods (Exercises)

13.9: Chromatographic and Electrophoretic Methods (Summary)

12.S: Chromatographic and Electrophoretic Methods (Summary)

CHAPTER OVERVIEW

14: Kinetic Methods

There are many ways to categorize analytical techniques, several of which we introduced in earlier chapters. In Chapter 3 we classified techniques by whether the signal is proportional to the absolute amount or the relative amount of analyte. For example, precipitation gravimetry is a total analysis technique because the precipitate’s mass is proportional to the absolute amount, or moles, of analyte. UV/Vis absorption spectroscopy, on the other hand, is a concentration technique because absorbance is proportional to the relative amount, or concentration, of analyte.

A second method for classifying analytical techniques is to consider the source of the analytical signal. For example, gravimetry encompasses all techniques in which the analytical signal is a measurement of mass or a change in mass. Spectroscopy, on the other hand, includes those techniques in which we probe a sample with an energetic particle, such as the absorption of a photon. This is the classification scheme used in organizing Chapters 8–11.

Another way to classify analytical techniques is by whether the analyte’s concentration is determined by an equilibrium reaction or by the kinetics of a chemical reaction or a physical process. The analytical methods described in Chapter 8–11 mostly involve measurements made on systems in which the analyte is always at equilibrium. In this chapter we turn our attention to measurements made under non-equilibrium conditions.

Thumbnail: Determination of a reaction’s intermediate rate from the slope of a line tangent to a curve showing the change in the analyte’s concentration as a function of time.

Contributors

- David Harvey (DePauw University)

14.1: Kinetic Methods Versus Equilibrium Methods

13.1: Kinetic Methods Versus Equilibrium Methods

14.2: Chemical Kinetics

13.2: Chemical Kinetics

14.3: Radiochemistry

14.4: Flow Injection Analysis

14.5: Kinetic Methods (Exercises)

14.6: Kinetic Methods (Summary)

CHAPTER OVERVIEW

15: Developing a Standard Method

In Chapter 1 we made a distinction between analytical chemistry and chemical analysis. Among the goals of analytical chemistry are improving established methods of analysis, extending existing methods of analysis to new types of samples, and developing new analytical methods. Once we develop a new method, its routine application is best described as chemical analysis. We recognize the status of these methods by calling them standard methods. Numerous examples of standard methods are presented and discussed in Chapters 8–13. What we have not yet considered is what constitutes a standard method. In this chapter we discuss how we develop a standard method, including optimizing the experimental procedure, verifying that the method produces acceptable precision and accuracy in the hands of a single analyst, and validating the method for general use.

Contributors

- David Harvey (DePauw University)

15.1: Optimizing the Experimental Procedure

14.1: Optimizing the Experimental Procedure

15.2: Verifying the Method

15.3: Validating the Method as a Standard Method

15.4: Using Excel and R for an Analysis of Variance

14.4: Using Excel and R for an Analysis of Variance

15.5: Developing a Standard Method (Exercises)

14.E: Developing a Standard Method (Exercises)

15.6: Developing a Standard Method (Summary)

CHAPTER OVERVIEW

16: Quality Assurance

In Chapter 14 we discussed the process of developing a standard method, including optimizing the experimental procedure, verifying that the method produces acceptable precision and accuracy in the hands of a signal analyst, and validating the method for general use by the broader analytical community. Knowing that a method meets suitable standards is important if we are to have confidence in our results. Even so, using a standard method does not guarantee that the result of an analysis is acceptable. In this chapter we introduce the quality assurance procedures used in industry and government labs for monitoring routine chemical analyses.

Thumbnail: Examples of property control charts that show a sequence of results.

Contributors

- David Harvey (DePauw University)

16.1: The Analytical Perspective—Revisited

15.1: The Analytical Perspective—Revisited

16.2: Quality Control

15.2: Quality Control

16.3: Quality Assessment

15.3: Quality Assessment

16.4: Evaluating Quality Assurance Data

15.4: Evaluating Quality Assurance Data

16.5: Quality Assurance (Exercises)

15.E: Quality Assurance (Exercises)

17: Additional Resources

Gathered here are three types of resources: suggested experiments, mostly from the *Journal of Chemical Education* and *The Chemical Educator*, that provide practical examples of concepts in the textbook; additional readings from the analytical literature that extend and supplement topics covered in the textbook; and electronic resources, many of which are cataloged in the Analytical Sciences Digital Library, that help illustrate concepts from the textbook. Although primarily intended for the use of instructors, these resources also will benefit students who wish to pursue a topic at more depth.

The following experiments provide useful introductions to the statistical analysis of data in the analytical chemistry laboratory.

- Bularzik, J. "The Penny Experiment Revisited: An Illustration of Significant Figures, Accuracy, Precision, and Data Analysis," *J. Chem. Educ.* **2007**, *84*, 1456–1458.
- Columbia, M. R. "The Statistics of Coffee: 1. Evaluation of Trace Metals for Establishing a Coffee's Country of Origin Based on a Means Comparison," *Chem. Educator* **2007**, *12*, 260–262.
- Cunningham, C. C.; Brown, G. R.; St Pierre, L. E. "Evaluation of Experimental Data," *J. Chem. Educ.* **1981**, *58*, 509–511.
- Edminston, P. L.; Williams, T. R. "An Analytical Laboratory Experiment in Error Analysis: Repeated Determination of Glucose Using Commercial Glucometers," *J. Chem. Educ.* **2000**, *77*, 377–379.
- Gordus, A. A. "Statistical Evaluation of Class Data for Two Buret Readings," *J. Chem. Educ.* **1987**, *64*, 376–377.
- Harvey, D. T. "Statistical Evaluation of Acid/Base Indicators," *J. Chem. Educ.* **1991**, *68*, 329–331.
- Hibbert, D. B. "Teaching modern data analysis with The Royal Austrian Chemical Institute's titration competition," *Aust. J. Ed. Chem.* **2006**, *66*, 5–11.
- Johll, M. E.; Poister, D.; Ferguson, J. "Statistical Comparison of Multiple Methods for the Determination of Dissolved Oxygen Levels in Natural Water," *Chem. Educator* **2002**, *7*, 146–148.
- Jordon, A. D. "Which Method is Most Precise; Which is Most Accurate?," *J. Chem. Educ.* **2007**, *84*, 1459–1460.
- Olsen, R. J. "Using Pooled Data and Data Visualization To Introduce Statistical Concepts in the General Chemistry Laboratory," *J. Chem. Educ.* **2008**, *85*, 544–545.
- O'Reilley, J. E. "The Length of a Pestle," *J. Chem. Educ.* **1986**, *63*, 894–896.
- Paselk, R. A. "An Experiment for Introducing Statistics to Students of Analytical and Clinical Chemistry," *J. Chem. Educ.* **1985**, *62*, 536.
- Puignou, L.; Llauradó, M. "An Experimental Introduction to Interlaboratory Exercises in Analytical Chemistry," *J. Chem. Educ.* **2005**, *82*, 1079–1081.
- Quintar, S. E.; Santagata, J. P.; Villegas, O. I.; Cortinez, V. A. "Detection of Method Effects on Quality of Analytical Data," *J. Chem. Educ.* **2003**, *80*, 326–329.
- Richardson, T. H. "Reproducible Bad Data for Instruction in Statistical Methods," *J. Chem. Educ.* **1991**, *68*, 310–311.
- Salzsieder, J. C. "Statistical Analysis Experiment for Freshman Chemistry Lab," *J. Chem. Educ.* **1995**, *72*, 623.
- Samide, M. J. "Statistical Comparison of Data in the Analytical Laboratory," *J. Chem. Educ.* **2004**, *81*, 1641–1643.
- Sheeran, D. "Copper Content in Synthetic Copper Carbonate: A Statistical Comparison of Experimental and Expected Results," *J. Chem. Educ.* **1998**, *75*, 453–456.
- Spencer, R. D. "The Dependence of Strength in Plastics upon Polymer Chain Length and Chain Orientation," *J. Chem. Educ.* **1984**, *61*, 555–563.
- Stolzberg, R. J. "Do New Pennies Lose Their Shells? Hypothesis Testing in the Sophomore Analytical Chemistry Laboratory," *J. Chem. Educ.* **1998**, *75*, 1453–1455.
- Stone, C. A.; Mumaw, L. D. "Practical Experiments in Statistics," *J. Chem. Educ.* **1995**, *72*, 518–524.
- Thomasson, K.; Lofthus-Merschman, S.; Humbert, M.; Kulevsky, N. "Applying Statistics in the Undergraduate Chemistry Laboratory: Experiments with Food Dyes," *J. Chem. Educ.* **1998**, *75*, 231–233.
- Vitha, M. F.; Carr, P. W. "A Laboratory Exercise in Statistical Analysis of Data," *J. Chem. Educ.* **1997**, *74*, 998–1000.

A more comprehensive discussion of the analysis of data, covering all topics considered in this chapter as well as additional material, can be found in any textbook on statistics or data analysis; several such texts are listed here.

- Anderson, R. L. *Practical Statistics for Analytical Chemists*, Van Nostrand Reinhold: New York; 1987.
- Graham, R. C. *Data Analysis for the Chemical Sciences*, VCH Publishers: New York; 1993.
- Mark, H.; Workman, J. *Statistics in Spectroscopy*, Academic Press: Boston; 1991.

- Mason, R. L.; Gunst, R. F.; Hess, J. L. *Statistical Design and Analysis of Experiments*; Wiley: New York, 1989.
- Massart, D. L.; Vandeginste, B. G. M.; Buydens, L. M. C.; De Jong, S.; Lewi, P. J.; Smeyers-Verbeke, J. *Handbook of Chemometrics and Qualimetrics*, Elsevier: Amsterdam, 1997.
- Miller, J. C.; Miller, J. N. *Statistics for Analytical Chemistry*, Ellis Horwood PTR Prentice-Hall: New York; 3rd Edition, 1993.
- *NIST/SEMATECH e-Handbook of Statistical Methods*, http://www.itl.nist.gov/div898/handbook/, 2006.
- Sharaf, M. H.; Illman, D. L.; Kowalski, B. R. *Chemometrics*, Wiley-Interscience: New York; 1986.

The importance of defining statistical terms is covered in the following papers.

- Analytical Methods Committee “Terminology—the key to understanding analytical science. Part 1: Accuracy, precision and uncertainty,” AMC Technical Brief No. 13, Sept. 2003 (http://www.rsc.org/lap/rsccom/amc/amc_index.htm).
- Goedart, M. J.; Verdonk, A. H. “The Development of Statistical Concepts in a Design-Oriented Laboratory Course in Scientific Measuring,” *J. Chem. Educ.* **1991**, *68*, 1005–1009.
- Sánchez, J. M. “Teaching Basic Applied Statistics in University Chemistry Courses: Students’ Misconceptions,” *Chem. Educator* **2006**, *11*, 1–4.
- Thompson, M. “Towards a unified model of errors in analytical measurements,” *Analyst* **2000**, *125*, 2020–2025.
- Treptow, R. S. “Precision and Accuracy in Measurements,” *J. Chem. Educ.* **1998**, *75*, 992–995.

The detection of outliers, particularly when working with a small number of samples, is discussed in the following papers.

- Analytical Methods Committee “Robust Statistics—How Not To Reject Outliers Part 1. Basic Concepts,” *Analyst* **1989**, *114*, 1693–1697.
- Analytical Methods Committee “Robust Statistics—How Not to Reject Outliers Part 2. Inter-laboratory Trials,” *Analyst* **1989**, *114*, 1699–1702.
- Analytical Methods Committee “Robust statistics: a method of coping with outliers,” AMC Technical Brief No. 6, April 2001 (http://www.rsc.org/lap/rsccom/amc/amc_index.htm).
- Efstathiou, C. “Stochastic Calculation of Critical Q-Test Values for the Detection of Outliers in Measurements,” *J. Chem. Educ.* **1992**, *69*, 773–736.
- Efstathiou, C. “Estimation of type 1 error probability from experimental Dixon’s Q parameter on testing for outliers within small data sets,” *Talanta* **2006**, *69*, 1068–1071.
- Kelly, P. C. “Outlier Detection in Collaborative Studies,” *Anal. Chem.* **1990**, *73*, 58–64.
- Mitschele, J. “Small Sample Statistics,” *J. Chem. Educ.* **1991**, *68*, 470–473.

The following papers provide additional information on error and uncertainty, including the propagation of uncertainty.

- Andraos, J. “On the Propagation of Statistical Errors for a Function of Several Variables,” *J. Chem. Educ.* **1996**, *73*, 150–154.
- Donato, H.; Metz, C. “A Direct Method for the Propagation of Error Using a Personal Computer Spreadsheet Program,” *J. Chem. Educ.* **1988**, *65*, 867–868.
- Gordon, R.; Pickering, M.; Bisson, D. “Uncertainty Analysis by the ‘Worst Case’ Method,” *J. Chem. Educ.* **1984**, *61*, 780–781.
- Guare, C. J. “Error, Precision and Uncertainty,” *J. Chem. Educ.* **1991**, *68*, 649–652.
- Guedens, W. J.; Yperman, J.; Mullens, J.; Van Poucke, L. C.; Pauwels, E. J. “Statistical Analysis of Errors: A Practical Approach for an Undergraduate Chemistry Lab Part 1. The Concept,” *J. Chem. Educ.* **1993**, *70*, 776–779
- Guedens, W. J.; Yperman, J.; Mullens, J.; Van Poucke, L. C.; Pauwels, E. J. “Statistical Analysis of Errors: A Practical Approach for an Undergraduate Chemistry Lab Part 2. Some Worked Examples,” *J. Chem. Educ.* **1993**, *70*, 838–841.
- Heydorn, K. “Detecting Errors in Micro and Trace Analysis by Using Statistics,” *Anal. Chim. Acta* **1993**, *283*, 494–499.
- Hund, E.; Massart, D. L.; Smeyers-Verbeke, J. “Operational definitions of uncertainty,” *Trends Anal. Chem.* **2001**, *20*, 394–406.
- Kragten, J. “Calculating Standard Deviations and Confidence Intervals with a Universally Applicable Spreadsheet Technique,” *Analyst* **1994**, *119*, 2161–2165.
- Taylor, B. N.; Kuyatt, C. E. “Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results,” NIST Technical Note 1297, 1994.
- Yates, P. C. “A Simple Method for Illustrating Uncertainty Analysis,” *J. Chem. Educ.* **2001**, *78*, 770–771.

Consult the following resources for a further discussion of detection limits.

- Boumans, P. W. J. M. “Detection Limits and Spectral Interferences in Atomic Emission Spectrometry,” *Anal. Chem.* **1984**, *66*, 459A–467A.

- Currie, L. A. “Limits for Qualitative Detection and Quantitative Determination: Application to Radiochemistry,” *Anal. Chem.* **1968**, *40*, 586–593.
- Currie, L. A. (ed.) *Detection in Analytical Chemistry: Importance, Theory and Practice*, American Chemical Society: Washington, D. C., 1988.
- Ferrus, R.; Egea, M. R. “Limit of discrimination, limit of detection and sensitivity in analytical systems,” *Anal. Chim. Acta* **1994**, *287*, 119–145.
- Glaser, J. A.; Foerst, D. L.; McKee, G. D.; Quave, S. A.; Budde, W. L. “Trace analyses for wastewaters,” *Environ. Sci. Technol.* **1981**, *15*, 1426–1435.
- Kimbrough, D. E.; Wakakuwa, J. “Quality Control Level: An Introduction to Detection Levels,” *Environ. Sci. Technol.* **1994**, *28*, 338–345.

The following resources provide additional information on using Excel, including reports of errors in its handling of some statistical procedures.

- McCollough, B. D.; Wilson, B. “On the accuracy of statistical procedures in Microsoft Excel 2000 and Excel XP,” *Comput. Statist. Data Anal.* **2002**, *40*, 713–721.
- Morgon, S. L.; Deming, S. N. “Guide to Microsoft *Excel* for calculations, statistics, and plotting data,” (http://www.chem.sc.edu/faculty/morgan/resources/Excel/Excel_Guide_Morgan.pdf).
- Pottel, H. “Statistical flaws in Excel,” (http://www.coventry.ac.uk/ec/~nhunt/pottel.pdf).

To learn more about using R, consult the following resources.

- Chambers, J. M. *Software for Data Analysis: Programming with R*, Springer: New York, 2008.
- Maindonald, J.; Braun, J. *Data Analysis and Graphics Using R*, Cambridge University Press: Cambridge, UK, 2003.
- Sarkar, D. *Lattice: Multivariate Data Visualization With R*, Springer: New York, 2008.

The following papers provide insight into visualizing data.

- Analytical Methods Committee “Representing data distributions with kernel density estimates,” AMC Technical Brief, March 2006 (http://www.rsc.org/lap/rsccom/amc/amc_index.htm).
- Frigge, M.; Hoaglin, D. C.; Iglewicz, B. “Some Implementations of the Boxplot,” *The American Statistician* **1989**, *43*, 50–54.

Gathered here are links to on-line computational tools, simulations, and tutorials, many of which are found on the Analytical Sciences Digital Library.

- Applets for Statistics (link).
- GraphPad QuickCalcs: Free On-Line Calculators (link).
- Introduction to Data Analysis (link).
- Introduction to Probability and Statistics (link).
- Overway, K. “Population versus Sampling Statistics: A Spreadsheet Exercise,” *J. Chem. Educ.* **2008** *85*, 749 (link).
- Van Bramer, S. E. “A Brief Introduction to the Gaussian Distribution, Sample Statistics, and the Student’s t Statistic,” *J. Chem. Educ.* **2007**, *84*, 1231 (link).
- Web Tutorials in Chemistry—Statistics (link).

Chapter 5

Although there are many experiments in the literature that incorporate external standards, the method of standard additions, or internal standards, the issue of choosing a method standardization is not the experiment’s focus. One experiment designed to consider the issue of selecting a method of standardization is given here.

- Harvey, D. T. “External Standards or Standard Additions? Selecting and Validating a Method of Standardization,” *J. Chem. Educ.* **2002**, *79*, 613–615.

In addition to the texts listed as suggested readings in Chapter 4, the following text provide additional details on linear regression.

- Draper, N. R.; Smith, H. *Applied Regression Analysis*, 2nd. ed.; Wiley: New York, 1981.

The following articles providing more details about linear regression.

- Analytical Methods Committee “Is my calibration linear?” AMC Technical Brief, December 2005 (http://www.rsc.org/pdf/amc/brief3.pdf).

- Badertscher, M.; Pretsch, E. “Bad results from good data,” *Trends Anal. Chem.* **2006**, *25*, 1131–1138.
- Boqué, R.; Rius, F. X.; Massart, D. L. “Straight Line Calibration: Something More Than Slopes, In• tercepts, and Correlation Coefficients,” *J. Chem. Educ.* **1993**, *70*, 230–232.
- Danzer, K.; Currie, L. A. “Guidelines for Calibration in Analytical Chemistry. Part 1. Fundamentals and Single Component Calibration,” *Pure Appl. Chem.* **1998**, *70*, 993–1014.
- Henderson, G. “Lecture Graphic Aids for Least-Squares Analysis,” *J. Chem. Educ.* **1988**, *65*, 1001–1003.
- Logan, S. R. “How to Determine the Best Straight Line,” *J. Chem. Educ.* **1995**, *72*, 896–898.
- Miller, J. N. “Basic Statistical Methods for Analytical Chemistry. Part 2. Calibration and Regression Methods,” *Analyst* **1991**, *116*, 3–14.
- Renman, L., Jagner, D. “Asymmetric Distribution of Results in Calibration Curve and Standard Addition Evaluations,” *Anal. Chim. Acta* **1997**, *357*, 157–166.
- Rodriguez, L. C.; Gamiz-Gracia; Almansa-Lopez, E. M.; Bosque-Sendra, J. M. “Calibration in chemical measurement processes. II. A methodological approach,” *Trends Anal. Chem.* **2001**, *20*, 620–636.

