

ANALYTICAL CHEMISTRY



Analytical Chemistry

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TABLE OF CONTENTS

Licensing

2: Analytical Chemistry 2.0 (Harvey)

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

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

- 11.1: Overview of Spectroscopy
- 11.2: Spectroscopy Based on Absorption
- 11.03: UV
 - 11.3: UV/Vis and IR Spectroscopy
- 11.4: Atomic Absorption Spectroscopy
- 11.5: Emission Spectroscopy
- 11.6: Photoluminescence Spectroscopy
- 11.7: Atomic Emission Spectroscopy
- 11.8: Spectroscopy Based on Scattering
- 11.9: Spectroscopic Methods (Exercises)
- 11.10: Spectroscopic Methods (Summary)
- 12: Electrochemical Methods
 - 12.1: Overview of Electrochemistry
 - 12.2: Potentiometric Methods
 - 12.3: Coulometric Methods
 - 12.4: Voltammetric Methods
 - 12.5: Electrochemical Methods (Exercises)
 - 12.6: Electrochemical Methods (Summary)
- 13: Chromatographic
 - 13.1: 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
 - 13.6: Other Forms of Liquid Chromatography
 - 13.7: Electrophoresis
 - 13.8: Chromatographic and Electrophoretic Methods (Exercises)
 - 13.9: Chromatographic and Electrophoretic Methods (Summary)
- 14: Kinetic Methods
 - 14.1: Kinetic Methods Versus Equilibrium Methods
 - 14.2: Chemical Kinetics
 - 14.3: Radiochemistry
 - 14.4: Flow Injection Analysis
 - 14.5: Kinetic Methods (Exercises)
 - 14.6: Kinetic Methods (Summary)
- 15: Developing a Standard Method
 - 15.1: 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
 - 15.5: Developing a Standard Method (Exercises)
 - 15.6: Developing a Standard Method (Summary)
- 16: Quality Assurance
 - 16.1: The Analytical Perspective—Revisited
 - 16.2: Quality Control
 - 16.3: Quality Assessment
 - 16.4: Evaluating Quality Assurance Data
 - 16.5: Quality Assurance (Exercises)
- 17: Additional Resources
- 18: Back Matter

[Index](#)

[Glossary](#)

[Detailed Licensing](#)

Licensing

A detailed breakdown of this resource's licensing can be found in [Back Matter/Detailed Licensing](#).

00: Front Matter

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

TABLE OF CONTENTS

00: Front Matter

- Table of Contents

1: Front Matter

- 1.1: TitlePage
- 1.2: InfoPage
- 1.3: Table of Contents

2: Introduction to Analytical Chemistry

- 2.1: What is Analytical Chemistry?
- 2.2: The Analytical Perspective
- 2.3: Common Analytical Problems
- 2.4: Introduction to Analytical Chemistry (Exercises)
- 2.5: Introduction to Analytical Chemistry (Summary)

3: Basic Tools of Analytical Chemistry

- 3.1: Measurements in Analytical Chemistry
- 3.2: Concentration
- 3.3: Stoichiometric Calculations
- 3.4: Basic Equipment
- 3.5: Preparing Solutions
- 3.6: Spreadsheets and Computational Software
- 3.7: The Laboratory Notebook
- 3.8: Basic Tools of Analytical Chemistry (Exercises)
- 3.9: Basic Tools of Analytical Chemistry (Summary)

4: The Vocabulary of Analytical Chemistry

- 4.1: Analysis, Determination, and Measurement
- 4.2: Techniques, Methods, Procedures, and Protocols
- 4.3: Classifying Analytical Techniques
- 4.4: Selecting an Analytical Method
- 4.5: Developing the Procedure
- 4.6: Protocols
- 4.7: The Importance of Analytical Methodology
- 4.8: The Vocabulary of Analytical Chemistry (Exercises)
- 4.9: The Vocabulary of Analytical Chemistry (Summary)

5: Evaluating Analytical Data

- 5.1: Characterizing Measurements and Results
- 5.2: Characterizing Experimental Errors
- 5.3: Propagation of Uncertainty
- 5.4: The Distribution of Measurements and Results
- 5.5: Statistical Analysis of Data
- 5.6: Statistical Methods for Normal Distributions
- 5.7: Detection Limits

- 5.8: Using Excel and R to Analyze Data
- 5.9: Evaluating Analytical Data (Exercises)
- 5.10: Evaluating Analytical Data (Summary)