Useful papers providing additional details on the method of standard additions are gathered here.

- Bader, M. “A Systematic Approach to Standard Addition Methods in Instrumental Analysis,” *J. Chem. Educ.* **1980**, *57*, 703–706.
- Brown, R. J. C.; Roberts, M. R.; Milton, M. J. T. “Systematic error arising form ‘Sequential’ Standard Addition Calibrations. 2. Determination of Analyte Mass Fraction in Blank Solutions,” *Anal. Chim. Acta* **2009**, *648*, 153–156.
- Brown, R. J. C.; Roberts, M. R.; Milton, M. J. T. “Systematic error arising form ‘Sequential’ Standard Addition Calibrations: Quantification and correction,” *Anal. Chim. Acta* **2007**, *587*, 158–163.
- Bruce, G. R.; Gill, P. S. “Estimates of Precision in a Standard Additions Analysis,” *J. Chem. Educ.* **1999**, *76*, 805–807.
- Kelly, W. R.; MacDonald, B. S.; Guthrie “Gravimetric Approach to the Standard Addition Method in Instrumental Analysis. 1.” *Anal. Chem.* **2008**, *80*, 6154–6158.
- Nimura, Y.; Carr, M. R. “Reduction of the Relative Error in the Standard Additions Method,” *Analyst* **1990**, *115*, 1589–1595.

The following papers discusses the importance of weighting experimental data when use linear regression.

- Analytical Methods Committee “Why are we weighting?” AMC Technical Brief, June 2007 (http://www.rsc.org/images/brief27_tcm18-92066.pdf)
- Karolczak, M. “To Weight or Not to Weight? An Analyst’s Dilemma,” *Current Separations* **1995**, *13*, 98–104.

Algorithms for performing a linear regression with errors in both *X* and *Y* are discussed in the following papers. Also included here are papers that address the difficulty of using linear regression to compare two analytical methods.

- Irvin, J. A.; Quickenden, T. L. “Linear Least Squares Treatment When There are Errors in Both x and y,” *J. Chem. Educ.* **1983**, *60*, 711–712.
- Kalantar, A. H. “Kerrich’s Method for y = αx Data When Both y and x Are Uncertain,” *J. Chem. Educ.* **1991**, *68*, 368–370.
- Macdonald, J. R.; Thompson, W. J. “Least-Squares Fitting When Both Variables Contain Errors: Pitfalls and Possibilities,” *Am. J. Phys.* **1992**, *60*, 66–73.
- Martin, R. F. “General Deming Regression for Estimating Systematic Bias and Its Confidence Interval in Method-Comparison Studies,” *Clin. Chem.* **2000**, *46*, 100–104.
- Ogren, P. J.; Norton, J. R. “Applying a Simple Linear Least-Squares Algorithm to Data with Uncertainties in Both Variables,” *J. Chem. Educ.* **1992**, *69*, A130–A131.
- Ripley, B. D.; Thompson, M. “Regression Techniques for the Detection of Analytical Bias,” *Analyst* **1987**, *112*, 377–383.

Outliers present a problem for a linear regression analysis. The following papers discuss the use of robust linear regression techniques.

- Glaister, P. “Robust Linear Regression Using Thiel’s Method,” *J. Chem. Educ.* **2005**, *82*, 1472–1473.
- Glasser, L. “Dealing with Outliers: Robust, Resistant Regression,” *J. Chem. Educ.* **2007**, *84*, 533–534.
- Ortiz, M. C.; Sarabia, L. A.; Herrero, A. “Robust regression techniques. A useful alternative for the detection of outlier data in chemical analysis,” *Talanta* **2006**, *70*, 499–512.

The following papers discusses some of the problems with using linear regression to analyze data that has been mathematically transformed into a linear form, as well as alternative methods of evaluating curvilinear data.

- Chong, D. P. “On the Use of Least Squares to Fit Data in Linear Form,” *J. Chem. Educ.* **1994**, *71*, 489–490.
- Hinshaw, J. V. “Nonlinear Calibration,” *LCGC* **2002**, *20*, 350–355.
- Lieb, S. G. “Simplex Method of Nonlinear Least-Squares - A Logical Complementary Method to Linear Least-Squares Analysis of Data,” *J. Chem. Educ.* **1997**, *74*, 1008–1011.
- Zielinski, T. J.; Allendoerfer, R. D. “Least Squares Fitting of Nonlinear Data in the Undergraduate Laboratory,” *J. Chem. Educ.* **1997**, *74*, 1001–1007.

More information on multivariate and multiple regression can be found in the following papers.

- Danzer, K.; Otto, M.; Currie, L. A. “Guidelines for Calibration in Analytical Chemistry. Part 2. Multispecies Calibration,” Pure Appl. Chem. 2004, 76, 1215–1225.
- Escandar, G. M.; Faber, N. M.; Goicoechea, H. C.; de la Pena, A. M.; Olivieri, A.; Poppi, R. J. “Second- and third-order multivariate calibration: data, algorithms and applications,” *Trends Anal. Chem.* **2007**, *26*, 752–765.
- Kowalski, B. R.; Seasholtz, M. B. “Recent Developments in Multivariate Calibration,” *J. Chemometrics* **1991**, *5*, 129–145.
- Lang, P. M.; Kalivas, J. H. “A Global Perspective on Multivariate Calibration Methods,” *J. Chemometrics* **1993**, *7*, 153–164.
- Madden, S. P.; Wilson, W.; Dong, A.; Geiger, L.; Mecklin, C. J. “Multiple Linear Regression Using a Graphing Calculator,” J. Chem. Educ. 2004, 81, 903–907.
- Olivieri, A. C.; Faber, N. M.; Ferré, J.; Boqué, R.; Kalivas, J. H.; Mark, H. “Uncertainty Estimation and Figures of Merit for Multivariate Calibration,” *Pure Appl. Chem.* **2006**, *78*, 633–661.

An additional discussion on method blanks, including the use of the total Youden blank, is found in the following papers.

- Cardone, M. J. “Detection and Determination of Error in Analytical Methodology. Part II. Correction for Corrigible Systematic Error in the Course of Real Sample Analysis,” *J. Assoc. Off. Anal. Chem.* **1983**, *66*, 1283–1294.
- Cardone, M. J. “Detection and Determination of Error in Analytical Methodology. Part IIB. Direct Calculational Technique for Making Corrigible Systematic Error Corrections,” *J. Assoc. Off. Anal. Chem.* **1985**, *68*, 199-202.
- Ferrus, R.; Torrades, F. “Bias-Free Adjustment of Analytical Methods to Laboratory Samples in Routine Analytical Procedures,” *Anal. Chem.* **1988**, *60*, 1281–1285.
- Vitha, M. F.; Carr, P. W.; Mabbott, G. A. “Appropriate Use of Blanks, Standards, and Controls in Chemical Measurements,” *J. Chem. Educ.* **2005**, *82*, 901–902.

There are a variety of computational packages for completing linear regression analyses. These papers provide details on there use in a variety of contexts.

- Espinosa-Mansilla, A.; de la Peña, A. M.; González-Gómez, D. “Using Univariate Linear Regression Calibration Software in the MATLAB Environment. Application to Chemistry Laboratory Practices,” *Chem. Educator* **2005**, *10*, 1–9.
- Harris, D. C. “Nonlinear Least-Squares Curve Fitting with Microsoft Excel Solver,” *J. Chem. Educ.* **1998**, *75*, 119–121.
- Kim, M. S.; Bukart, M.; Kim, M. H. “A Method Visual Interactive Regression,” *J. Chem. Educ.* **2006**, *83*, 1884.
- Machuca-Herrera, J. G. “Nonlinear Curve Fitting with Spreadsheets,” *J. Chem. Educ.* **1997**, *74*, 448–449.
- Smith, E. T.; Belogay, E. A.; Hõim “Linear Regression and Error Analysis for Calibration Curves and Standard Additions: An Excel Spreadsheet Exercise for Undergraduates,” Chem. Educator 2010, 15, 100–102.
- Smith, E. T.; Belogay, E. A.; Hõim “Using Multiple Linear Regression to Analyze Mixtures: An Excel Spreadsheet Exercise for Undergraduates,” Chem. Educator 2010, 15, 103–107.
- Young, S. H.; Wierzbicki, A. “Mathcad in the Chemistry Curriculum. Linear Least-Squares Regression,” *J. Chem. Educ.* **2000**, *77*, 669.
- Young, S. H.; Wierzbicki, A. “Mathcad in the Chemistry Curriculum. Non-Linear Least-Squares Regression,” *J. Chem. Educ.* **2000**, *77*, 669.

Gathered here are links to on-line computational tools, simulations, and tutorials, many of which are found on the Analytical Sciences Digital Library.

- Multiple Regression (link).
- Non-Parametric Regression with Errors in X and Y (link).
- Linear Regression Tutorial (link).
- Modeling Data Tutorial (link).

Chapter 6

The following experiments involve the experimental determination of equilibrium constants, the characterization of buffers, and, in some cases, demonstrate the importance of activity effects.

- "The Effect of Ionic Strength on an Equilibrium Constant (A Class Study)" in *Chemical Principles in Practice*, J. A. Bell, Ed., Addison-Wesley: Reading, MA, 1967.
- "Equilibrium Constants for Calcium Iodate Solubility and Iodic Acid Dissociation" in *Chemical Principles in Practice*, J. A. Bell, Ed., Addison-Wesley: Reading, MA, 1967.
- "The Solubility of Silver Acetate" in *Chemical Principles in Practice*, J. A. Bell, Ed., Addison-Wesley: Reading, MA, 1967.
- Cobb, C. L.; Love, G. A. "Iron(III) Thiocyanate Revisited: A Physical Chemistry Equilibrium Lab Incorporating Ionic Strength Effects," *J. Chem. Educ.* **1998**, *75*, 90–92.
- Green, D. B.; Rechtsteiner, G.; Honodel, A. "Determination of the Thermodynamic Solubility Product, K_{sp}, of PbI_2 Assuming Nonideal Behavior," *J. Chem. Educ.* **1996**, *73*, 789–792.
- Russo, S. O.; Hanania, I. H. "Buffer Capacity," *J. Chem. Educ.* **1987**, *64*, 817–819.
- Stolzberg, R. J. "Discovering a Change in Equilibrium Constant with Change in Ionic Strength," *J. Chem. Educ.* **1999**, *76*, 640–641.
- Wiley, J. D. "The Effect of Ionic Strength on the Solubility of an Electrolyte," *J. Chem. Educ.* **2004**, *81*, 1644–1646.

A nice discussion of Berthollet's discovery of the reversibility of reactions is found in

- Roots-Bernstein, R. S. *Discovering*, Harvard University Press: Cambridge, MA, 1989.

The following texts provide additional coverage of equilibrium chemistry.

- Butler, J. N. *Ionic Equilibria: A Mathematical Approach*; Addison-Wesley: Reading, MA, 1964.
- Butler, J. N. *Solubility and pH Calculations*; Addison-Wesley: Reading, MA, 1973.
- Fernando, Q.; Ryan, M. D. *Calculations in Analytical Chemistry*, Harcourt Brace Jovanovich: New York, 1982.
- Freiser, H.; Fernando, Q. *Ionic Equilibria in Analytical Chemistry*, Wiley: New York, 1963.
- Freiser, H. *Concepts and Calculations in Analytical Chemistry*, CRC Press: Boca Raton, 1992.
- Gordus, A. A. *Schaum's Outline of Analytical Chemistry*; McGraw-Hill: New York, 1985.
- Ramette, R. W. *Chemical Equilibrium and Analysis*, Addison-Wesley: Reading, MA, 1981.

The following papers discuss a variety of general aspects of equilibrium chemistry.

- Gordus, A. A. "Chemical Equilibrium I. The Thermodynamic Equilibrium Concept," *J. Chem. Educ.* **1991**, *68*, 138–140.
- Gordus, A. A. "Chemical Equilibrium II. Deriving an Exact Equilibrium Equation," *J. Chem. Educ.* **1991**, *68*, 215–217.
- Gordus, A. A. "Chemical Equilibrium III. A Few Math Tricks," *J. Chem. Educ.* **1991**, *68*, 291–293.
- Gordus, A. A. "Chemical Equilibrium IV. Weak Acids and Bases," *J. Chem. Educ.* **1991**, *68*, 397–399.
- Gordus, A. A. "Chemical Equilibrium VI. Buffer Solutions," *J. Chem. Educ.* **1991**, *68*, 656–658.
- Gordus, A. A. "Chemical Equilibrium VII. Precipitates, "*J. Chem. Educ.* **1991**, *68*, 927–930.
- Thomson, B. M.; Kessick, M. A. "On the Preparation of Buffer Solutions," *J. Chem. Educ.* **1981**, *58*, 743-746.
- Weltin, E. "Are the Equilibrium Concentrations for a Chemical Reaction Always Uniquely Determined by the Initial Concentrations?" *J. Chem. Educ.* **1990**, *67*, 548.
- Weltin, E. "Are the Equilibrium Compositions Uniquely Determined by the Initial Compositions? Properties of the Gibbs Free Energy Function," J. Chem. Educ. 1995, 72, 508–511.

Collected here are a papers discussing a variety of approaches to solving equilibrium problems.

- Ault, A. "Do pH in Your Head," *J. Chem. Educ.* **1999**, *76*, 936–938.
- Chaston, S. "Calculating Complex Equilibrium Concentrations by a Next Guess Factor Method," *J. Chem. Educ.* **1993**, *70*, 622–624.
- Donato, H. "Graphing Calculator Strategies for Solving Chemical Equilibrium Problems," J. Chem. Educ. 1999, 76, 632–634.
- Olivieri, A. C. "Solution of Acid-Base Equilibria by Successive Approximations," *J. Chem. Educ.* **1990**, *67*, 229–231.
- Weltin, E. "A Numerical Method to Calculate Equilibrium Concentrations for Single-Equation Systems," *J. Chem. Educ.* **1991**, *68*, 486–487.
- Weltin, E. "Calculating Equilibrium Concentrations," *J. Chem. Educ.* **1992**, *69*, 393–396.

- Weltin, E. “Calculating Equilibrium Concentrations for Stepwise Binding of Ligands and Polyprotic Acid-Base Systems,” J. *Chem. Educ.* **1993**, *70*, 568–571.
- Weltin, E. “Equilibrium Calculations are Easier Than You Think - But You do Have to Think!” *J. Chem. Educ.* **1993**, *70*, 571–573.
- Weltin, E. “Calculating Equilibrium Concentrations by Iteration: Recycle Your Approximations,” J. Chem. Educ. 1995, 72, 36–38.

Additional historical background on the development of the Henderson-Hasselbalch equation is provided by the following papers.

- de Levie, R. “The Henderson Approximation and the Mass Action Law of Guldberg and Waage,” *Chem. Educator* **2002**, *7*, 132–135.
- de Levie, R. “The Henderson-Hasselbalch Equation: Its History and Limitations,” *J. Chem. Educ.* **2003**, *80*, 146.

A simulation is a useful tool for helping students gain an intuitive understanding of a topic. Gathered here are some simulations for teaching equilibrium chemistry.

- Edmonson, L. J.; Lewis, D. L. “Equilibrium Principles: A Game for Students,” *J. Chem. Educ.* **1999**, *76*, 502.
- Huddle, P. A.; White, M. W.; Rogers, F. “Simulations for Teaching Chemical Equilibrium,” *J. Chem. Educ.* **2000**, *77*, 920–926.

The following papers provide additional resources on ionic strength, activity, and the effect of ionic strength and activity on equilibrium reactions and pH.

- Clark, R. W.; Bonicamp, J. M. “The Ksp-Solubility Conundrum,” *J. Chem. Educ.* **1998**, *75*, 1182–1185.
- de Levie, R. “On Teaching Ionic Activity Effects: What, When, and Where?” *J. Chem. Educ.* **2005**, *82*, 878–884.
- McCarty, C. G.; Vitz, E. “pH Paradoxes: Demonstrating That It Is Not True That $\text{pH} = -\log[\text{H}^+]$,” *J. Chem. Educ.* **2006**, *83*, 752–757.
- Ramshaw, J. D. “Fugacity and Activity in a Nutshell,” *J. Chem. Educ.* **1995**, *72*, 601–603.
- Sastre de Vicente, M. E. “The Concept of Ionic Strength Eighty Years After Its Introduction,” *J. Chem. Educ.* **2004**, *81*, 750–753.
- Solomon, T. “The Definition and Unit of Ionic Strength,” *J. Chem. Educ.* **2001**, *78*, 1691–1692.

For a contrarian’s view of equilibrium chemistry, please see the following papers.

- Hawkes, S. J. “Buffer Calculations Deceive and Obscure,” *Chem. Educator*, **1996**, *1*, 1–8.
- Hawkes, S. J. “What Should We Teach Beginners About Solubility and Solubility Products?” *J. Chem. Educ.* **1998**, *75*, 1179–1181.
- Hawkes, S. J. “Complexation Calculations are Worse Than Useless,” *J. Chem. Educ.* **1999**, *76*, 1099–1100.
- Hawkes, S. J. “Easy Deviation of $\text{pH} \approx (\text{p}K_{a1} + \text{p}K_{a2})/2$ Using Autoprotolysis of HA^-: Doubtful Value of the Supposedly More Rigorous Equation,” *J. Chem. Educ.* **2000**, *77*, 1183–1184. See, also, an exchange of letters between J. J. Roberts and S. J. Hawkes, *J. Chem. Educ.* **2002**, *79*, 161–162.

Chapter 7

The following set of experiments and class exercises introduce students to the importance of sampling on the quality of analytical results.

- Bauer, C. F. “Sampling Error Lecture Demonstration,” *J. Chem. Educ.* **1985**, *62*, 253.
- Canaes, L. S.; Brancalion, M. L.; Rossi, A. V.; Rath, S. “Using Candy Samples to Learn About Sampling Techniques and Statistical Evaluation of Data,” *J. Chem. Educ.* **2008**, *85*, 1083–1088.
- Clement, R. E. “Environmental Sampling for Trace Analysis,” *Anal. Chem.* **1992**, *64*, 1076A–1081A.
- Dunn, J. G.; Phillips, D. N.; van Bronswijk, W. “An Exercise to Illustrate the Importance of Sample Preparation in Chemical Analysis,” *J. Chem. Educ.* **1997**, *74*, 1188–1191.
- Fritz, M. D. “A Demonstration of Sample Segregation,” *J. Chem. Educ.* **2005**, *82*, 255–256.
- Guy, R. D.; Ramaley, L.; Wentzell, P. D. “An Experiment in the Sampling of Solids for Chemical Analysis”, J. Chem. Educ. 1998, 75, 1028-1033.
- Hartman, J. R. “An In-Class Experiment to Illustrate the Importance of Sampling Techniques and Statistical Analysis of Data to Quantitative Analysis Students,” *J. Chem. Educ.* **2000**, *77*, 1017–1018.
- Harvey, D. T. “Two Experiments Illustrating the Importance of Sampling in a Quantitative Chemical Analysis,” *J. Chem. Educ.* **2002**, *79*, 360–363.

- Herrington, B. L. “A Demonstration of the Necessity for Care in Sampling,” *J. Chem. Educ.* **1937**, *14*, 544.
- Kratochvil, B.; Reid, R. S.; Harris, W. E. “Sampling Error in a Particulate Mixture”, *J. Chem. Educ.* **1980**, *57*, 518–520.
- Ross, M. R. “A Classroom Exercise in Sampling Technique,” *J. Chem. Educ.* **2000**, *77*, 1015–1016.
- Settle, F. A.; Pleva, M. “The Weakest Link Exercise,” *Anal. Chem.* **1999**, *71*, 538A–540A.
- Vitt, J. E.; Engstrom, R. C. “Effect of Sample Size on Sampling Error,” *J. Chem. Educ.* **1999**, *76*, 99–100.

The following experiments describe homemade sampling devices for collecting samples in the field.

- Delumyea, R. D.; McCleary, D. L. “A Device to Collect Sediment Cores,” *J. Chem. Educ.* **1993**, *70*, 172–173.
- Rockwell, D. M.; Hansen, T. “Sampling and Analyzing Air Pollution,” *J. Chem. Educ.* **1994**, *71*, 318–322.
- Saxena, S., Upadhyay, R.; Upadhyay, P. “A Simple and Low-Cost Air Sampler,” *J. Chem. Educ.* **1996**, *73*, 787–788.
- Shooter, D. “Nitrogen Dioxide and Its Determination in the Atmosphere,” *J. Chem. Educ.* **1993**, *70*, A133–A140.

The following experiments introduce students to methods for extracting analytes from their matrix.

- “Extract-Clean™ SPE Sample Preparation Guide Volume 1”, Bulletin No. 83, Alltech Associates, Inc. Deerfield, IL.
- Freeman, R. G.; McCurdy, D. L. “Using Microwave Sample Decomposition in Undergraduate Analytical Chemistry,” *J. Chem. Educ.* **1998**, *75*, 1033–1032.
- Snow, N. H.; Dunn, M.; Patel, S. “Determination of Crude Fat in Food Products by Supercritical Fluid Extraction and Gravimetric Analysis,” *J. Chem. Educ.* **1997**, *74*, 1108–1111.
- Yang, M. J.; Orton, M. L.; Pawliszyn, J. “Quantitative Determination of Caffeine in Beverages Using a Combined SPME-GC/MS Method,” *J. Chem. Educ.* **1997**, *74*, 1130–1132.

The following paper provides a general introduction to the terminology used in describing sampling.

- “Terminology—The key to understanding analytical science. Part 2: Sampling and sample preparation,” AMC Technical Brief No. 19, March 2005 (link).
- Majors, R. E. “Nomenclature for Sampling in Analytical Chemistry” *LC•GC* **1992**, *10*, 500–506.

Further information on the statistics of sampling is covered in the following papers and textbooks.

- “What is uncertainty from sampling, and why is it important?” AMC Technical Brief No. 16A, June 2004 (link).
- “Analytical and sampling strategy, fitness for purpose, and computer games,” AMC Technical Brief No. 20, August 2005 (link).
- “Measurement uncertainty arising from sampling: the new Eurachem Guide,” AMC Technical Brief No. 31, July 2008 (link).
- *Sampling for Analytical Purpose*, Gy, P. ed., Wiley: NY, 1998.
- Baiulescu, G. E.; Dumitrescu, P.; Zuaravescu, P. G. *Sampling*, Ellis Horwood: NY, 1991.
- Cohen, R. D. “How the Size of a Random Sample Affects How Accurately It Represents a Population,” *J. Chem. Educ.* **1992**, *74*, 1130–1132.
- Efstathiou, C. E. “On the sampling variance of ultra-dilute solutions,” *Talanta* **2000**, *52*, 711–715.
- Gerlach, R. W.; Dobb, D. E.; Raab, G. A.; Nocerino, J. M. *J. Chemom.* **2002**, *16*, 321–328.
- Gy, P. M. *Sampling of Particulate Materials: Theory and Practice*; Elsevier: Amsterdam, 1979.
- Gy, P. M. *Sampling of Heterogeneous and Dynamic Materials: Theories of Heterogeneity, Sampling and Homogenizing*; Elsevier: Amsterdam, 1992.
- Kratochvil, B.; Taylor, J. K. “Sampling for Chemical Analysis,” *Anal. Chem.* **1981**, *53*, 924A–938A.
- Kratochvil, B.; Goewie, C. E.; Taylor, J. K. “Sampling Theory for Environmental Analysis,” *Trends Anal. Chem.* **1986**, *5*, 253–256.
- Meyer, V. R. *LC•GC* **2002**, *20*, 106–112.
- Rohlf, F. J.; Akçakaya, H. R.; Ferraro, S. P. “Optimizing Composite Sampling Protocols,” *Environ. Sci. Technol.* **1996**, *30*, 2899–2905.
- Smith, R.; James, G. V. *The Sampling of Bulk Materials*; Royal Society of Chemistry: London, 1981.

The process of collecting a sample presents a variety of difficulties, particularly with respect to the analyte’s integrity. The following papers provide representative examples of sampling problems.

- Barceló, D.; Hennion, M. C. “Sampling of Polar Pesticides from Water Matrices,” *Anal. Chim. Acta* **1997**, *338*, 3–18.
- Batley, G. E.; Gardner, D. “Sampling and Storage of Natural Waters for Trace Metal Analysis,” *Wat. Res.* **1977**, *11*, 745–756.

- Benoit, G.; Hunter, K. S.; Rozan, T. F. “Sources of Trace Metal Contamination Artifacts during Collection, Handling, and Analysis of Freshwaters,” *Anal. Chem.* **1997**, *69*, 1006–1011
- Brittain, H. G. “Particle-Size Distribution II: The Problem of Sampling Powdered Solids,” *Pharm. Technol.* July **2002**, 67–73.
- Ramsey, M. H. “Measurement Uncertainty Arising from Sampling: Implications for the Objectives of Geoanalysis,” *Analyst*, **1997**, *122*, 1255–1260.
- Seiler, T-B; Schulze, T.; Hollert, H. “The risk of altering soil and sediment samples upon extract preparation for analytical and bio-analytical investigations—a review,” *Anal. Bioanal. Chem.* **2008**, 390, 1975–1985.

The following texts and articles provide additional information on methods for separating analytes and interferents.

- “Guide to Solid Phase Extraction,” Bulletin 910, Sigma-Aldrich, 1998.
- “Solid Phase Microextraction: Theory and Optimization of Conditions,” Bulletin 923, Sigma-Aldrich, 1998.
- *Microwave-Enhanced Chemistry: Fundamentals, Sample Preparation, and Applications*, Kingston, H. M.; Haswell, S. J., eds.; American Chemical Society: Washington, D.C., 1997.
- Anderson, R. *Sample Pretreatment and Separation*, Wiley: Chichester, 1987.
- Bettiol, C.; Stievano, L.; Bertelle, M.; Delfino, F.; Argese, E. “Evaluation of microwave-assisted acid extraction procedures for the determination of metal content and potential bioavailability in sediments,” Appl. Geochem. 2008, 23, 1140–1151.
- Compton, T. R. *Direct Preconcentration Techniques*, Oxford Science Publications: Oxford, 1993.
- Compton, T. R. *Complex-Formation Preconcentration Techniques*, Oxford Science Publications: Oxford, 1993.
- Hinshaw, J. V. “Solid-Phase Microextraction,” *LC•GC Europe* **2003**, *December*, 2–5.
- Karger, B. L.; Snyder, L. R.; Harvath, C. *An Introduction to Separation Science*, Wiley-Interscience: N. Y.; 1973.
- Majors, R. E.; Raynie, D. E. “Sample Preparation and Solid-Phase Extraction”, *LC•GC* **1997**, *15*, 1106–1117.
- Luque de Castro, M. D.; Priego-Capote, F.; Sánchez-Ávila, N. “Is dialysis alive as a membrane-based separation technique?” *Trends Anal. Chem.* **2008**, *27*, 315–326.
- Mary, P.; Studer, V.; Tabeling, P. “Microfluidic Droplet-Based Liquid–Liquid Extraction,” *Anal. Chem.* **2008**, *80*, 2680–2687.
- Miller, J. M. *Separation Methods in Chemical Analysis*, Wiley-Interscience: N. Y.; 1975.
- Morrison, G. H.; Freiser, H. *Solvent Extraction in Analytical Chemistry*, John Wiley and Sons: N. Y.; 1957.
- Pawliszyn, J. *Solid-Phase Microextraction: Theory and Practice*, Wiley: NY, 1997.
- Pawliszyn, J. “Sample Preparation: Quo Vadis?” *Anal. Chem.* **2003**, *75*, 2543–2558.
- Sulcek, Z.; Povondra, P. *Methods of Decomposition in Inorganic Analysis*; CRC Press: Boca Raton, FL, 1989.
- Theis, A. L.; Waldack, A. J.; Hansen, S. M.; Jeannot, M. A. “Headspace Solvent Microextraction,” *Anal. Chem.* **2001**, *73*, 5651–5654.
- Thurman, E. M.; Mills, M. S. *Solid-Phase Extraction: Principles and Practice*, Wiley: NY, 1998.
- Zhang, Z.; Yang, M.; Pawliszyn, J. “Solid-Phase Microextraction,” *Anal. Chem.* **1994**, *66*, 844A–853A.

Chapter 8

The following set of experiments introduce students to the applications of gravimetry.

- Burrows, H. D.; Ellis, H. A.; Odilora, C. A. “The Dehydrochlorination of PVC,” *J. Chem. Educ.* **1995**, *72*, 448–450.
- Carmosini, N.; Ghoreshy, S. Koether, M. C. “The Gravimetric Analysis of Nickel Using a Microwave Oven,” *J. Chem. Educ.* **1997**, *74*, 986–987.
- Harris, T. M. “Revitalizing the Gravimetric Determination in Quantitative Analysis Laboratory,” *J. Chem. Educ.* **1995**, *72*, 355–356.
- Henrickson, C. H.; Robinson, P. R. “Gravimetric Determination of Calcium as $CaC_2O_4{\bullet}H_2O$,” *J. Chem. Educ.* **1979**, *56*, 341–342.
- Shaver, L. A. “Determination of Phosphates by the Gravimetric Quimociac Technique,” *J. Chem. Educ.* **2008**, *85*, 1097–1098.
- Snow, N. H.; Dunn, M.; Patel, S. “Determination of Crude Fat in Food Products by Supercritical Fluid Extraction and Gravimetric Analysis,” *J. Chem. Educ.* **1997**, *74*, 1108–1111.
- Thompson, R. Q.; Ghadiali, M. “Microwave Drying of Precipitates for Gravimetric Analysis,” *J. Chem. Educ.* **1993**, *70*, 170–171.
- Wynne, A. M. “The Thermal Decomposition of Urea,” *J. Chem. Educ.* **1987**, *64*, 180–182.

The following resources provide a general history of gravimetry.

- A History of Analytical Chemistry; Laitinen, H. A.; Ewing, G. W., Eds.; The Division of Analytical Chemistry of the American Chemical Society: Washington, D. C., 1977, pp. 10-24.
- Beck, C. M. "Classical Analysis: A Look at the Past, Present, and Future," *Anal. Chem.* **1991**, *63*, 993A–1003A; *Anal. Chem.* **1994**, *66*, 224A–239A

Consult the following texts for additional examples of inorganic and organic gravimetric methods include the following texts.

- Bassett, J.; Denney, R. C.; Jeffery, G. H.; Mendham, J. *Vogel's Textbook of Quantitative Inorganic Analysis*, Longman: London, 4th Ed., 1981.
- Erdey, L. *Gravimetric Analysis*, Pergamon: Oxford, 1965.
- Steymark, A. *Quantitative Organic Microanalysis*, The Blakiston Co.: NY, 1951.
- Wendlandt, W. W. *Thermal Methods of Analysis*, 2nd Ed. Wiley: NY. 1986.

For a review of isotope dilution mass spectrometry see the following article.

- Fassett, J. D.; Paulsen, P. J. "Isotope Dilution Mass Spectrometry for Accurate Elemental Analysis," *Anal. Chem.* **1989**, *61*, 643A–649A.

Chapter 9

The following set of experiments introduce students to the applications of titrimetry. Experiments are grouped into four categories based on the type of reaction (acid–base, complexation, redox, and precipitation). Additional experiments emphasizing potentiometric electrodes are found in Chapter 11.

Acid–base titrimetry

- Boiani, J. A. "The Gran Plot Analysis of an Acid Mixture," *J. Chem. Educ.* **1986**, *63*, 724–726.
- Castillo, C. A.; Jaramillo, A. "An Alternative Procedure for Titration Curves of a Mixture of Acids of Different Strengths," *J. Chem. Educ.* **1989**, *66*, 341.
- Clark, R. W.; White, G. D.; Bonicamp, J. M.; Watts, E. D. "From Titration Data to Buffer Capacities: A Compter Experiment for the Chemistry Lab or Lecture," *J. Chem. Educ.* **1995**, *72*, 746–750.
- Clay, J. T.; Walters, E. A.; Brabson, G. D. "A Dibasic Acid Titration for the Physical Chemistry Laboratory" *J. Chem. Educ.* **1995**, *72*, 665–667.
- Crossno, S. K; Kalbus, L. H.; Kalbus, G. E. "Determinations of Carbon Dioxide by Titration," *J. Chem. Educ.* **1996**, *73*, 175–176.
- Flowers, P. A. "Potentiometric Measurement of Transition Ranges and Titration Errors for Acid/Base Indicators," *J. Chem. Educ.* **1997**, *74*, 846–847.
- Fuchsam, W. H.; Garg, Sandhya "Acid Content of Beverages," *J. Chem. Educ.* **1990**, *67*, 67–68
- Graham. R.C.; DePew, S. "Determination of Ammonia in Household Cleaners," *J. Chem. Educ.* **1983**, *60*, 765–766.
- Kalbus, L. H.; Petrucci, R. H.; Forman, J. E.; Kalbus, G. E. "Titration of Chromate-Dichromate Mixtures," *J. Chem. Educ.* **1991**, *68*, 677–678.
- Kooser, A. S.; Jenkins, J. L.; Welch, L. E. "Acid–Base Indicators: A New Look at an Old Topic," *J. Chem. Educ.* **2001**, *78*, 1504–1506.
- Kraft, A. "The Determination of the pK_a of Multiprotic, Weak Acids by Analyzing Potentiometric Acid–Base Titration Data with Difference Plots," *J. Chem. Educ.* **2003**, *80*, 554–559.
- Murphy, J. "Determination of Phosphoric Acid in Cola Beverages," *J. Chem. Educ.* **1983**, *60*, 420–421.
- Nyasulu, F.; Barlag, R.; Macklin, J. *Chem. Educator* **2008**, *13*, 289–294.
- Ophardt, C. E. "Acid Rain Analysis by Standard Addition Titration," *J. Chem. Educ.* **1985**, *62*, 257–258.
- Partanen, J. I.; Kärki, M. H. "Determination of the Thermodynamic Dissociation Constant of a Weak Acid by Potentiometric Acid-Base Titration," *J. Chem. Educ.* **1994**, *71*, A120–A122.
- Thompson, R. Q. "Identification of Weak Acids and Bases by Titration with Primary Standards," *J. Chem. Educ.* **1988**, *65*, 179–180.
- Tucker, S. A.; Amszi, V. L.; Acree, Jr. W. E. "Studying Acid-Base Equilibria in Two-Phase Solvent Media," *J. Chem. Educ.* **1993**, *70*, 80–82.
- Tucker, S. A.; Acree, Jr., W. E. "A Student-Designed Analytical Laboratory Method," *J. Chem. Educ.* **1994**, *71*, 71–74.
- Werner, J. A.; Werner, T. C. "Multifunctional Base Unknowns in the Introductory Analytical Chemistry Lab," *J. Chem. Educ.* **1991**, *68*, 600–601.

Complexation Titrimetry

- Ceretti, H.; Hughes, E. A.; Zalts, A. “The Softening of Hard Water and Complexometric Titrations,” *J. Chem. Educ.* **1999**, *76*, 1420–1421.
- Fulton, R.; Ross, M.; Schroeder, K. “Spectrophotometric Titration of a Mixture of Calcium and Magnesium,” *J. Chem. Educ.* **1986**, *63*, 721–723.
- Novick, S. G. “Complexometric Titration of Zinc,” *J. Chem. Educ.* **1997**, *74*, 1463.
- Olsen, K. G.; Ulicny, L. J. “Reduction of Calcium Concentrations by the Brita Water Filtration System: A Practical Experiment in Titrimetry and Atomic Absorption Spectroscopy,” *J. Chem. Educ.* **2001**, *78*, 941.
- Smith, R. L.; Popham, R. E. “The Quantitative Resolution of a Mixture of Group II Metal Ions by Thermometric Titration with EDTA,” *J. Chem. Educ.* **1983**, *60*, 1076–1077.
- Yappert, M. C.; DuPré, D. B. “Complexometric Titrations: Competition of Complexing Agents in the Determination of Water Hardness with EDTA,” *J. Chem. Educ.* **1997**, *74*, 1422–1423.

Redox Titrimetry

- Guenther, W. B. “Supertitrations: High-Precision Methods,” *J. Chem. Educ.* **1988**, *65*, 1097–1098.
- Haddad, P. “Vitamin C Content of Commercial Orange Juices,” *J. Chem. Educ.* **1977**, *54*, 192–193.
- Harris, D. C.; Hills, M. E.; Hewston, T. A. “Preparation, Iodometric Analysis and Classroom Demonstration of Superconductivity in $YBa_2Cu_3O_{8-x}$,” *J. Chem. Educ.* **1987**, *64*, 847–850.
- Lau, O.-W.; Luk, S.-F.; Cheng, N. L. N.; Woo, H.-O. “Determination of Free Lime in Clinker and Cement by Iodometry,” *J. Chem. Educ.* **2001**, *78*, 1671–1673.
- Phinyocheep, P.; Tang, I. M. “Determination of the Hole Concentration (Copper Valency) in the High Tc Superconductors,” *J. Chem. Educ.* **1994**, *71*, A115–A118.
- Powell, J. R.; Tucker, S. A.; Acree, Jr., W. E.; Sees, J. A.; Hall, L. M. “A Student-Designed Potentiometric Titration: Quantitative Determination of Iron(II) by Caro’s Acid Titration,” *J. Chem. Educ.* **1996**, *73*, 984–986.

Precipitation Titrimetry

- Ueno, K.; Kina, K. “Colloid Titration - A Rapid Method for the Determination of Charged Colloid,” *J. Chem. Educ.* **1985**, *62*, 627–629.

For a general history of titrimetry, see the following sources.

- *A History of Analytical Chemistry*; Laitinen, H. A.; Ewing, G. W., Eds.; The Division of Analytical Chemistry of the American Chemical Society: Washington, D. C., 1977, pp. 52–93.
- Kolthoff, I. M. “Analytical Chemistry in the USA in the First Quarter of This Century,” *Anal. Chem.* **1994**, *66*, 241A–249A.

The use of weight instead of volume as a signal for titrimetry is reviewed in the following paper.

- Kratochvil, B.; Maitra, C. “Weight Titrations: Past and Present,” *Am. Lab.* **1983**, January, 22–29.

A more thorough discussion of non-aqueous titrations, with numerous practical examples, is provided in the following text.

- Fritz, J. S. *Acid-Base Titrations in Nonaqueous Solvents*; Allyn and Bacon, Boston; 1973.

The sources listed below provides more details on the how potentiometric titration data may be used to calculate equilibrium constants.

- Babić, S.; Horvat, A. J. M.; Pavlović, D. M.; Kaštelan-Macan, M. “Determination of pKa values of active pharmaceutical ingredients,” *Trends Anal. Chem.* **2007**, *26*, 1043–1061.
- Meloun, M.; Havel, J.; Högfeldt, E. *Computation of Solution Equilibria*, Ellis Horwood Limited: Chichester, England; 1988.

The following provides additional information about Gran plots.

- Michalowski, T.; Kupiec, K.; Rymanowski, M. *Anal. Chim. Acta* **2008**, *606*, 172–183.
- Schwartz, L. M. “Advances in Acid-Base Gran Plot Methodology,” *J. Chem. Educ.* **1987**, *64*, 947–950.
- Schwartz, L. M. “Uncertainty of a Titration Equivalence Point,” *J. Chem. Educ.* **1992**, *69*, 879–883.