6: Standardizing Analytical Methods

- 6.1: Analytical Standards
- 6.2: Calibrating the Signal
- 6.3: Determining the Sensitivity
- 6.4: Linear Regression and Calibration Curves
- 6.5: Blank Corrections
- 6.6: Using Excel and R for a Regression Analysis
- 6.7: Standardizing Analytical Methods (Exercises)
- 6.8: Standardizing Analytical Methods (Summary)

7: Equilibrium Chemistry

- 7.1: Reversible Reactions and Chemical Equilibria
- 7.2: Thermodynamics and Equilibrium Chemistry
- 7.3: Manipulating Equilibrium Constants
- 7.4: Equilibrium Constants for Chemical Reactions
- 7.5: Le Châtelier's Principle
- 7.6: Ladder Diagrams
- 7.7: Solving Equilibrium Problems
- 7.8: Buffer Solutions
- 7.9: Activity Effects
- 7.10: Using Excel and R to Solve Equilibrium Problems
- 7.11: Some Final Thoughts on Equilibrium Calculations
- 7.12: Equilibrium Chemistry (Exercises)
- 7.13: Equilibrium Chemistry (Summary)

8: Collecting and Preparing Samples

- 8.1: The Importance of Sampling
- 8.2: Designing a Sampling Plan
- 8.3: Implementing the Sampling Plan
- 8.4: Separating the Analyte from Interferents
- 8.5: General Theory of Separation Efficiency
- 8.6: Classifying Separation Techniques
- 8.7: Liquid–Liquid Extractions
- 8.8: Separation Versus Preconcentration
- 8.9: Collecting and Preparing Samples (Exercises)
- 8.10: Collecting and Preparing Samples (Summary)

9: Gravimetric Methods

- 9.1: Overview of Gravimetric Methods
- 9.2: Precipitation Gravimetry
- 9.3: Volatilization Gravimetry
- 9.4: Particulate Gravimetry
- 9.5: Gravimetric Methods (Exercises)
- 9.6: Gravimetric Methods (Summary)

10: Titrimetric Methods

- 10.1: Overview of Titrimetry
- 10.2: Acid–Base Titrations
- 10.3: Complexation Titrations
- 10.4: Redox Titrations
- 10.5: Precipitation Titrations
- 10.6: Titrimetric Methods (Exercises)
- 10.7: Titrimetric Methods (Summary)

11: Spectroscopic Methods

- 11.1: Overview of Spectroscopy
- 11.2: Spectroscopy Based on Absorption
- 11.03: UV
 - 11.3: UV/Vis and IR Spectroscopy
- 11.4: Atomic Absorption Spectroscopy
- 11.5: Emission Spectroscopy
- 11.6: Photoluminescence Spectroscopy
- 11.7: Atomic Emission Spectroscopy
- 11.8: Spectroscopy Based on Scattering
- 11.9: Spectroscopic Methods (Exercises)
- 11.10: Spectroscopic Methods (Summary)

12: Electrochemical Methods

- 12.1: Overview of Electrochemistry
- 12.2: Potentiometric Methods
- 12.3: Coulometric Methods
- 12.4: Voltammetric Methods
- 12.5: Electrochemical Methods (Exercises)
- 12.6: Electrochemical Methods (Summary)

13: Chromatographic

- 13.1: 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
- 13.6: Other Forms of Liquid Chromatography
- 13.7: Electrophoresis
- 13.8: Chromatographic and Electrophoretic Methods (Exercises)
- 13.9: Chromatographic and Electrophoretic Methods (Summary)

14: Kinetic Methods

- 14.1: Kinetic Methods Versus Equilibrium Methods
- 14.2: Chemical Kinetics
- 14.3: Radiochemistry
- 14.4: Flow Injection Analysis
- 14.5: Kinetic Methods (Exercises)
- 14.6: Kinetic Methods (Summary)

15: Developing a Standard Method

- 15.1: 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
- 15.5: Developing a Standard Method (Exercises)
- 15.6: Developing a Standard Method (Summary)

16: Quality Assurance

- 16.1: The Analytical Perspective—Revisited
- 16.2: Quality Control
- 16.3: Quality Assessment
- 16.4: Evaluating Quality Assurance Data
- 16.5: Quality Assurance (Exercises)

17: Additional Resources

18: Back Matter

CHAPTER OVERVIEW

1: Front Matter

[1.1: TitlePage](#)

[1.2: InfoPage](#)

[1.3: Table of Contents](#)

DePauw University
Analytical Chemistry 2.1

David Harvey

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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.