The following provide additional information about calculating or sketching titration curves.

- Barnum, D. “Predicting Acid–Base Titration Curves without Calculations,” *J. Chem. Educ.* **1999**, *76*, 938–942.
- de Levie, R. “A simple expression for the redox titration curve,” *J. Electroanal. Chem.* **1992**, *323*, 347–355.

- King, D. W. “A General Approach for Calculating Speciation and Posing Capacity of Redox Systems with Multiple Oxidation States: Application to Redox Titrations and the Generation of pε–pH,” *J. Chem. Educ.* **2002**, *79*, 1135–1140.

For a complete discussion of the application of complexation titrimetry see the texts listed below.

- Pribil, R. *Applied Complexometry*, Pergamon Press: Oxford, 1982.
- Ringbom, A. *Complexation in Analytical Chemistry*, John Wiley and Sons, Inc.: New York, 1963.
- Schwarzenbach, G. *Complexometric Titrations*, Methuen & Co. Ltd: London, 1957.

A good source for additional examples of the application of all forms of titrimetry is

- *Vogel’s Textbook of Quantitative Inorganic Analysis*, Longman: London, 4th Ed., 1981.

Chapter 10

The following set of experiments introduce students to the applications of spectroscopy. Experiments are grouped into five categories: UV/Vis spectroscopy, IR spectroscopy, atomic absorption and atomic emission, fluorescence and phosphorescence, and signal averaging.

UV/Vis Spectroscopy

- Abney, J. R.; Scalettar, B. A. “Saving Your Students’ Skin. Undergraduate Experiments That Probe UV Protection by Sunscreens and Sunglasses,” *J. Chem. Educ.* **1998**, *75*, 757–760.
- Ainscough, E. W.; Brodie, A. M. “The Determination of Vanillin in Vanilla Extract,” *J. Chem. Educ.* **1990**, *67*, 1070–1071.
- Allen, H. C.; Brauers, T.; Finlayson-Pitts, B. J. “Illustrating Deviations in the Beer-Lambert Law in an Instrumental Analysis Laboratory: Measuring Atmospheric Pollutants by Differential Optical Absorption Spectrometry,” *J. Chem. Educ.* **1997**, *74*, 1459–1462.
- Blanco, M.; Iturriaga, H.; Maspoch, S.; Tarîn, P. “A Simple Method for Spectrophotometric Determination of Two-Components with Overlapped Spectra,” *J. Chem. Educ.* **1989**, *66*, 178–180.
- Bonicamp, J. M.; Martin, K. L.; McBride, G. R.; Clark, R. W. “Beer’s Law is Not a Straight Line: Amplification of Errors by Transformation,” *Chem. Educator* **1999**, *4*, 81–88.
- Bruneau, E.; Lavabre, D.; Levy, G.; Micheau, J. C. “Quantitative Analysis of Continuous-Variation Plots with a Comparison of Several Methods,” *J. Chem. Educ.* **1992**, *69*, 833–837.
- Cappas, C.; Hoffman, N.; Jones, J.; Young, S. “Determination of Concentrations of Species Whose Absorption Bands Overlap Extensively,” *J. Chem. Educ.* **1991**, *68*, 300–303.
- Crisp, P. T.; Eckert, J. M.; Gibson, N. A. “The Determination of Anionic Surfactants in Natural and Waste Waters,” *J. Chem. Educ.* **1983**, *60*, 236–238.
- Dilbeck, C. W.; Ganske, J. A. “Detection of NOx in Automobile Exhaust: An Applied Experiment in Atmospheric/Environmental Chemistry for the General Chemistry Laboratory,” *Chem. Educator* **2008**, *13*, 1–5.
- Domínguez, A., Fernández, A.; González, N.; Iglesias, E.; Montenegro, L. “Determination of Critical Micelle Concentration of Some Surfactants by Three Techniques,” *J. Chem. Educ.* **1997**, *74*, 1227–1231.
- Gilbert, D. D. “Determining Optimum Spectral Bandwidth,” *J. Chem. Educ.* **1991**, *68*, A278–A281.
- Han, J.; Story, T.; Han, G. “A Spectrophotometric Method for Quantitative Determination of Bromine Using Tris(2-carboxyethyl)phophine,” *J. Chem. Educ.* **1999**, *76*, 976–977.
- Higginbotham, C.; Pike, C. F.; Rice, J, K. “Spectroscopy in Sol-Gel Matricies,” *J. Chem. Educ.* **1998**, *75*, 461–464.
- Hill, Z. D.; MacCarthy, P. “Novel Approach to Job’s Method,” *J. Chem. Educ.* **1986**, *63*, 162–167.
- Ibañez, G. A.; Olivieri, A. C.; Escandar, G. M. “Determination of Equilibrium Constants of Metal Complexes from Spectrophotometric Measurements,” *J. Chem. Educ.* **1999**, *76*, 1277–1281.
- Long, J. R.; Drago, R. S. “The Rigorous Evaluation of Spectrophotometric Data to Obtain an Equilibrium Constant,” *J. Chem. Educ.* **1982**, *59*, 1037–1039.
- Lozano-Calero; D.; Martin-Palomeque, P. “Determination of Phosphorous in Cola Drinks,” *J. Chem. Educ.* **1996**, *73*, 1173–1174.
- Maloney, K. M.; Quiazon, E. M.; Indralingam, R. “Measurement of Iron in Egg Yolk: An Instrumental Analysis Measurement Using Biochemical Principles,” *J. Chem. Educ.* **2008**, *85*, 399–400.
- Mascotti, D. P.; Waner, M. J. “Complementary Spectroscopic Assays for Investigation Protein-Ligand Binding Activity: A Project for the Advanced Chemistry Laboratory,” *J. Chem. Educ.* **2010**, *87*, 735–738.

- McDevitt, V. L.; Rodriquez, A.; Williams, K. R. “Analysis of Soft Drinks: UV Spectrophotometry, Liquid Chromatography, and Capillary Electrophoresis,” *J. Chem. Educ.* **1998**, *75*, 625–629.
- Mehra, M. C.; Rioux, J. “An Analytical Chemistry Experiment in Simultaneous Spectrophotometric Determination of Fe(III) and Cu(II) with Hexacyanoruthenate(II) Reagent,” *J. Chem. Educ.* **1982**, *59*, 688–689.
- Mitchell-Koch, J. T.; Reid, K. R.; Meyerhoff, M. E. “Salicylate Detection by Complexation with Iron(III) and Optical Absorbance Spectroscopy,” *J. Chem. Educ.* **2008**, *85*, 1658–1659.
- Msimanga, H. Z.; Wiese, J. “Determination of Acetaminophen in Analgesics by the Standard Addition Method: A Quantitative Analytical Chemistry Laboratory,” *Chem. Educator* **2005**, *10*, 1–7.
- Örstan, A.; Wojcik, J. F. “Spectroscopic Determination of Protein-Ligand Binding Constants,” *J. Chem. Educ.* **1987**, *64*, 814–816.
- Pandey, S.; Powell, J. R.; McHale, M. E. R.; Acree Jr., W. E. “Quantitative Determination of Cr(III) and Co(II) Using a Spectroscopic H-Point Standard Addition,” *J. Chem. Educ.* **1997**, *74*, 848–850.
- Parody-Morreale, A.; Cámara-Artigas, A.; Sánchez-Ruiz, J. M. “Spectrophotometric Determination of the Binding Constants of Succinate and Chloride to Glutamic Oxalacetic Transaminase,” *J. Chem. Educ.* **1990**, *67*, 988–990.
- Ravelo-Perez, L. M.; Hernández-Borges, J.; Rodríguez-Delgado, M. A.; Borges-Miquel, T. “Spectrophotometric Analysis of Lycopene in Tomatoes and Watermelons: A Practical Class,” *Chem. Educator* **2008**, *13*, 1–3.
- Russell, D. D.; Potts, J.; Russell, R. M.; Olson, C.; Schimpf, M. “Spectroscopic and Potentiometric Investigation of a Diprotic Acid: An Experimental Approach to Understanding Alpha Functions,” *Chem. Educator* **1999**, *4*, 68–72.
- Smith, E. T.; Matachek, J. R. “A Colorful Investigation of a Diprotic Acid: A General Chemistry Laboratory Exercise,” *Chem. Educator* **2002**, *7*, 359–363
- Tello-Solis, S. R. “Thermal Unfolding of Lysozyme Studied by UV Difference Spectroscopy,” *Chem. Educator* **2008**, *13*, 16–18.
- Tucker, S.; Robinson, R.; Keane, C.; Boff, M.; Zenko, M.; Batish, S.; Street, Jr., K. W. “Colorimetric Determination of pH,” *J. Chem. Educ.* **1989**, *66*, 769–771.
- Vitt, J. E. “Troubleshooting 101: An Instrumental Analysis Experiment,” *J. Chem. Educ.* **2008**, *85*, 1660–1662.
- Williams, K. R.; Cole, S. R.; Boyette, S. E.; Schulman, S. G. “The Use of Dristan Nasal Spray as the Unknown for Simultaneous Spectrophotometric Analysis of a Mixture,” *J. Chem. Educ.* **1990**, *67*, 535.
- Walmsley, F. “Aggregation in Dyes: A Spectrophotometric Study,” *J. Chem. Educ.* **1992**, *69*, 583.
- Wells, T. A. “Construction of a Simple Myoglobin-Based Optical Biosensor,” *Chem. Educator* **2007**, *12*, 1–3.
- Yarnelle, M. K.; West, K. J. “Modification of an Ultraviolet Spectrophotometric Determination of the • Active Ingredients in APC Tablets,” *J. Chem. Educ.* **1989**, *66*, 601–602.

IR Spectroscopy

- Dragon, S.; Fitch, A. “Infrared Spectroscopy Determination of Lead Binding to Ethylenediaminetetraacetic Acid,” *J. Chem. Educ.* **1998**, *75*, 1018–1021.
- Frohlich, H. “Using Infrared Spectroscopy Measurements to Study Intermolecular Hydrogen Bonding,” *J. Chem. Educ.* **1993**, *70*, A3–A6.
- Garizi, N.; Macias, A.; Furch, T.; Fan, R.; Wagenknecht, P.; Singmaster, K. A. “Cigarette Smoke Analysis Using an Inexpensive Gas-Phase IR Cell,” *J. Chem. Educ.* **2001**, *78*, 1665–1666.
- Indralingam, R.; Nepomuceno, A. I. “The Use of Disposable IR Cards for Quantitative Analysis Using an Internal Standard,” *J. Chem. Educ.* **2001**, *78*, 958–960.
- Mathias, L. J.; Hankins, M. G.; Bertolucci, C. M.; Grubb, T. L.; Muthiah, J. “Quantitative Analysis by FTIR: Thin Films of Copolymers of Ethylene and Vinyl Acetate,” *J. Chem. Educ.* **1992**, *69*, A217–A219.
- Schuttlefield, J. D.; Grassian, V. H. “ATR-FTIR Spectroscopy in the Undergraduate Chemistry Laboratory. Part I: Fundamentals and Examples,” *J. Chem. Educ.* **2008**, *85*, 279–281.
- Schuttlefield, J. D.; Larsen, S. C.; Grassian, V. H. “ATR-FTIR Spectroscopy in the Undergraduate Chemistry Laboratory. Part II: A Physical Chemistry Laboratory Experiment on Surface Adsorption,” *J. Chem. Educ.* **2008**, *85*, 282–284.
- Seasholtz, M. B.; Pence, L. E.; Moe Jr., O. A. “Determination of Carbon Monoxide in Automobile Exhaust by FTIR Spectroscopy,” *J. Chem. Educ.* **1988**, *65*, 820–823.

Atomic Absorption and Atomic Emission Spectroscopy

- Amarasiriwardena, D. “Teaching analytical atomic spectroscopy advances in an environmental chemistry class using a project-based laboratory approach: investigation of lead and arsenic distributions in a lead arsenate contaminated apple orchard,” *Anal.*

Bioanal. Chem. **2007**, *388*, 307–314.

- Buffen, B. P. “Removal of Heavy Metals from Water: An Environmentally Significant Atomic Absorption Spectrometry Experiment,” *J. Chem. Educ.* **1999**, *76*, 1678–1679.
- Dockery, C. R.; Blew, M. J.; Goode, S. R. “Visualizing the Solute Vaporization Interference in Flame Atomic Absorption Spectroscopy,” *J. Chem. Educ.* **2008**, *85*, 854–858.
- Donas, M. K.; Whissel, G.; Dumas, A.; Golden, K. “Analyzing Lead Content in Ancient Bronze Coins by Flame Atomic Absorption Spectroscopy,” *J. Chem. Educ.* **2009**, *86*, 343–346.
- Gilles de Pelichy, L. D.; Adams, C.; Smith, E. T. “Analysis of the Essential Nutrient Strontium in Marine Aquariums by Atomic Absorption Spectroscopy,” *J. Chem. Educ.* **1997**, *74*, 1192–1194.
- Hoskins, L. C.; Reichardt, P. B.; Stolzberg, R. J. “Determination of the Extraction Constant for Zinc Pyrrolidinecarbodithioate,” *J. Chem. Educ.* **1981**, *58*, 580–581.
- Kooser, A. S.; Jenkins, J. L.; Welch, L. E. “Inductively Coupled Plasma-Atomic Emission Spectroscopy: Two Laboratory Activities for the Undergraduate Instrumental Analysis Course,” *J. Chem. Educ.* **2003**, *80*, 86–88.
- Kostecka, K. S. “Atomic Absorption Spectroscopy of Calcium in Foodstuffs in Non-Science-Major Courses,” *J. Chem. Educ.* **2000**, *77*, 1321–1323.
- Lehman, T. A.; Everett, W. W. “Solubility of Lead Sulfate in Water and in Sodium Sulfate Solutions,” *J. Chem. Educ.* **1982**, *59*, 797.
- Markow, P. G. “Determining the Lead Content of Paint Chips,” *J. Chem. Educ.* **1996**, *73*, 178–179.
- Masina, M. R.; Nkosi, P. A.; Rasmussen, P. W.; Shelembe, J. S.; Tyobeka, T. E. “Determination of Metal Ions in Pineapple Juice and Effluent of a Fruit Canning Industry,” *J. Chem. Educ.* **1989**, *66*, 342–343.
- Quigley, M. N. “Determination of Calcium in Analgesic Tablets using Atomic Absorption Spectrophotometry,” *J. Chem. Educ.* **1994**, *71*, 800.
- Quigley, M. N.; Vernon, F. “Determination of Trace Metal Ion Concentrations in Seawater,” *J. Chem. Educ.* **1996**, *73*, 671–675.
- Quigley, M. N.; Vernon, F. “A Matrix Modification Experiment for Use in Electrothermal Atomic Absorption Spectrophotometry,” *J. Chem. Educ.* **1996**, *73*, 980–981.
- Rheingold, A. L.; Hues, S.; Cohen, M. N. “Strontium and Zinc Content in Bones as an Indication of Diet,” *J. Chem. Educ.* **1983**, *60*, 233–234.
- Rocha, F. R. P.; Nóbrega, J. A. “Effects of Solution Physical Properties on Copper and Chromium Signals in Flame Atomic Absorption Spectrometry,” *J. Chem. Educ.* **1996**, *73*, 982–984.

Fluorescence and Phosphorescence Spectroscopy

- Buccigross, J. M.; Bedell, C. M.; Suding-Moster, H. L. “Fluorescent Measurement of TNS Binding to Calmodulin,” *J. Chem. Educ.* **1996**, *73*, 275–278.
- Henderleiter, J. A.; Hyslopo, R. M. “The Analysis of Riboflavin in Urine by Fluorescence,” *J. Chem. Educ.* **1996**, *73*, 563–564.
- Lagoria, M. G.; Román, E. S. “How Does Light Scattering Affect Luminescence? Fluorescence Spectra and Quantum Yields in the Solid Form,” *J. Chem. Educ.* **2002**, *79*, 1362–1367.
- Richardson, D. P.; Chang, R. “Lecture Demonstrations of Fluorescence and Phosphorescence,” *Chem. Educator* **2007**, *12*, 272–274.
- Seixas de Melo, J. S.; Cabral, C.; Burrows, H. D. “Photochemistry and Photophysics in the Laboratory. Showing the Role of Radiationless and Radiative Decay of Excited States,” *Chem. Educator* **2007**, *12*, 1–6.
- Sheffield, M. C.; Nahir, T. M. “Analysis of Selenium in Brazil Nuts by Microwave Digestion and Fluorescence Detection,” *J. Chem. Educ.* **2002**, *79*, 1345–1347.

Signal Averaging

- Blitz, J. P.; Klarup, D. G. “Signal-to-Noise Ratio, Signal Processing, and Spectral Information in the Instrumental Analysis Laboratory,” *J. Chem. Educ.* **2002**, *79*, 1358–1360.
- Stolzberg, R. J. “Introduction to Signals and Noise in an Instrumental Method Course,” *J. Chem. Educ.* **1983**, *60*, 171–172.
- Tardy, D. C. “Signal Averaging. A Signal-to-Noise Enhancement Experiment for the Advanced Chemistry Laboratory,” *J. Chem. Educ.* **1986**, *63*, 648–650.

The following sources provide additional information on spectroscopy in the following areas: general spectroscopy, Beer’s law, instrumentation, Fourier transforms, , IR spectroscopy, atomic absorption and emission, luminescence, and applications.

General Spectroscopy

- Ball, D. W. “Units! Units! Units!” *Spectroscopy* **1995**, *10(8)*, 44–47.
- *A History of Analytical Chemistry*, Laitinen, H. A.; Ewing, G. W, Eds. The Division of Analytical Chemistry of the American Chemical Society: Washington, D. C., 1977, p103–243.
- Ingle, J. D.; Crouch, S. R. *Spectrochemical Analysis*, Prentice Hall, Englewood Cliffs, N. J.; 1988.
- Macomber, R. S. “A Unifying Approach to Absorption Spectroscopy at the Undergraduate Level,” *J. Chem. Educ.* **1997**, *74*, 65–67.
- Orchin, M.; Jaffe, H. H. *Symmetry, Orbitals and Spectra*, Wiley-Interscience: New York, 1971.
- Thomas, N. C. “The Early History of Spectroscopy,” *J. Chem. Educ.* **1991**, *68*, 631–633.

Beer’s Law

- Lykos, P. “The Beer-Lambert Law Revisited: A Development without Calculus,” *J. Chem. Educ.* **1992**, *69*, 730–732.
- Ricci, R. W.; Ditzler, M. A.; Nestor, L. P. “Discovering the Beer-Lambert Law,” *J. Chem. Educ.* **1994**, *71*, 983–985.

Instrumentation

- Altermose, I. R. “Evolution of Instrumentation for UV-Visible Spectrophotometry: Part I,” *J. Chem. Educ.* **1986**, *63*, A216–A223.
- Altermose, I. R. “Evolution of Instrumentation for UV-Visible Spectrophotometry: Part II,” *J. Chem. Educ.* **1986**, *63*, A262–A266.
- Grossman, W. E. L. “The Optical Characteristics and Production of Diffraction Gratings,” *J. Chem. Educ.* **1993**, *70*, 741–748.
- Jones, D. G. “Photodiode Array Detectors in UV-Vis Spectroscopy: Part I,” *Anal. Chem.* **1985**, *57*, 1057A–1073A.
- Jones, D. G. “Photodiode Array Detectors in UV-Vis Spectroscopy: Part II,” *Anal. Chem.* **1985**, 11, 1207A–1214A.
- Palmer, C. “Diffraction Gratings,” *Spectroscopy*, **1995**, *10(2)*, 14–15.

Fourier Transforms

- Bracewell, R. N. “The Fourier Transform,” *Sci. American* **1989**, *260(6)*, 85–95.
- Glasser, L. “Fourier Transforms for Chemists: Part I. Introduction to the Fourier Transform,” *J. Chem. Educ.* **1987**, *64*, A228–A233.
- Glasser, L. “Fourier Transforms for Chemists: Part II. Fourier Transforms in Chemistry and Spectroscopy,” *J. Chem. Educ.* **1987**, *64*, A260–A266.
- Glasser, L. “Fourier Transforms for Chemists: Part III. Fourier Transforms in Data Treatment,” *J. Chem. Educ.* **1987**, *64*, A306–A313.
- Graff, D. K. “Fourier and Hadamard: Transforms in Spectroscopy,” *J. Chem. Educ.* **1995**, *72*, 304–309.
- Griffiths, P. R. *Chemical Fourier Transform Spectroscopy*, Wiley-Interscience: New York, 1975.
- *Transform Techniques in Chemistry*, Griffiths, P. R. Ed., Plenum Press: New York, 1978.
- Perkins, W. E. “Fourier Transform Infrared Spectroscopy: Part I. Instrumentation,” *J. Chem. Educ.* **1986**, *63*, A5–A10.
- Perkins, W. E. “Fourier Transform Infrared Spectroscopy: Part II. Advantages of FT-IR,” *J. Chem. Educ.* **1987**, *64*, A269–A271.
- Perkins, W. E. “Fourier Transform Infrared Spectroscopy: Part III. Applications,” *J. Chem. Educ.* **1987**, *64*, A296–A305.
- Strong III, F. C. “How the Fourier Transform Infrared Spectrophotometer Works,” *J. Chem. Educ.* **1979**, *56*, 681–684.

IR Spectroscopy

- *Optical Spectroscopy: Sampling Techniques Manual*, Harrick Scientific Corporation: Ossining, N. Y., 1987.
- Leyden, D. E.; Shreedhara Murthy, R. S. “Surface-Selective Sampling Techniques in Fourier Transform Infrared Spectroscopy,” *Spectroscopy* **1987**, *2(2)*, 28–36.
- Porro, T. J.; Pattacini, S. C. “Sample Handling for Mid-Infrared Spectroscopy, Part I: Solid and Liquid Sampling,” *Spectroscopy* **1993**, *8(7)*, 40–47.
- Porro, T. J.; Pattacini, S. C. “Sample Handling for Mid-Infrared Spectroscopy, Part II: Specialized Techniques,” *Spectroscopy* **1993**, *8(8)*, 39–44.

Atomic Absorption and Emission

- Blades, M. W.; Weir, D. G. “Fundamental Studies of the Inductively Coupled Plasma,” *Spectroscopy* **1994**, *9*, 14–21.
- Greenfield, S. “Invention of the Annular Inductively Coupled Plasma as a Spectroscopic Source,” *J. Chem. Educ.* **2000**, *77*, 584–591.
- Hieftje, G. M. “Atomic Absorption Spectrometry - Has it Gone or Where is it Going?” *J. Anal. At. Spectrom.* **1989**, *4*, 117–122.

- Jarrell, R. F. “A Brief History of Atomic Emission Spectrochemical Analysis, 1666-1950,” *J. Chem. Educ.* **2000**, *77*, 573–576
- Koirtyohann, S. R. “A History of Atomic Absorption Spectrometry From an Academic Perspective,” *Anal. Chem.* **1991**, *63*, 1024A–1031A.
- L’Vov, B. V. “Graphite Furnace Atomic Absorption Spectrometry,” *Anal. Chem.* **1991**, *63*, 924A–931A.
- Slavin, W. “A Comparison of Atomic Spectroscopic Analytical Techniques,” *Spectroscopy*, **1991**, *6*, 16–21.
- Van Loon, J. C. *Analytical Atomic Absorption Spectroscopy*, Academic Press: New York, 1980.
- Walsh, A. “The Development of Atomic Absorption Methods of Elemental Analysis 1952-1962,” *Anal. Chem.* **1991**, *63*, 933A–941A.
- Welz, B. *Atomic Absorption Spectroscopy*, VCH: Deerfield Beach, FL, 1985.

Luminescence Spectroscopy

- Guilbault, G. G. *Practical Fluorescence*, Decker: New York, 1990.
- Schenk, G. “Historical Overview of Fluorescence Analysis to 1980,” *Spectroscopy* **1997**, *12*, 47–56.
- Vo-Dinh, T. *Room-Temperature Phosphorimetry for Chemical Analysis*, Wiley-Interscience: New York, 1984.
- Winefordner, J. D.; Schulman, S. G.; O’Haver, T. C. *Luminescence Spectroscopy in Analytical Chemistry*, Wiley-Interscience: New York, 1969.

Applications

- *Trace Analysis and Spectroscopic Methods for Molecules*, Christian, G. D.; Callis, J. B. Eds., Wiley-Interscience: New York, 1986.
- Vandecasteele, C.; Block, C. B. *Modern Methods for Trace Element Determination*, Wiley: Chichester, England, 1994.
- Skoog, D. A.; Holler, F. J.; Nieman, T. A. *Principles of Instrumental Analysis*, Saunders: Philadelphia, 1998.
- Van Loon, J. C. *Selected Methods of Trace Metal Analysis: Biological and Environmental Samples*, Wiley-Interscience: New York, 1985.

Gathered here are resources and experiments for analyzing multicomponent samples using mathematical techniques not covered in this textbook.

- Aberasturi, F.; Jimenez, A. I.; Jimenez, F.; Arias, J. J. “UV-Visible First-Derivative Spectrophotometry Applied to an Analysis of a Vitamin Mixture,” *J. Chem. Educ.* **2001**, *78*, 793–795.
- Afkhami, A.; Abbasi-Tarighat, M.; Bahram, M.; Abdollahi, H. “A new strategy for solving matrix effect in multivariate calibration standard addition data using combination of H-point curve isolation and H-point standard addition methods,” *Anal. Chim. Acta* **2008**, *613*, 144–151.
- Brown, C. W.; Obremski, R. J. “Multicomponent Quantitative Analysis,” *Appl. Spectrosc. Rev.* **1984**, *20*, 373–418.
- Charles, M. J.; Martin, N. W.; Msimanga, H. Z. “Simultaneous Determination of Aspirin, Salicylamide, and Caffeine in Pain Relievers by Target Factor Analysis,” *J. Chem. Educ.* **1997**, *74*, 1114–1117.
- Dado, G.; Rosenthal, J. “Simultaneous Determination of Cobalt, Copper, and Nickel by Multivariate Linear Regression,” *J. Chem. Educ.* **1990**, *67*, 797–800.
- DiTusa, M. R.; Schilt, A. A. “Selection of Wavelengths for Optimum Precision in Simultaneous Spectrophotometric Determinations,” *J. Chem. Educ.* **1985**, *62*, 541–542.
- Gómez, D. G.; de la Peña, A. M.; Mansilla, A. E.; Olivieri, A. C. “Spectrophotometric Analysis of Mixtures by Classical Least-Squares Calibration: An Advanced Experiment Introducing MATLAB,” *Chem. Educator* **2003**, *8*, 187–191.
- Harvey, D. T.; Bowman, A. “Factor Analysis of Multicomponent Samples,” *J. Chem. Educ.* **1990**, *67*, 470–472.
- Lucio-Gutierrez, J. R.; Salazar-Cavazos, M. L.; de Torres, N. W. “Chemometrics in the Teaching Lab. Quantification of a Ternary Mixture of Common Pharmaceuticals by First- and Second-Derivative IR Spectroscopy,” *Chem. Educator* **2004**, *9*, 234–238.
- Padney, S.; McHale, M. E. R.; Coym, K. S.; Acree Jr., W. E. “Bilinear Regression Analysis as a Means to Reduce Matrix Effects in Simultaneous Spectrophotometric Determination of Cr(III) and Co(II),” *J. Chem. Educ.* **1998**, *75*, 878–880.
- Raymond, M.; Jochum, C.; Kowalski, B. R. “Optimal Multicomponent Analysis Using the Generalized Standard Addition Method,” *J. Chem. Educ.* **1983**, *60*, 1072–1073.
- Ribone, M. E.; Pagani, A. P.; Olivieri, A. C.; Goicoechea, H. C. “Determination of the Active Principle in a Spectrophotometry and Principal Component Regression Analysis,” *J. Chem. Educ.* **2000**, *77*, 1330–1333.
- Rojas, F. S.; Ojeda, C. B. “Recent developments in derivative ultraviolet/visible absorption spectrophotometry: 2004-2008,” *Anal. Chim. Acta* **2009**, *635*, 22–44.

Chapter 11

The following set of experiments introduce students to the applications of electrochemistry. Experiments are grouped into four categories: general electrochemistry, preparation of electrodes, potentiometry, coulometry, and voltammetry and amperometry.

General Electrochemistry

- Mills, K. V.; Herrick, R. S.; Guilmette, L. W.; Nestor, L. P.; Shafer, H.; Ditzler, M. A. "Introducing Undergraduate Students to Electrochemistry: A Two-Week Discovery Chemistry Experiment," J. Chem. Educ. 2008, 85, 1116–119.

Preparation of Electrodes

- Christopoulos, T. K.; Diamandis, E. P. "Use of a Sintered Glass Crucible for Easy Construction of Liquid-Membrane Ion-Selective Electrodes," *J. Chem. Educ.* **1988**, *65*, 648.
- Fricke, G. H.; Kuntz, M. J. "Inexpensive Solid-State Ion-Selective Electrodes for Student Use," *J. Chem. Educ.* **1977**, *54*, 517–520.
- Inamdar, S. N.; Bhat, M. A.; Haram, S. K. "Construction of Ag/AgCl Reference Electrode form Used Felt-Tipped Pen Barrel for Undergraduate Laboratory," *J. Chem. Educ.* **2009**, *86*, 355–356.
- Lloyd, B. W.; O'Brien, F. L.; Wilson, W. D. "Student Preparation and Analysis of Chloride and Calcium Ion Selective Electrodes," *J. Chem. Educ.* **1976**, *53*, 328–330.
- Mifflin, T. E.; Andriano, K. M.; Robbins, W. B. "Determination of Penicillin Using an Immobilized Enzyme Electrode," *J. Chem. Educ.* **1984**, *61*, 638–639.
- Palanivel, A.; Riyazuddin, P. "Fabrication of an Inexpensive Ion-Selective Electrode," *J. Chem. Educ.* **1984**, *61*, 290.
- Ramaley, L; Wedge, P. J.; Crain, S. M. "Inexpensive Instrumental Analysis: Part 1. Ion-Selective Electrodes," *J. Chem. Educ.* **1994**, *71*, 164–167.
- Selig, W. S. "Potentiometric Titrations Using Pencil and Graphite Sensors," *J. Chem. Educ.* **1984**, *61*, 80–81.

Potentiometry

- Chan, W. H; Wong, M. S.; Yip, C. W. "Ion-Selective Electrode in Organic Analysis: A Salicylate Electrode," *J. Chem. Educ.* **1986**, *63*, 915–916.
- Harris, T. M. "Potentiometric Measurement in a Freshwater Aquarium," *J. Chem. Educ.* **1993**, *70*, 340–341.
- Kauffman, C. A.; Muza, A. L.; Porambo, M. W.; Marsh, A. L. "Use of a Commercial Silver-Silver Chloride Electrode for the Measurement of Cell Potentials to Determine Mean Ionic Activity Coefficients," *Chem. Educator* **2010**, *15*, 178–180.
- Martínez-Fàbregas, E.; Alegret, S. "A Practical Approach to Chemical Sensors through Potentiometric Transducers: Determination of Urea in Serum by Means of a Biosensor," *J. Chem. Educ.* **1994**, *71*, A67–A70.
- Moresco, H.; Sansón, P.; Seoane, G. "Simple Potentiometric Determination of Reducing Sugars," *J. Chem. Educ.* **2008**, *85*, 1091–1093.
- Radic, N.; Komijenovic, J. "Potentiometric Determination of an Overall Formation Constant Using an Ion-Selective Membrane Electrode," *J. Chem. Educ.* **1993**, *70*, 509–511.
- Riyazuddin, P.; Devika, D. "Potentiometric Acid–Base Titrations with Activated Graphite Electrodes," *J. Chem. Educ.* **1997**, *74*, 1198–1199.

Coulometry

- Bertotti, M.; Vaz, J. M.; Telles, R. "Ascorbic Acid Determination in Natural Orange Juice," *J. Chem. Educ.* **1995**, *72*, 445–447.
- Kalbus, G. E.; Lieu, V. T. "Dietary Fat and Health: An Experiment on the Determination of Iodine Number of Fats and Oils by Coulometric Titration," *J. Chem. Educ.* **1991**, *68*, 64–65.
- Lötz, A. "A Variety of Electrochemical Methods in a Coulometric Titration Experiment," *J. Chem. Educ.* **1998**, *75*, 775–777.
- Swim, J.; Earps, E.; Reed, L. M.; Paul, D. "Constant-Current Coulometric Titration of Hydrochloric Acid," *J. Chem. Educ.* **1996**, *73*, 679–683.

Voltammetry and Amperometry

- Blanco-López, M. C.; Lobo-Castañón, M. J.; Miranda-Ordieres, A. J. "Homemade Bienzymatic-Amperometric Biosensor for Beverages Analysis," *J. Chem. Educ.* **2007**, *84*, 677–680.
- García-Armada, P.; Losada, J.; de Vicente-Pérez, S. "Cation Analysis Scheme by Differential Pulse Polarography," *J. Chem. Educ.* **1996**, *73*, 544–547.

- Herrera-Melián, J. A.; Doña-Rodríguez, J. M.; Hernández-Brito, J.; Pérez-Peña, J. “Voltammetric Determination of Ni and Co in Water Samples,” *J. Chem. Educ.* **1997**, *74*, 1444–1445.
- King, D.; Friend, J.; Kariuki, J. “Measuring Vitamin C Content of Commercial Orange Juice Using a Pencil Lead Electrode,” *J. Chem. Educ.* **2010**, *87*, 507–509.
- Marin, D.; Mendicuti, F. “Polarographic Determination of Composition and Thermodynamic Stability Constant of a Complex Metal Ion,” *J. Chem. Educ.* **1988**, *65*, 916–918.
- Sadik, O. A.; Brenda, S.; Joasil, P.; Lord, J. “Electropolymerized Conducting Polymers as Glucose Sensors,” *J. Chem. Educ.* **1999**, *76*, 967–970.
- Sittampalam, G.; Wilson, G. S. “Amperometric Determination of Glucose at Parts Per Million Levels with Immobilized Glucose Oxidase,” *J. Chem. Educ.* **1982**, *59*, 70–73.
- Town, J. L.; MacLaren, F.; Dewald, H. D. “Rotating Disk Voltammetry Experiment,” *J. Chem. Educ.* **1991**, *68*, 352–354.
- Wang, J. “Sensitive Electroanalysis Using Solid Electrodes,” *J. Chem. Educ.* **1982**, *59*, 691–692.
- Wang, J. “Anodic Stripping Voltammetry,” *J. Chem. Educ.* **1983**, *60*, 1074–1075.
- Wang, J.; Maccà, C. “Use of Blood-Glucose Test Strips for Introducing Enzyme Electrodes and Modern Biosensors,” *J. Chem. Educ.* **1996**, *73*, 797–800.
- Wang, Q.; Geiger, A.; Frias, R; Golden, T. D. “An Introduction to Electrochemistry for Undergraduates: Detection of Vitamin C (Ascorbic Acid) by Inexpensive Electrode Sensors,” *Chem. Educator* **2000**, *5*, 58–60.

The following general references providing a broad introduction to electrochemistry.

- Adams, R. N. *Electrochemistry at Solid Surfaces*, Marcel Dekker: New York, 1969.
- Bard, A. J.; Faulkner, L. R. *Electrochemical Methods*, Wiley: New York, 1980.
- Faulkner, L. R. “Electrochemical Characterization of Chemical Systems” in Kuwana, T. E., ed. *Physical Methods in Modern Chemical Analysis*, Vol. 3, Academic Press: New York, 1983, pp. 137–248.
- Kissinger, P. T.; Heineman, W. R. *Laboratory Techniques in Electroanalytical Chemistry*, Marcel Dekker: New York, 1984.
- Lingane, J. J. *Electroanalytical Chemistry*, 2nd Ed., Interscience: New York, 1958.
- Sawyer, D. T.; Roberts, J. L., Jr. *Experimental Electrochemistry for Chemists*, Wiley-Interscience: New York, 1974.
- Vassos, B. H.; Ewing, G. W. *Electroanalytical Chemistry*, Wiley-Interscience: New York, 1983.

These short articles provide a good introduction to important principles of electrochemistry.

- Faulkner, L. R. “Understanding Electrochemistry: Some Distinctive Concepts,” *J. Chem. Educ.* **1983**, *60*, 262–264.
- Huddle, P. A.; White, M. D.; Rogers, F. “Using a Teaching Model to Correct Known Misconceptions in Electrochemistry,” *J. Chem. Educ.* **2000**, *77*, 104–110.
- Maloy, J. T. “Factors Affecting the Shape of Current-Potential Curves,” *J. Chem. Educ.* **1983**, *60*, 285–289.
- Thompson, R. Q.; Craig, N. C. “Unified Electroanalytical Chemistry: Application of the Concept of Equilibrium,” *J. Chem. Educ.* **2001**, *78*, 928–934.
- Zoski, C. G. “Charging Current Discrimination in Analytical Voltammetry,” *J. Chem. Educ.* **1986**, *63*, 910–914.

Additional information on potentiometry and ion-selective electrodes can be found in the following sources.

- Bakker, E.; Diamond, D.; Lewenstam, A.; Pretsch, E. “Ions Sensors: Current Limits and New Trends,” *Anal. Chim. Acta* **1999**, *393*, 11–18.
- Bates, R. G. *Determination of pH: Theory and Practice*, 2nd ed., Wiley: New York, 1973.
- Buck, R. P. “Potentiometry: pH Measurements and Ion Selective Electrodes” in Weissberger, A., ed. *Physical Methods of Organic Chemistry*, Vol. 1, Part IIA, Wiley: New York, 1971, pp. 61–162.
- Cammann, K. *Working With Ion-Selective Electrodes*, Springer-Verlag: Berlin, 1977.
- Evans, A. *Potentiometry and Ion-Selective Electrodes*, Wiley: New York, 1987.
- Frant, M. S. “Where Did Ion Selective Electrodes Come From?” *J. Chem. Educ.* **1997**, *74*, 159–166.
- Light, T. S. “Industrial Use and Application of Ion-Selective Electrodes,” *J. Chem. Educ.* **1997**, *74*, 171–177.
- Rechnitz, G. A. “Ion and Bio-Selective Membrane Electrodes,” *J. Chem. Educ.* **1983**, *60*, 282–284.
- Ruzicka, J. “The Seventies—Golden Age for Ion-Selective Electrodes,” *J. Chem. Educ.* **1997**, *74*, 167–170.
- Young, C. C. “Evolution of Blood Chemistry Analyzers Based on Ion Selective Electrodes,” *J. Chem. Educ.* **1997**, *74*, 177–182.

The following sources provide additional information on electrochemical biosensors.

- Alvarez-Icasa, M.; Bilitewski, U. “Mass Production of Biosensors,” *Anal. Chem.* **1993**, *65*, 525A–533A.

- Meyerhoff, M. E.; Fu, B.; Bakker, E. Yun, J-H; Yang, V. C. “Polyion-Sensititve Membrane Electrodes for Biomedical Analysis,” *Anal. Chem.* **1996**, *68*, 168A–175A.
- Nicolini, C.; Adami, M; Antolini, F.; Beltram, F.; Sartore, M.; Vakula, S. “Biosensors: A Step to Bioelectronics,” *Phys. World*, May 1992, 30–34.
- Rogers, K. R.; Williams. L. R. “Biosensors for Environmental Monitoring: A Regulatory Perspective,” *Trends Anal. Chem.* **1995**, *14*, 289–294.
- Schultz, J. S. “Biosensors,” *Sci. Am.* August 1991, 64–69.
- Thompson, M.; Krull, U. “Biosensors and the Transduction of Molecular Recognition,” *Anal. Chem.* **1991**, *63*, 393A–405A.
- Vadgama, P. “Designing Biosensors,” *Chem. Brit.* **1992**, *28*, 249–252.