- **1: Introduction to Analytical Chemistry**

- [1.1: What is Analytical Chemistry?](#)
- [1.2: The Analytical Perspective](#)
- [1.3: Common Analytical Problems](#)
- [1.E: Introduction to Analytical Chemistry \(Exercises\)](#)
- [1.S: Introduction to Analytical Chemistry \(Summary\)](#)

- **2: 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.

- [2.1: Measurements in Analytical Chemistry](#)
- [2.2: Concentration](#)
- [2.3: Stoichiometric Calculations](#)
- [2.4: Basic Equipment](#)
- [2.5: Preparing Solutions](#)
- [2.6: Spreadsheets and Computational Software](#)
- [2.7: The Laboratory Notebook](#)
- [2.E: Basic Tools of Analytical Chemistry \(Exercises\)](#)
- [2.S: Basic Tools of Analytical Chemistry \(Summary\)](#)

- **3: 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.

- [3.1: Analysis, Determination, and Measurement](#)
- [3.2: Techniques, Methods, Procedures, and Protocols](#)
- [3.3: Classifying Analytical Techniques](#)
- [3.4: Selecting an Analytical Method](#)
- [3.5: Developing the Procedure](#)
- [3.6: Protocols](#)
- [3.7: The Importance of Analytical Methodology](#)
- [3.E: The Vocabulary of Analytical Chemistry \(Exercises\)](#)
- [3.S: The Vocabulary of Analytical Chemistry \(Summary\)](#)

- **4: 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.

- [4.1: Characterizing Measurements and Results](#)
- [4.2: Characterizing Experimental Errors](#)
- [4.3: Propagation of Uncertainty](#)

- 4.4: The Distribution of Measurements and Results
- 4.5: Statistical Analysis of Data
- 4.6: Statistical Methods for Normal Distributions
- 4.7: Detection Limits
- 4.8: Using Excel and R to Analyze Data
- 4.E: Evaluating Analytical Data (Exercises)
- 4.S: Evaluating Analytical Data (Summary)

- **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 $S_{\text{total}} = k_A C_A + S_{\text{reag}}$ 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} , which is the subject of this chapter.

- 5.1: Analytical Standards
- 5.2: Calibrating the Signal
- 5.3: Determining the Sensitivity
- 5.4: Linear Regression and Calibration Curves
- 5.5: Blank Corrections
- 5.6: Using Excel and R for a Regression Analysis
- 5.E: Standardizing Analytical Methods (Exercises)
- 5.S: Standardizing Analytical Methods (Summary)

- **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.

- 6.1: Reversible Reactions and Chemical Equilibria
- 6.2: Thermodynamics and Equilibrium Chemistry
- 6.3: Manipulating Equilibrium Constants
- 6.4: Equilibrium Constants for Chemical Reactions
- 6.5: Le Châtelier's Principle
- 6.6: Ladder Diagrams
- 6.7: Solving Equilibrium Problems
- 6.8: Buffer Solutions
- 6.9: Activity Effects
- 6.10: Using Excel and R to Solve Equilibrium Problems
- 6.11: Some Final Thoughts on Equilibrium Calculations
- 6.E: Equilibrium Chemistry (Exercises)
- 6.S: Equilibrium Chemistry (Summary)

- **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.

- 7.1: The Importance of Sampling
- 7.2: Designing a Sampling Plan
- 7.3: Implementing the Sampling Plan
- 7.4: Separating the Analyte from Interferents
- 7.5: General Theory of Separation Efficiency

- 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.

- [15.1: The Analytical Perspective—Revisited](#)
- [15.2: Quality Control](#)
- [15.3: Quality Assessment](#)
- [15.4: Evaluating Quality Assurance Data](#)
- [15.E: Quality Assurance \(Exercises\)](#)

- [Additional Resources](#)

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

- [2.1: What is Analytical Chemistry?](#)
- [2.2: The Analytical Perspective](#)
- [2.3: Common Analytical Problems](#)
- [2.4: Introduction to Analytical Chemistry \(Exercises\)](#)
- [2.5: Introduction to Analytical Chemistry \(Summary\)](#)

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).

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2.1: What is Analytical Chemistry?

1.1: What is Analytical Chemistry?

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2.2: The Analytical Perspective

1.2: The Analytical Perspective

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2.3: Common Analytical Problems

1.3: Common Analytical Problems

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2.4: Introduction to Analytical Chemistry (Exercises)

1.E: Introduction to Analytical Chemistry (Exercises)

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2.5: Introduction to Analytical Chemistry (Summary)

[1.S: Introduction to Analytical Chemistry \(Summary\)](#)

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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.