A good source covering the clinical application of electrochemistry is listed below.

- Wang, J. *Electroanalytical Techniques in Clinical Chemistry and Laboratory Medicine*, VCH: New York, 1998.

Coulometry is covered in the following texts.

- Rechnitz, G. A. *Controlled-Potential Analysis*, Macmillan: New York, 1963.
- Milner, G. W. C.; Philips, G. *Coulometry in Analytical Chemistry*, Pergamon: New York, 1967.

For a description of electrogravimetry, see the following resource.

- Tanaka, N. “Electrodeposition”, in Kolthoff, I. M.; Elving, P. J., eds. *Treatise on Analytical Chemistry, Part I: Theory and Practice*, Vol. 4, Interscience: New York, 1963.

The following sources provide additional information on polarography and pulse polarography.

- Flato, J. B. “The Renaissance in Polarographic and Voltammetric Analysis,” *Anal. Chem.* **1972**, *44(11)*, 75A–87A.
- Kolthoff, I. M.; Lingane, J. J. *Polarography*, Interscience: New York, 1952.
- Osteryoung, J. “Pulse Voltammetry,” *J. Chem. Educ.* **1983**, *60*, 296–298.

Additional Information on stripping voltammetry is available in the following text.

- Wang, J. *Stripping Analysis*, VCH Publishers: Deerfield Beach, FL, 1985.

The following papers discuss the numerical simulation of voltammetry.

- Bozzini, B. “A Simple Numerical Procedure for the Simulation of “Lifelike” Linear-Sweep Voltammograms,” *J. Chem. Educ.* **2000**, *77*, 100–103.
- Howard, E.; Cassidy, J. “Analysis with Microelectrodes Using Microsoft Excel Solver,” *J. Chem. Educ.* **2000**, *77*, 409–411.

Gathered together here are many useful resources for cyclic voltammetry, including experiments.

- Carriedo, G. A. “The Use of Cyclic Voltammetry in the Study of the Chemistry of Metal–Carbonyls,” *J. Chem. Educ.* **1988**, *65*, 1020–1022.
- García-Jareño, J. J.; Benito, D.; Navarro-Laboulais, J.; Vicente, F. “Electrochemical Behavior of Electrodeposited Prussian Blue Films on ITO Electrodes,” *J. Chem. Educ.* **1998**, *75*, 881–884.
- Gilles de Pelichy, L. D.; Smith, E. T. “A Study of the Oxidation Pathway of Adrenaline by Cyclic Voltammetry,” *Chem. Educator* **1997**, *2(2)*, 1–13.
- Gomez, M. E.; Kaifer, A. E. “Voltammetric Behavior of a Ferrocene Derivative,” *J. Chem. Educ.* **1992**, *69*, 502–505.
- Heffner, J. E.; Raber, J. C.; Moe, O. A.; Wigal, C. T. “Using Cyclic Voltammetry and Molecular Modeling to Determine Substituent Effects in the One-Electron Reduction of Benzoquinones,” *J. Chem. Educ.* **1998**, *75*, 365–367.
- Heinze, J. “Cyclic Voltammetry—Electrochemical Spectroscopy,” *Angew. Chem, Int. Ed. Eng.* **1984**, *23*, 831–918.
- Holder, G. N.; Farrar, D. G.; McClure, L. L. “Voltammetric Reductions of Ring-Substituted Acetophenones. 1. Determination of an Electron-Transfer Mechanism Using Cyclic Voltammetry and Computer Modeling: The Formation and Fate of a Radical Anion,” *Chem. Educator* **2001**, *6*, 343–349.
- Ibanez, J. G.; Gonzalez, I.; Cardenas, M. A. “The Effect of Complex Formation Upon the Redox Potentials of Metal Ions: Cyclic Voltammetry Experiments,” *J. Chem. Educ.* **1988**, *65*, 173–175.
- Ito, T.; Perara, D. M. N. T.; Nagasaka, S. “Gold Electrodes Modified with Self-Assembled Monolayers for Measuring L-Ascobric acid,” *J. Chem. Educ.* **2008**, *85*, 1112–1115.

- Kissinger, P. T.; Heineman, W. R. “Cyclic Voltammetry,” *J. Chem. Educ.* **1983**, *60*, 702–706.
- Mabbott, G. A. “An Introduction to Cyclic Voltammetry,” *J. Chem. Educ.* **1983**, *60*, 697–702.

- Petrovic, S. “Cyclic Voltammetry of Hexachloroiridate (IV): An Alternative to the Electrochemical Study of the Ferricyanide Ion,” *Chem. Educator* **2000**, *5*, 231–235.
- Toma, H. E.; Araki, K.; Dovidauskas, S. “A Cyclic Voltammetry Experiment Illustrating Redox Potentials, Equilibrium Constants and Substitution Reaction in Coordination Chemistry,” *J. Chem. Educ.* **2000**, *77*, 1351–1353.
- Walczak, M. W.; Dryer, D. A.; Jacobson, D. D,; Foss, M. G.; Flynn, N. T. “pH-Dependent Redox Couple: Illustrating the Nernst Equation Using Cyclic Voltammetry,” *J. Chem. Educ.* **1997**, *74*, 1195–1197.

Chapter 12

The following set of experiments introduce students to the applications of chromatography and electrophoresis. Experiments are grouped into five categories: gas chromatography, high-performance liquid chromatography, ion-exchange chromatography, size-exclusion chromatography, and electrophoresis.

Gas Chromatography

- Bishop, R. D., Jr. “Using GC–MS to Determine Relative Reactivity Ratios,” *J. Chem. Educ.* **1995**, *72*, 743–745.
- Elderd, D. M.; Kildahl, N. K.; Berka, L. H. “Experiments for Modern Introductory Chemistry: Identification of Arson Accelerants by Gas Chromatography,” *J. Chem. Educ.* **1996**, *73*, 675–677.
- Fleurat-Lessard, P.; Pointet, K.; Renou-Gonnord, M.-F. “Quantitative Determination of PAHs in Diesel Engine Exhausts by GC–MS,” *J. Chem. Educ.* **1999**, *76*, 962–965.
- Galipo, R. C.; Canhoto, A. J.; Walla, M. D.; Morgan, S. L. “Analysis of Volatile Fragrance and Flavor Compounds by Headspace Solid Phase Microextraction and GC–MS,” *J. Chem. Educ.* **1999**, *76*, 245–248.
- Graham, R. C.; Robertson, J. K. “Analysis of Trihalomethanes in Soft Drinks,” *J. Chem. Educ.* **1988**, *65*, 735–737.
- Heinzen, H.; Moyan, P.; Grompone, A. “Gas Chromatographic Determination of Fatty Acid Compositions,” *J. Chem. Educ.* **1985**, *62*, 449–450.
- Kegley, S. E.; Hansen, K. J.; Cunningham, K. L. “Determination of Polychlorinated Biphenyls (PCBs) in River and Bay Sediments,” *J. Chem. Educ.* **1996**, *73*, 558–562.
- Kostecka, K. S.; Rabah, A.; Palmer, C. F., Jr. “GC/MS Analysis of the Aromatic Composition of Gasoline,” *J. Chem. Educ.* **1995**, *72*, 853–854.
- Quach, D. T.; Ciszkowski, N. A.; Finlayson-Pitts, B. J. “A New GC-MS Experiment for the Undergraduate Instrumental Analysis Laboratory in Environmental Chemistry: Methyl-t-butyl Ether and Benzene in Gasoline,” *J. Chem. Educ.* **1998**, *75*, 1595–1598.
- Ramachandran, B. R.; Allen, J. M.; Halpern, A. M. “Air–Water Partitioning of Environmentally Important Organic Compounds,” *J. Chem. Educ.* **1996**, *73*, 1058–1061.
- Rice, G. W. “Determination of Impurities in Whiskey Using Internal Standard Techniques,” *J. Chem. Educ.* **1987**, *64*, 1055–1056.
- Rubinson, J. F.; Neyer-Hilvert, J. “Integration of GC-MS Instrumentation into the Undergraduate Laboratory: Separation and Identification of Fatty Acids in Commercial Fats and Oils,” *J. Chem. Educ.* **1997**, *74*, 1106–1108.
- Rudzinski, W. E.; Beu, S. “Gas Chromatographic Determination of Environmentally Significant Pesticides,” *J. Chem. Educ.* **1982**, *59*, 614–615.
- Sobel, R. M.; Ballantine, D. S.; Ryzhov, V. “Quantitation of Phenol Levels in Oil of Wintergreen Using Gas Chromatography–Mass Spectrometry with Selected Ion Monitoring,” *J. Chem. Educ.* **2005**, *82*, 601–603.
- Welch, W. C.; Greco, T. G. “An Experiment in Manual Multiple Headspace Extraction for Gas Chromatography,” *J. Chem. Educ.* **1993**, *70*, 333–335.
- Williams, K. R.; Pierce, R. E. “The Analysis of Orange Oil and the Aqueous Solubility of d-Limone,” *J. Chem. Educ.* **1998**, *75*, 223–226.
- Wong, J. W.; Ngim, K. K.; Shibamoto, T.; Mabury, S. A.; Eiserich, J. P.; Yeo, H. C. H. “Determination of Formaldehyde in Cigarette Smoke,” *J. Chem. Educ.* **1997**, *74*, 1100–1103.
- Yang, M. J.; Orton, M. L., Pawliszyn, J. “Quantitative Determination of Caffeine in Beverages Using a Combined SPME-GC/MS Method,” *J. Chem. Educ.* **1997**, *74*, 1130–1132.

High-Performance Liquid Chromatography

- Batchelor, J. D.; Jones, B. T. “Determination of the Scoville Heat Value for Hot Sauces and Chilies: An HPLC Experiment,” *J. Chem. Educ.* **2000**, *77*, 266–267.
- Beckers, J. L. “The Determination of Caffeine in Coffee: Sense or Nonsense?” *J. Chem. Educ.* **2004**, *81*, 90–93.

- Betts, T. A. “Pungency Quantitation of Hot Pepper Sauces Using HPLC,” *J. Chem. Educ.* **1999**, *76*, 240–244.
- Bidlingmeyer, B. A.; Schmitz, S. “The Analysis of Artificial Sweeteners and Additives in Beverages by HPLC,” *J. Chem. Educ.* **1991**, *68*, A195–A200.
- Bohman, O.; Engdahl, K.-A.; Johnsson, H. “High Performance Liquid Chromatography of Vitamin A: A Quantitative Determination,” *J. Chem. Educ.* **1982**, *59*, 251–252.
- Brenneman, C. A.; Ebeler, S. E. “Chromatographic Separations Using Solid-Phase Extraction Cartridges: Separation of Wine Phenolics,” *J. Chem. Educ.* **1999**, *76*, 1710–1711.
- Cantwell, F. F.; Brown, D. W. “Liquid Chromatographic Determination of Nitroanilines,” *J. Chem. Educ.* **1981**, *58*, 820–823.
- DiNunzio, J. E. “Determination of Caffeine in Beverages by High Performance Liquid Chromatography,” *J. Chem. Educ.* **1985**, *62*, 446–447.
- Ferguson, G. K. “Quantitative HPLC Analysis of an Analgesic/Caffeine Formulation: Determination of Caffeine,” *J. Chem. Educ.* **1998**, *75*, 467–469.
- Ferguson, G. K. “Quantitative HPLC Analysis of a Psychotherapeutic Medication: Simultaneous Determination of Amitriptyline Hydrochloride and Perphenazine,” *J. Chem. Educ.* **1998**, *75*, 1615–1618.
- Goodney, D. E. “Analysis of Vitamin C by High-Pressure Liquid Chromatography,” *J. Chem. Educ.* **1987**, *64*, 187–188.
- Guevremont, R.; Quigley, M. N. “Determination of Paralytic Shellfish Poisons Using Liquid Chromatography,” *J. Chem. Educ.* **1994**, *71*, 80–81.
- Haddad, P.; Hutchins, S.; Tuffy, M. “High Performance Liquid Chromatography of Some Analgesic Compounds,” *J. Chem. Educ.* **1983**, *60*, 166–168.
- Huang, J.; Mabury, S. A.; Sagebiel, J. C. “Hot Chili Peppers: Extraction, Cleanup, and Measurement of Capscaicin,” *J. Chem. Educ.* **2000**, *77*, 1630–1631.
- Joeseph, S. M.; Palasota, J. A. “The Combined Effect of pH and Percent Methanol on the HPLC Separation of Benzoic Acid and Phenol,” *J. Chem. Educ.* **2001**, *78*, 1381–1383.
- Lehame, S. “The Separation of Copper, Iron, and Cobalt Tetramethylene Dithiocarbamates by HPLC,” *J. Chem. Educ.* **1986**, *63*, 727–728.
- Luo, P.; Luo, M. Z.; Baldwin, R. P. “Determination of Sugars in Food Products,” *J. Chem. Educ.* **1993**, *70*, 679–681.
- Mueller, B. L.; Potts, L. W. “HPLC Analysis of an Asthma Medication,” *J. Chem. Educ.* **1988**, *65*, 905–906.
- Munari, M.; Miurin, M.; Goi, G. “Didactic Application to Riboflavin HPLC Analysis,” *J. Chem. Educ.* **1991**, *68*, 78–79.
- Orth, D. L. “HPLC Determination of Taurine in Sports Drinks,” *J. Chem. Educ.* **2001**, *78*, 791–792.
- Remcho, V. T.; McNair, H. M.; Rasmussen, H. T. “HPLC Method Development with the Photodiode Array Detector,” *J. Chem. Educ.* **1992**, *69*, A117–A119.
- Richardson, W. W., III; Burns, L. “HPLC of the Polypeptides in a Hydrolyzate of Egg-White Lysozyme,” *J. Chem. Educ.* **1988**, *65*, 162–163.
- Silveira, A., Jr.; Koehler, J. A.; Beadel, E. F., Jr.; Monore, P. A. “HPLC Analysis of Chlorophyll a, Chlorophyll b, and β-Carotene in Collard Greens,” *J. Chem. Educ.* **1984**, *61*, 264–265.
- Siturmorang, M.; Lee, M. T. B.; Witzeman, L. K.; Heineman, W. R. “Liquid Chromatography with Electrochemical Detection (LC-EC): An Experiment Using 4-Aminophenol,” *J. Chem. Educ.* **1998**, *75*, 1035–1038.
- Sottofattori, E.; Raggio, R.; Bruno, O. “Milk as a Drug Analysis Medium: HPLC Determination of Isoniazid,” *J. Chem. Educ.* **2003**, *80*, 547–549.
- Strohl, A. N. “A Study of Colas: An HPLC Experiment,” *J. Chem. Educ.* **1985**, *62*, 447–448.
- Tran, C. D.; Dotlich, M. “Enantiomeric Separation of Beta-Blockers by High Performance Liquid Chromatography,” *J. Chem. Educ.* **1995**, *72*, 71–73.
- Van Arman, S. A.; Thomsen, M. W. “HPLC for Undergraduate Introductory Laboratories,” *J. Chem. Educ.* **1997**, *74*, 49–50.
- Wingen, L. M.; Low, J. C.; Finlayson-Pitts, B. J. “Chromatography, Absorption, and Fluorescence: A New Instrumental Analysis Experiment on the Measurement of Polycyclic Aromatic Hydrocarbons in Cigarette Smoke,” *J. Chem. Educ.* **1998**, *75*, 1599–1603.

Ion-Exchange Chromatography

- Bello, M. A.; Gustavo González, A. “Determination of Phosphate in Cola Beverages Using Nonsuppressed Ion Chromatography,” *J. Chem. Educ.* **1996**, *73*, 1174–1176.
- Kieber, R. J.; Jones, S. B. “An Undergraduate Laboratory for the Determination of Sodium, Potassium, and Chloride,” *J. Chem. Educ.* **1994**, *71*, A218–A222.
- Koubek, E.; Stewart, A. E. “The Analysis of Sulfur in Coal,” *J. Chem. Educ.* **1992**, *69*, A146–A148.

- Sinniah, K.; Piers, K. “Ion Chromatography: Analysis of Ions in Pond Water,” *J. Chem. Educ.* **2001**, *78*, 358–362.
- Xia, K.; Pierzynski, G. “Competitive Sorption between Oxalate and Phosphate in Soil: An Environmental Chemistry Laboratory Using Ion Chromatography,” *J. Chem. Educ.* **2003**, *80*, 71–75.

Size-Exchange Chromatography

- Brunauer, L. S.; Davis, K. K. “Size Exclusion Chromatography: An Experiment for High School and Community College Chemistry and Biotechnology Laboratory Programs,” *J. Chem. Educ.* **2008**, *85*, 683–685.
- Saiz, E.; Tarazona, M. P. “Size-Exclusion Chromatography Using Dual Detection,” *Chem. Educator* **2000**, *5*, 324–328.

Electrophoresis

- Almarez, R. T.; Kochis, M. “Microscale Capillary Electrophoresis: A Complete Instrumentation Experiment for Chemistry Students at the Undergraduate Junior or Senior Level,” *J. Chem. Educ.* **2003**, *80*, 316–319.
- Beckers, J. L. “The Determination of Caffeine in Coffee: Sense or Nonsense?” *J. Chem. Educ.* **2004**, *81*, 90–93.
- Beckers, J. L. “The Determination of Vanillin in a Vanilla Extract,” *J. Chem. Educ.* **2005**, *82*, 604–606.
- Boyce, M. “Separation and Quantification of Simple Ions by Capillary Zone Electrophoresis,” *J. Chem. Educ.* **1999**, *76*, 815–819.
- Conradi, S.; Vogt, C.; Rohde, E. “Separation of Enatiomeric Barbiturates by Capillary Electrophoresis Using a Cyclodextrin-Containing Run Buffer,” *J. Chem. Educ.* **1997**, *74*, 1122–1125.
- Conte, E. D.; Barry, E. F.; Rubinstein, H. “Determination of Caffeine in Beverages by Capillary Zone Electrophoresis,” *J. Chem. Educ.* **1996**, *73*, 1169–1170.
- Demay, S.; Martin-Girardeau, A.; Gonnord, M.-F. “Capillary Electrophoretic Quantitative Analysis of Anions in Drinking Water,” *J. Chem. Educ.* **1999**, *76*, 812–815.
- Emry, R.; Cutright, R. D.; Wright, J.; Markwell, J. “Candies to Dye for: Cooperative, Open-Ended Student Activities to Promote Understanding of Electrophoretic Fractionation,” *J. Chem. Educ.* **2000**, *77*, 1323–1324.
- Gardner, W. P.; Girard, J. E. “Analysis of Common Household Cleaner-Disinfectants by Capillary Electrophoresis,” *J. Chem. Educ.* **2000**, *77*, 1335–1338.
- Gruenhagen, J. A.; Delaware, D.; Ma, Y. “Quantitative Analysis of Non-UV-Absorbing Cations in Soil Samples by High-Performance Capillary Electrophoresis,” *J. Chem. Educ.* **2000**, *77*, 1613–1616.
- Hage, D. S.; Chattopadhyay, A.; Wolfe, C. A. C.; Grundman, J.; Kelter, P. B. “Determination of Nitrate and Nitrite in Water by Capillary Electrophoresis,” *J. Chem. Educ.* **1998**, *75*, 1588–1590.
- Herman, H. B.; Jezorek, J. R.; Tang, Z. “Analysis of Diet Tonic Water Using Capillary Electrophoresis,” *J. Chem. Educ.* **2000**, *77*, 743–744.
- Janusa, M. A.; Andermann, L. J.; Kliebert, N. M.; Nannie, M. H. “Determination of Chloride Concentration Using Capillary Electrophoresis,” *J. Chem. Educ.* **1998**, *75*, 1463–1465.
- McDevitt, V. L.; Rodríguez, A.; Williams, K. R. “Analysis of Soft Drinks: UV Spectrophotometry, Liquid Chromatography, and Capillary Electrophoresis,” *J. Chem. Educ.* **1998**, *75*, 625–629.
- Palmer, C. P. “Demonstrating Chemical and Analytical Concepts in the Undergraduate Laboratory Using Capillary Electrophoresis and Micellar Electrokinetic Chromatography,” *J. Chem. Educ.* **1999**, *76*, 1542–1543.
- Pursell, C. J.; Chandler, B.; Bushey, M. M. “Capillary Electrophoresis Analysis of Cations in Water Samples,” *J. Chem. Educ.* **2004**, *81*, 1783–1786.
- Solow, M. “Weak Acid pK_a Determination Using Capillary Zone Electrophoresis,” *J. Chem. Educ.* **2006**, *83*, 1194–1195.
- Thompson, L.; Veening, H.; Strain, T. G. “Capillary Electrophoresis in the Undergraduate Instrumental Analysis Laboratory: Determination of Common Analgesic Formulations,” *J. Chem. Educ.* **1997**, *74*, 1117–1121.
- Vogt, C.; Conradi, S.; Rhode, E. “Determination of Caffeine and Other Purine Compounds in Food and Pharmaceuticals by Micellar Electrokinetic Chromatography” *J. Chem. Educ.* **1997**, *74*, 1126–1130.
- Weber, P. L.; Buck, D. R. “Capillary Electrophoresis: A Fast and Simple Method for the Determination of the Amino Acid Composition of Proteins,” *J. Chem. Educ.* **1994**, *71*, 609–612.
- Welder, F.; Colyer, C. L. “Using Capillary Electrophoresis to Determine the Purity of Acetylsalicylic Acid Synthesized in the Undergraduate Laboratory,” *J. Chem. Educ.* **2001**, *78*, 1525–1527.
- Williams, K. R.; Adhyaru, B.; German, I.; Russell, T. “Determination of a Diffusion Coefficient by Capillary Electrophoresis,” *J. Chem. Educ.* **2002**, *79*, 1475–1476.

The following texts provide a good introduction to the broad field of separations, including chromatography and electrophoresis.

- Giddings, J. C. *Unified Separation Science*, Wiley-Interscience: New York 1991.
- Karger, B. L.; Snyder, L. R.; Harvath, C. *An Introduction to Separation Science*, Wiley-Interscience: New York, 1973
- Miller, J. M. *Separation Methods in Chemical Analysis*, Wiley-Interscience: New York, 1975.
- Poole, C. F. *The Essence of Chromatography*, Elsevier: Amsterdam, 2003.

A more recent discussion of peak capacity is presented in the following paper.

- Davis, J. M.; Stoll, D. R.; Carr, P. W. "Dependence of Effective Peak Capacity in Comprehensive Two-Dimensional Separations on the Distribution of Peak Capacity between the Two Dimensions," *Anal. Chem.* **2008**, *80*, 8122–8134.
- Li, X.; Stoll, D. R.; Carr, P. W. "Equation for Peak Capacity Estimation in Two-Dimensional Liquid Chromatography," *Anal. Chem.* **2009**, *81*, 845–850.
- Shen, Y.; Lee, M. "General Equation for Peak Capacity in Column Chromatography," *Anal. Chem.* **1998**, *70*, 3853–3856.

The following references may be consulted for more information on gas chromatography.

- Grob, R. L., ed, *Modern Practice of Gas Chromatography*, Wiley-Interscience: New York, 1972.
- Hinshaw, J. V. "A Compendium of GC Terms and Techniques," *LC•GC* **1992**, *10*, 516–522.
- Ioffe, B. V.; Vitenberg, A. G. *Head-Space Analysis and Related Methods in Gas Chromatography*, Wiley-Interscience: New York, 1982.
- Kitson, F. G.; Larsen, B. S.; McEwen, C. N. *Gas Chromatography and Mass Spectrometry: A Practical Guide*, Academic Press: San Diego, 1996.
- McMaster, M. C. *GC/MS: A Practical User's Guide*, Wiley-Interscience: Hoboken, NJ, 2008.

The following references provide more information on high-performance liquid chromatography.

- Dorschel, C. A.; Ekmanis, J. L.; Oberholtzer, J. E.; Warren, Jr. F. V.; Bidlingmeyer, B. A. "LC Detectors," *Anal. Chem.* **1989**, *61*, 951A–968A.
- Ehlert, S.; Tallarek, U. "High-pressure liquid chromatography in lab-on-a-chip devices," *Anal. Bioanal. Chem.* **2007**, *388*, 517–520.
- Francois, I.; Sandra, K.; Sandra, P. "Comprehensive liquid chromatography: Fundamental aspects and practical considerations—A review," *Anal. Chim. Acta* **2009**, *641*, 14–31.
- Harris, C. M. "Shrinking the LC Landscape," *Anal. Chem.* **2003**, *75*, 64A–69A.
- Meyer, V. R. *Pitfalls and Errors of HPLC in Pictures*, Wiley-VCH: Weinheim, Germany, 2006.
- Pozo, O. J.; Van Eenoo, P.; Deventer, K.; Delbeke, F. T. "Detection and characterization of anabolic steroids in doping analysis by LC–MS," *Trends Anal. Chem.* **2008**, *27*, 657–671.
- Scott, R. P. W. "Modern Liquid Chromatography," *Chem. Soc. Rev.* **1992**, *21*, 137–145.
- Simpson, C. F., ed. *Techniques in Liquid Chromatography*, Wiley-Hayden: Chichester, England; 1982.
- Snyder, L. R.; Glajch, J. L.; Kirkland, J. J. *Practical HPLC Method Development*, Wiley-Interscience: New York,1988.
- van de Merbel, N. C. "Quantitative determination of endogenous compounds in biological samples using chromatographic techniques," *Trends Anal. Chem.* **2008**, *27*, 924–933.
- Yeung, E. S. "Chromatographic Detectors: Current Status and Future Prospects," *LC•GC* **1989**, *7*, 118–128.

The following references may be consulted for more information on ion chromatography.

- Shpigun, O. A.; Zolotov, Y. A. *Ion Chromatography in Water Analysis*, Ellis Horwood: Chichester, England, 1988.
- Smith, F. C. Jr.; Chang, R. C. *The Practice of Ion Chromatography*, Wiley-Interscience: New York, 1983.

The following references may be consulted for more information on supercritical fluid chromatography.

- Palmieri, M. D. "An Introduction to Supercritical Fluid Chromatography. Part I: Principles and Applications," *J. Chem. Educ.* **1988**, *65*, A254–A259.
- Palmieri, M. D. "An Introduction to Supercritical Fluid Chromatography. Part II: Applications and Future Trends," *J. Chem. Educ.* **1989**, *66*, A141–A147.

The following references may be consulted for more information on capillary electrophoresis.

- Baker, D. R. *Capillary Electrophoresis*, Wiley-Interscience: New York, 1995.
- Copper, C. L. "Capillary Electrophoresis: Part I. Theoretical and Experimental Background," *J. Chem. Educ.* **1998**, *75*, 343–347.
- Copper, C. L.; Whitaker, K. W. "Capillary Electrophoresis: Part II. Applications," *J. Chem. Educ.* **1998**, *75*, 347–351.

- DeFrancesco, L. “Capillary Electrophoresis: Finding a Niche,” *Today’s Chemist at Work*, February 2002, 59–64.
- Ekins, R. P. “Immunoassay, DNA Analysis, and Other Ligand Binding Assay Techniques: From Electropherograms to Multiplexed, Ultrasensative Microarrays on a Chip,” *J. Chem. Educ.* **1999**, *76*, 769–780.
- Revermann, T.; Götz, S.; Künnemeyer, J.; Karst, U. “Quantitative analysis by microchip capillary electrophoresis—current limitations and problem-solving strategies,” *Analyst* **2008**, *133*, 167–174.
- Timerbaev, A. R. “Capillary electrophoresis coupled to mass spectrometry for biospeciation analysis: critical evaluation,” *Trends Anal. Chem.* **2009**, *28*, 416–425.
- Unger, K. K.; Huber, M.; Hennessy, T. P.; Hearn, M. T. W.; Walhagen, K. “A Critical Appraisal of Capillary Electrochromatography,” *Anal. Chem.* **2002**, *74*, 200A–207A.
- Varenne, A.; Descroix, S. “Recent strategies to improve resolution in capillary electrophoresis—A review,” *Anal. Chim. Acta* **2008**, *628*, 9–23.
- Vetter, A. J.; McGowan, G. J. “The Escalator—An Analogy for Explaining Electroosmotic Flow,” J. Chem. Educ. 2001, 78, 209–211.
- Xu, Y. “Tutorial: Capillary Electrophoresis,” *Chem. Educator*, **1996**, *1(2)*, 1–14.

The application of spreadsheets and computer programs for modeling chromatography is described in the following papers.

- Abbay, G. N.; Barry, E. F.; Leepipatpiboon, S.; Ramstad, T.; Roman, M. C.; Siergiej, R. W.; Snyder, L. R.; Winniford, W. L. “Practical Applications of Computer Simulation for Gas Chromatography Method Development,” *LC•GC* **1991**, *9*, 100–114.
- Drouen, A.; Dolan, J. W.; Snyder, L. R.; Poile, A.; Schoenmakers, P. J. “Software for Chromatographic Method Development,” *LC•GC* **1991**, *9*, 714–724.
- Kevra, S. A.; Bergman, D. L.; Maloy, J. T. “A Computational Introduction to Chromatographic Bandshape Analysis,” *J. Chem. Educ.* **1994**, *71*, 1023–1028.
- Rittenhouse, R. C. “HPLC for Windows: A Computer Simulation of High-Performance Liquid Chromatography,” *J. Chem. Educ.* **1995**, *72*, 1086–1087.
- Shalliker, R. A.; Kayillo, S.; Dennis, G. R. “Optimizing Chromatographic Separations: An Experiment Using an HPLC Simulator,” *J. Chem. Educ.* **2008**, *85*, 1265–1268.
- Sundheim, B. R. “Column Operations: A Spreadsheet Model,” *J. Chem. Educ.* **1992**, *69*, 1003–1005**.**

The following papers discuss column efficiency, peak shapes, and overlapping chromatographic peaks.

- Bildingmeyer, B. A.; Warren, F. V., Jr. “Column Efficiency Measurement,” *Anal. Chem.* **1984**, *56*, 1583A–1596A.
- Hawkes, S. J. “Distorted Chromatographic Peaks,” *J. Chem. Educ.* **1994**, *71*, 1032–1033.
- Hinshaw, J. “Pinning Down Tailing Peaks,” *LC•GC* **1992**, *10*, 516–522.
- Meyer, V. K. “Chromatographic Integration Errors: A Closer Look at a Small Peak,” *LC•GC North* America **2009**, *27*, 232–244.
- Reid, V. R.; Synovec, R. E. “High-speed gas chromatography: The importance of instrumentation optimization and the elimination of extra-column band broadening,” *Talanta***2008**, *76*, 703–717.

Chapter 13

The following set of experiments introduce students to the applications of chemical kinetic methods, including enzyme kinetic methods, and flow injection analysis.

Chemical Kinetic Methods

- Abramovitch, D. A.; Cunningham, L. K.; Litwer, M. R. “Decomposition Kinetics of Hydrogen Perox- ide: Novel Lab Experiments Employing Computer Technology,” *J. Chem. Educ.* **2003**, *80*, 790–792.
- Bateman, Jr. R. C.; Evans, J. A. “Using the Glucose Oxidase/Peroxidase Systems in Enzyme Kinetics,” *J. Chem. Educ.* **1995**, *72*, A240–A241.
- Bendinskas, K.; DiJacomo, C.; Krill, A.; Vitz, E. “Kinetics of Alcohol Dehydrogenase-Catalyzed Oxidation of Ethanol Followed by Visible Spectroscopy,” *J. Chem. Educ.* **1068**, *82*, 1068–1070.
- Clark, C. R. “A Stopped-Flow Kinetics Experiment for Advanced Undergraduate Laboratories: Formation of Iron(III) Thiocyanate,” *J. Chem. Educ.* **1997**, *74*, 1214–1217.
- Diamandis, E. P.; Koupparis, M. A.; Hadjiionnou, T. P. “Kinetic Studies with Ion-Selective Electrodes: Determination of Creatinine in Urine with a Picrate Ion-Selective Electrode,” *J. Chem. Educ.* **1983**, *60*, 74–76.

- Frey, M. W.; Frey, S. T.; Soltau, S. R. “Exploring the pH Dependence of L-leucine-*p*-nitroanilide Cleavage by Aminopeptidase Aeromonas Proteolytica: A Combined Buffer-Enzyme Kinetics Experiment for the General Chemistry Laboratory,” *Chem. Educator* **2010**, *15*, 117-120.
- Gooding, J. J.; Yang, W.; Situmorang, M. “Bioanalytical Experiments for the Undergraduate Laboratory: Monitoring Glucose in Sport Drinks,” *J. Chem. Educ.* **2001**, *78*, 788–790.
- Hamilton, T. M.; Dobie-Galuska, A. A.; Wietstock, S. M. “The *o*-Phenylenediamine-Horseradish Peroxidase System: Enzyme Kinetics in the General Chemistry Lab,” *J. Chem. Educ.* **1999**, *76*, 642– 644.
- Johnson, K. A. “Factors Affecting Reaction Kinetics of Glucose Oxidase,” *J. Chem. Educ.* **2002**, *79*,74–76.
- Mowry, S.; Ogren, P. J. “Kinetics of Methylene Blue Reduction by Ascorbic Acid,” *J. Chem. Educ.* **1999**, *76*, 970–974.
- Nyasulu, F. W.; Barlag, R. “Gas Pressure Sensor Monitored Iodide-Catalyzed Decomposition Kinetics of Hydrogen Peroxide: An Initial Rate Approach,” *Chem. Educator* **2008**, *13*, 227–230.
- Nyasulu, F. W.; Barlag, R. “Thermokinetics: Iodide-Catalyzed Decomposition Kinetics of Hydrogen Peroxide; An Integrated Rate Approach,” *Chem. Educator* **2010**, *15*, 168–170.
- Pandey, S.; McHale, M. E. R.; Horton, A. M.; Padilla, S. A.; Trufant, A. L.; De La Sancha, N. U.; Vela, E.; Acree, Jr., W. E. “Kinetics-Based Indirect Spectrophotometric Method for the Simultaneous Determination of $MnO^{4-}$and $Cr_2O_7^{2-}$,” *J. Chem. Educ.* **1998**, *75*, 450–452.
- Stock, E.; Morgan, M. “A Spectroscopic Analysis of the Kinetics of the Iodine Clock Reaction without Starch,” *Chem. Educator* **2010**, *15*, 158–161.
- Vasilarou, A.-M. G.; Georgiou, C. A. “Enzymatic Spectrophotometric Reaction Rate Determination of Glucose in Fruit Drinks and Carbonated Beverages,” *J. Chem. Educ*. **2000**, *77*, 1327–1329.
- Williams, K. R.; Adhyaru, B.; Timofeev, J.; Blankenship, M. K. “Decomposition of Aspartame. A Kinetics Experiment for Upper-Level Chemistry Laboratories,” *J. Chem. Educ.* **2005**, *82*, 924–925.

Flow Injection Methods

- Carroll, M. K.; Tyson, J. F. “An Experiment Using Time-Based Detection in Flow Injection Analysis,” *J. Chem. Educ.* **1993**, *70*, A210–A216.
- Conceição, A. C. L.; Minas da Piedade, M. E. “Determination of Acidity Constants by Gradient Flow-Injection Titration,” *J. Chem. Educ.* **2006**, *83*, 1853–1856.
- Hansen, E. H.; Ruzicka, J. “The Principles of Flow Injection Analysis as Demonstrated by Three Lab Exercises,” *J. Chem. Educ.* **1979**, *56*, 677–680.
- McKelvie, I. D.; Cardwell, T. J.; Cattrall, R. W. “A Microconduit Flow Injection Analysis Demonstration using a 35-mm Slide Projector,” *J. Chem. Educ.* **1990**, *67*, 262–263.
- Meyerhoff, M. E.; Kovach, P. M. “An Ion-Selective Electrode/Flow Injection Analysis Experiment: Determination of Potassium in Serum,” *J. Chem. Educ.* **1983**, *60*, 766–768.
- Nóbrega, J. A.; Rocha, F. R. P. “Ionic Strength Effect on the Rate of Reduction of Hexacyanoferrate(II) by Ascorbic Acid,” *J. Chem. Educ.* **1997**, *74*, 560–562.
- Ríos, A.; Luque de Castro, M.; Valcárcel, M. “Determination of Reaction Stoichiometries by Flow Injection Analysis,” *J. Chem. Educ.* **1986**, *63*, 552–553.
- Stults, C. L. M.; Wade, A. P.; Crouch, S. R. “Investigation of Temperature Effects on Dispersion in a Flow Injection Analyzer,” *J. Chem. Educ.* **1988**, *65*, 645–647.
- Wolfe, C. A. C.; Oates, M. R.; Hage, D. S. “Automated Protein Assay Using Flow Injection Analysis,” *J. Chem. Educ.* **1998**, *75*, 1025–1028.

The following sources provides a general review of the importance of chemical kinetics in analytical chemistry.

- Bergmyer, H. U.; Grassl, M. *Methods of Enzymatic Analysis*, Verlag Chemie: Deerfield Beach, FL, 3rd Ed., 1983.
- Laitinen, H. A.; Ewing, G. W., eds., *A History of Analytical Chemistry*, The Division of Analytical Chemistry of the American Chemical Society: Washington, D. C., 1977, pp. 97–102.
- Malmstadt, H. V.; Delaney, C. J.; Cordos, E. A. “Reaction-Rate Methods of Chemical Analysis,” *Crit. Rev. Anal. Chem.* **1972**, *2, 559–619.*
- Mark, H. B.; Rechnitz, G. A. *Kinetics in Analytical Chemistry*, Wiley: New York, 1968.

- Mottola, H. A. “Catalytic and Differential Reaction-Rate Methods of Chemical Analysis,” *Crit. Rev. Anal. Chem.* ***1974**, 4, 229–280*.
- Mottola, H. A. “Some Kinetic Aspects Relevant to Contemporary Analytical Chemistry,” *J. Chem. Educ.* **1981**, *58*, 399–403.
- Mottola, H. A. *Kinetic Aspects of Analytical Chemistry*, Wiley: New York, 1988.
- Pardue, H. L. “A Comprehensive Classification of Kinetic Methods of Analysis Used in Clinical Chemistry,” Clin. Chem. 1977, 23, 2189–2201.
- Pardue, H. L. “Kinetic Aspects of Analytical Chemistry,” *Anal. Chim. Acta*, **1989**, *216*, 69–107.
- Perez-Bendito, D.; Silva, M. *Kinetic Methods in Analytical Chemistry*, Ellis Horwood: Chichester, 1988.
- Pisakiewicz, D. *Kinetics of Chemical and Enzyme-Catalyzed Reactions*, Oxford University Press: New York, 1977.

The following instrumental analysis textbooks may be consulted for further information on the detectors and signal analyzers used in radiochemical methods of analysis.

- Skoog, D. A.; Holler, F. J.; Nieman, T. A. *Principles of Instrumental Analysis*, 5th Ed., Saunders College Publishing/Harcourt Brace and Co.: Philadelphia., 1998, Chapter 32.
- Strobel, H. A.; Heineman, W. R. *Chemical Instrumentation: A Systematic Approach*, 3rd Ed., Wiley-Interscience: New York, 1989.

The following resources provide additional information on the theory and application of flow injection analysis.

- Andrew, K. N.; Blundell, N. J.; Price, D.; Worsfold, P. J. “Flow Injection Techniques for Water Monitoring,” *Anal. Chem.* **1994**, *66*, 916A–922A.
- Betteridge, D. “Flow Injection Analysis,” *Anal. Chem.* **1978**, *50*, 832A–846A.
- Kowalski, B. R.; Ruzicka, J. Christian, G. D. “Flow Chemography - The Future of Chemical Education,” *Trends Anal. Chem.* **1990**, *9*, 8–13.
- Mottola, H. A. “Continuous Flow Analysis Revisited,” *Anal. Chem.* **1981**, *53*, 1312A–1316A.
- Ruzicka, J. “Flow Injection Analysis: From Test Tube to Integrated Microconduits,” *Anal. Chem.* **1983**, *55*, 1040A–1053A.
- Ruzicka, J.; Hansen, E. H. *Flow-Injection Analysis*, Wiley-Interscience: New York, 1989.
- Ruzicka, J.; Hansen, E. H. “Retro-Review of Flow-Injection Analysis,” *Trends Anal. Chem.* **2008**, *27*,390–393.
- Silvestre, C. I. C.; Santos, J. L. M.; Lima, J. L. F. C.; Zagatto, E. A. G. “Liquid-Liquid Extraction in Flow Analysis: A Critical Review,” *Anal. Chim. Acta* **2009**, *652*, 54–65.
- Stewart, K. K. “Flow Injection Analysis: New Tools for Old Assays, New Approaches to Analytical Measurements,” *Anal. Chem.* **1983**, *55*, 931A–940A.
- Tyson, J. F. “Atomic Spectrometry and Flow Injection Analysis: A Synergic Combination,” *Anal. Chim. Acta*, **1988**, *214, 57–75*.
- Valcarcel, M.; Luque de Castro, M. D. *Flow-Injection Analysis: Principles and Applications*, Ellis Horwood: Chichester, England, 1987.

Chapter 14

The following set of experiments provide practical examples of the optimization of experimental conditions. Examples include simplex optimization, factorial designs for developing empirical models of response surfaces, and fitting experimental data to theoretical models of the response surface.

- Amenta, D. S.; Lamb, C. E.; Leary, J. J. “Simplex Optimization of Yield of *sec*-Butylbenzene in a Friedel-Crafts Alkylation,” *J. Chem. Educ.* **1979**, *56*, 557–558.
- Harvey, D. T.; Byerly, S.; Bowman, A.; Tomlin, J. “Optimization of HPLC and GC Separations Using Response Surfaces,” *J. Chem. Educ.* **1991**, *68*, 162–168.
- Leggett, D. L. “Instrumental Simplex Optimization,” *J. Chem. Educ.* **1983**, *60*, 707–710.
- Oles, P. J. “Fractional Factorial Experimental Design as a Teaching Tool for Quantitative Analysis,” *J. Chem. Educ.* **1998**, *75*, 357–359.
- Palasota, J. A.; Deming, S.N. “Central Composite Experimental Design,” *J. Chem. Educ.* **1992**, *69*, 560–561.
- Sangsila, S.; Labinaz, G.; Poland, J. S.; vanLoon, G. W. “An Experiment on Sequential Simplex Optimization of an Atomic Absorption Analysis Procedure,” *J. Chem. Educ.* **1989**, *66*, 351–353.

- Santos-Delgado, M. J.; Larrea-Tarruella, L. “A Didactic Experience of Statistical Analysis for the Determination of Glycine in a Nonaqueous Medium using ANOVA and a Computer Program,” *J. Chem. Educ.* **2004**, *81*, 97–99.
- Shavers, C. L.; Parsons, M. L.; Deming, S. N. “Simplex Optimization of Chemical Systems,” *J. Chem Educ.* **1979**, *56*, 307–309.
- Stieg, S. “A Low-Noise Simplex Optimization Experiment,” *J. Chem. Educ.* **1986**, *63*, 547–548.
- Stolzberg, R. J. “Screening and Sequential Experimentation: Simulations and Flame Atomic Absorption Spectrometry Experiments,” *J. Chem. Educ.* **1997**, *74*, 216–220.
- Van Ryswyk, H.; Van Hecke, G. R. “Attaining Optimal Conditions,” *J. Chem. Educ.* **1991**, *66*, 878–882.

The following texts and articles provide an excellent discussion of optimization methods based on searching algorithms and mathematical modeling use factorial designs, including a discussion of the relevant calculations. A few of these sources discuss other types of experimental designs.

- Bayne, C. K.; Rubin, I. B. *Practical Experimental Designs and Optimization Methods for Chemists*, VCH Publishers: Deerfield Beach, FL; 1986.
- Box, G. E. P. “Statistical Design in the Study of Analytical Methods,” *Analyst* **1952**, *77*, 879–891.
- Deming, S. N.; Morgan, S. L. *Experimental Design: A Chemometric Approach*, Elsevier: Amsterdam, 1987.
- Ferreira, S. L. C.; dos Santos, W. N. L.; Quintella, C. M.; Neto, B. B.; Bosque-Sendra, J. M. “Doehlert • Matrix: A Chemometric Tool for Analytical Chemistry—Review,” *Talanta* **2004**, *63*, 1061–1067.
- Ferreira, S. L. C.; Bruns, R. E.; Ferreira, H. S.; Matos, G. D.; David, J. M.; Brandão, G. C.; da Silva, E. G. P.; Portugal, L. A.; dos Reis, P. S.; Souza, A. S.; dos Santos, W. N. L. “Box-Behnken Design: An Alternative for the Optimization of Analytical Methods,” *Anal. Chim. Acta* **2007**, *597*, 179–186.
- Gonzalez, A. G. “Two Level Factorial Experimental Designs Based on Multiple Linear Regression Models: A Tutorial Digest Illustrated by Case Studies,” *Anal. Chim. Acta* **1998**, *360*, 227–241.
- Goupy, J. “What Kind of Experimental Design for Finding and Checking Robustness of Analytical Methods?” *Anal. Chim. Acta* **2005**, *544*, 184–190.
- Hendrix, C. D. “What Every Technologist Should Know About Experimental Design,” *Chemtech* **1979**, *9*, 167–174.
- Hendrix, C. D. “Through the Response Surface with Test Tube and Pipe Wrench,” *Chemtech* **1980**, *10*, 488–497.
- Leardi, R. “Experimental Design: A Tutorial,” *Anal. Chim. Acta* **2009**, *652*, 161–172.
- Liang, Y. “Comparison of Optimization Methods,” *Chromatography Review* **1985**, *12(2)*, 6–9.
- Morgan, E. *Chemometrics: Experimental Design*, John Wiley and Sons: Chichester, 1991.
- Thompson, M., ed. “Experimental Design and Optimization (1): An Introduction to Some Basic Concepts,” AMC Technical Brief 24, June 2006.
- Thompson, M., ed. “Experimental Design and Optimization (2): Handling Uncontrolled Factors” AMC Technical Brief 26, December 2006.
- Thompson, M., ed. “Experimental Design and Optimization (3): Some Fractional Factorial Designs,” AMC Technical Brief 36, January 2009.
- Walters, F. H.; Morgan, S. L.; Parker, L. P., Jr.; Deming, S. N. *Sequential Simplex Optimization*, CRC Press: Boca Raton, FL, 1991. (an electronic version of this text is freely available from Multisimplex AB and is available here.)

The following texts provide additional information about ANOVA calculations, including discussions of two-way analysis of variance.

- Graham, R. C. *Data Analysis for the Chemical Sciences*, VCH Publishers: New York, 1993.
- Miller, J. C.; Miller, J. N. *Statistics for Analytical Chemistry*, Ellis Horwood Limited: Chichester, 1988.

The following resources provide additional information on the validation of analytical methods.

- Gonzalez, A. G.; Herrador, M. A. “A Practical Guide to Analytical Method Validation, Including Measurement Uncertainty and Accuracy Profiles,” *Trends Anal. Chem.* **2007**, *26*, 227–238.
- Thompson, M.; Ellison, S. L. R.; Wood, R. “Harmonized Guidelines for Single-Laboratory Validation of Analytical Methods,” *Pure Appl. Chem.* **2002**, *74*, 835–855.

Chapter 15

The following three experiments introduce aspects of quality assurance and quality control.

- Bell, S. C.; Moore, J. “Integration of Quality Assurance/Quality Control into Quantitative Analysis,” *J. Chem. Educ.* **1998**, *75*, 874–877.
- Cancilla, D. A. “Integration of Environmental Analytical Chemistry with Environmental Law: The Development of a Problem-Based Laboratory,” *J. Chem. Educ.* **2001**, *78*, 1652–1660.
- Laquer, F. C. “Quality Control Charts in the Quantitative Analysis Laboratory Using Conductance Measurement,” *J. Chem. Educ.* **1990**, *67*, 900–902.
- Marcos, J.; Ríos, A.; Valcárcel, M. “Practicing Quality Control in a Bioanalytical Experiment,” *J. Chem. Educ.* **1995**, *72*, 947–949.

The following texts and articles may be consulted for an additional discussion of quality assurance and quality control.

- Amore, F. “Good Analytical Practices,” *Anal. Chem.* **1979**, *51*, 1105A–1110A.
- Barnard, Jr. A. J.; Mitchell, R. M.; Wolf, G. E. “Good Analytical Practices in Quality Control,” *Anal. Chem.* **1978**, *50*, 1079A–1086A.
- Cairns, T.; Rogers, W. M. “Acceptable Analytical Data for Trace Analysis,” *Anal. Chem.* **1993**, *55*, 54A–57A.
- Taylor, J. K. *Quality Assurance of Chemical Measurements*, Lewis Publishers: Chelsa, MI, 1987.

Additional information about the construction and use of control charts may be found in the following sources.

- Miller, J. C.; Miller, J. N. Statistics for Analytical Chemistry, 2nd Ed., Ellis Horwood Limited: Chichester, 1988.
- Ouchi, G. I. “Creating Control Charts with a Spreadsheet Program,” *LC•GC* **1993**, *11*, 416–423.
- Ouchi, G. I. “Creating Control Charts with a Spreadsheet Program,” *LC•GC* **1997**, *15*, 336–344.
- Simpson, J. M. “Spreadsheet Statistics,” *J. Chem. Educ.* **1994**, *71*, A88–A89.

Contributors

- David Harvey (DePauw University)

CHAPTER OVERVIEW

18: Back Matter

Index

Glossary

Sample Word 1 | Sample Definition 1

Detailed Licensing

Overview

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        - Table of Contents - *Undeclared*
        - Licensing - *Undeclared*
    - 2: Analytical Chemistry 2.0 (Harvey) - *CC BY-NC-SA 4.0*
        - 00: Front Matter - *Undeclared*
            - Table of Contents - *Undeclared*
        - 1: Front Matter - *Undeclared*
            - 1.1: TitlePage - *Undeclared*
            - 1.2: InfoPage - *Undeclared*
            - 1.3: Table of Contents - *Undeclared*
        - 2: Introduction to Analytical Chemistry - *CC BY-NC-SA 4.0*
            - 2.1: What is Analytical Chemistry? - *Undeclared*
            - 2.2: The Analytical Perspective - *CC BY-NC-SA 4.0*
            - 2.3: Common Analytical Problems - *CC BY-NC-SA 4.0*
            - 2.4: Introduction to Analytical Chemistry (Exercises) - *CC BY-NC-SA 4.0*
            - 2.5: Introduction to Analytical Chemistry (Summary) - *CC BY-NC-SA 4.0*
        - 3: Basic Tools of Analytical Chemistry - *CC BY-NC-SA 4.0*
            - 3.1: Measurements in Analytical Chemistry - *CC BY-NC-SA 4.0*
            - 3.2: Concentration - *CC BY-NC-SA 4.0*
            - 3.3: Stoichiometric Calculations - *CC BY-NC-SA 4.0*
            - 3.4: Basic Equipment - *CC BY-NC-SA 4.0*
            - 3.5: Preparing Solutions - *CC BY-NC-SA 4.0*
            - 3.6: Spreadsheets and Computational Software - *CC BY-NC-SA 4.0*
            - 3.7: The Laboratory Notebook - *CC BY-NC-SA 4.0*
            - 3.8: Basic Tools of Analytical Chemistry (Exercises) - *CC BY-NC-SA 4.0*
            - 3.9: Basic Tools of Analytical Chemistry (Summary) - *CC BY-NC-SA 4.0*
        - 4: The Vocabulary of Analytical Chemistry - *CC BY-NC-SA 4.0*
            - 4.1: Analysis, Determination, and Measurement - *CC BY-NC-SA 4.0*
            - 4.2: Techniques, Methods, Procedures, and Protocols - *CC BY-NC-SA 4.0*
            - 4.3: Classifying Analytical Techniques - *CC BY-NC-SA 4.0*
            - 4.4: Selecting an Analytical Method - *CC BY-NC-SA 4.0*
            - 4.5: Developing the Procedure - *CC BY-NC-SA 4.0*
            - 4.6: Protocols - *CC BY-NC-SA 4.0*
            - 4.7: The Importance of Analytical Methodology - *CC BY-NC-SA 4.0*
            - 4.8: The Vocabulary of Analytical Chemistry (Exercises) - *CC BY-NC-SA 4.0*
            - 4.9: The Vocabulary of Analytical Chemistry (Summary) - *CC BY-NC-SA 4.0*
        - 5: Evaluating Analytical Data - *CC BY-NC-SA 4.0*
            - 5.1: Characterizing Measurements and Results - *CC BY-NC-SA 4.0*
            - 5.2: Characterizing Experimental Errors - *CC BY-NC-SA 4.0*
            - 5.3: Propagation of Uncertainty - *CC BY-NC-SA 4.0*
            - 5.4: The Distribution of Measurements and Results - *CC BY-NC-SA 4.0*
            - 5.5: Statistical Analysis of Data - *CC BY-NC-SA 4.0*
            - 5.6: Statistical Methods for Normal Distributions - *CC BY-NC-SA 4.0*

  - 5.7: Detection Limits - *CC BY-NC-SA 4.0*
  - 5.8: Using Excel and R to Analyze Data - *CC BY-NC-SA 4.0*
  - 5.9: Evaluating Analytical Data (Exercises) - *CC BY-NC-SA 4.0*
  - 5.10: Evaluating Analytical Data (Summary) - *CC BY-NC-SA 4.0*
- 6: Standardizing Analytical Methods - *CC BY-NC-SA 4.0*
  - 6.1: Analytical Standards - *CC BY-NC-SA 4.0*
  - 6.2: Calibrating the Signal - *CC BY-NC-SA 4.0*
  - 6.3: Determining the Sensitivity - *CC BY-NC-SA 4.0*
  - 6.4: Linear Regression and Calibration Curves - *CC BY-NC-SA 4.0*
  - 6.5: Blank Corrections - *CC BY-NC-SA 4.0*
  - 6.6: Using Excel and R for a Regression Analysis - *CC BY-NC-SA 4.0*
  - 6.7: Standardizing Analytical Methods (Exercises) - *CC BY-NC-SA 4.0*
  - 6.8: Standardizing Analytical Methods (Summary) - *CC BY-NC-SA 4.0*
- 7: Equilibrium Chemistry - *CC BY-NC-SA 4.0*
  - 7.1: Reversible Reactions and Chemical Equilibria - *CC BY-NC-SA 4.0*
  - 7.2: Thermodynamics and Equilibrium Chemistry - *CC BY-NC-SA 4.0*
  - 7.3: Manipulating Equilibrium Constants - *CC BY-NC-SA 4.0*
  - 7.4: Equilibrium Constants for Chemical Reactions - *CC BY-NC-SA 4.0*
  - 7.5: Le Châtelier’s Principle - *CC BY-NC-SA 4.0*
  - 7.6: Ladder Diagrams - *CC BY-NC-SA 4.0*
  - 7.7: Solving Equilibrium Problems - *CC BY-NC-SA 4.0*
  - 7.8: Buffer Solutions - *CC BY-NC-SA 4.0*
  - 7.9: Activity Effects - *CC BY-NC-SA 4.0*
  - 7.10: Using Excel and R to Solve Equilibrium Problems - *CC BY-NC-SA 4.0*
  - 7.11: Some Final Thoughts on Equilibrium Calculations - *CC BY-NC-SA 4.0*
  - 7.12: Equilibrium Chemistry (Exercises) - *CC BY-NC-SA 4.0*
  - 7.13: Equilibrium Chemistry (Summary) - *CC BY-NC-SA 4.0*
- 8: Collecting and Preparing Samples - *CC BY-NC-SA 4.0*
  - 8.1: The Importance of Sampling - *CC BY-NC-SA 4.0*
  - 8.2: Designing a Sampling Plan - *CC BY-NC-SA 4.0*
  - 8.3: Implementing the Sampling Plan - *CC BY-NC-SA 4.0*
  - 8.4: Separating the Analyte from Interferents - *CC BY-NC-SA 4.0*
  - 8.5: General Theory of Separation Efficiency - *CC BY-NC-SA 4.0*
  - 8.6: Classifying Separation Techniques - *CC BY-NC-SA 4.0*
  - 8.7: Liquid–Liquid Extractions - *CC BY-NC-SA 4.0*
  - 8.8: Separation Versus Preconcentration - *CC BY-NC-SA 4.0*
  - 8.9: Collecting and Preparing Samples (Exercises) - *CC BY-NC-SA 4.0*
  - 8.10: Collecting and Preparing Samples (Summary) - *CC BY-NC-SA 4.0*
- 9: Gravimetric Methods - *CC BY-NC-SA 4.0*
  - 9.1: Overview of Gravimetric Methods - *CC BY-NC-SA 4.0*
  - 9.2: Precipitation Gravimetry - *CC BY-NC-SA 4.0*
  - 9.3: Volatilization Gravimetry - *CC BY-NC-SA 4.0*
  - 9.4: Particulate Gravimetry - *CC BY-NC-SA 4.0*
  - 9.5: Gravimetric Methods (Exercises) - *CC BY-NC-SA 4.0*
  - 9.6: Gravimetric Methods (Summary) - *CC BY-NC-SA 4.0*
- 10: Titrimetric Methods - *CC BY-NC-SA 4.0*
  - 10.1: Overview of Titrimetry - *CC BY-NC-SA 4.0*
  - 10.2: Acid–Base Titrations - *CC BY-NC-SA 4.0*
  - 10.3: Complexation Titrations - *CC BY-NC-SA 4.0*
  - 10.4: Redox Titrations - *CC BY-NC-SA 4.0*
  - 10.5: Precipitation Titrations - *CC BY-NC-SA 4.0*
  - 10.6: Titrimetric Methods (Exercises) - *CC BY-NC-SA 4.0*
  - 10.7: Titrimetric Methods (Summary) - *CC BY-NC-SA 4.0*
- 11: Spectroscopic Methods - *CC BY-NC-SA 4.0*
  - 11.1: Overview of Spectroscopy - *CC BY-NC-SA 4.0*
  - 11.2: Spectroscopy Based on Absorption - *CC BY-NC-SA 4.0*
  - 11.03: UV - *Undeclared*
    - 11.3: UV/Vis and IR Spectroscopy - *CC BY-NC-SA 4.0*
  - 11.4: Atomic Absorption Spectroscopy - *CC BY-NC-SA 4.0*
  - 11.5: Emission Spectroscopy - *CC BY-NC-SA 4.0*
  - 11.6: Photoluminescence Spectroscopy - *CC BY-NC-SA 4.0*
  - 11.7: Atomic Emission Spectroscopy - *CC BY-NC-SA 4.0*