[3.1: Measurements in Analytical Chemistry](#)

[3.2: Concentration](#)

[3.3: Stoichiometric Calculations](#)

[3.4: Basic Equipment](#)

[3.5: Preparing Solutions](#)

[3.6: Spreadsheets and Computational Software](#)

[3.7: The Laboratory Notebook](#)

[3.8: Basic Tools of Analytical Chemistry \(Exercises\)](#)

[3.9: Basic Tools of Analytical Chemistry \(Summary\)](#)

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](#))

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3.1: Measurements in Analytical Chemistry

2.1: Measurements in Analytical Chemistry

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3.2: Concentration

2.2: Concentration

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3.3: Stoichiometric Calculations

2.3: Stoichiometric Calculations

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3.4: Basic Equipment

2.4: Basic Equipment

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3.5: Preparing Solutions

2.5: Preparing Solutions

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3.6: Spreadsheets and Computational Software

2.6: Spreadsheets and Computational Software

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3.7: The Laboratory Notebook

2.7: The Laboratory Notebook

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3.8: Basic Tools of Analytical Chemistry (Exercises)

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

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3.9: Basic Tools of Analytical Chemistry (Summary)

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

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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.

- [4.1: Analysis, Determination, and Measurement](#)
- [4.2: Techniques, Methods, Procedures, and Protocols](#)
- [4.3: Classifying Analytical Techniques](#)
- [4.4: Selecting an Analytical Method](#)
- [4.5: Developing the Procedure](#)
- [4.6: Protocols](#)
- [4.7: The Importance of Analytical Methodology](#)
- [4.8: The Vocabulary of Analytical Chemistry \(Exercises\)](#)
- [4.9: The Vocabulary of Analytical Chemistry \(Summary\)](#)

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.

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4.1: Analysis, Determination, and Measurement

3.1: Analysis, Determination, and Measurement

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4.2: Techniques, Methods, Procedures, and Protocols

3.2: Techniques, Methods, Procedures, and Protocols

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4.3: Classifying Analytical Techniques

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4.4: Selecting an Analytical Method

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4.5: Developing the Procedure

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4.6: Protocols

3.6: Protocols

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4.7: The Importance of Analytical Methodology

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4.8: The Vocabulary of Analytical Chemistry (Exercises)

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

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4.9: The Vocabulary of Analytical Chemistry (Summary)

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

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CHAPTER OVERVIEW

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.

- [5.1: Characterizing Measurements and Results](#)
- [5.2: Characterizing Experimental Errors](#)
- [5.3: Propagation of Uncertainty](#)
- [5.4: The Distribution of Measurements and Results](#)
- [5.5: Statistical Analysis of Data](#)
- [5.6: Statistical Methods for Normal Distributions](#)
- [5.7: Detection Limits](#)
- [5.8: Using Excel and R to Analyze Data](#)
- [5.9: Evaluating Analytical Data \(Exercises\)](#)
- [5.10: Evaluating Analytical Data \(Summary\)](#)

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) .

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5.1: Characterizing Measurements and Results

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5.2: Characterizing Experimental Errors

4.2: Characterizing Experimental Errors

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5.3: Propagation of Uncertainty

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5.4: The Distribution of Measurements and Results

4.4: The Distribution of Measurements and Results

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5.5: Statistical Analysis of Data

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5.6: Statistical Methods for Normal Distributions

4.6: Statistical Methods for Normal Distributions

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5.7: Detection Limits

4.7: Detection Limits

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5.8: Using Excel and R to Analyze Data

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5.9: Evaluating Analytical Data (Exercises)

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5.10: Evaluating Analytical Data (Summary)

4.S: Evaluating Analytical Data (Summary)

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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

$$S_{\text{total}} = k_A n_A + S_{\text{reag}} \quad \text{or} \quad S_{\text{total}} = k_A C_A + S_{\text{reag}} \quad (6.1)$$

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.

[6.1: Analytical Standards](#)

[6.2: Calibrating the Signal](#)

[6.3: Determining the Sensitivity](#)

[6.4: Linear Regression and Calibration Curves](#)

[6.5: Blank Corrections](#)

[6.6: Using Excel and R for a Regression Analysis](#)

[6.7: Standardizing Analytical Methods \(Exercises\)](#)

[6.8: Standardizing Analytical Methods \(Summary\)](#)

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, $\hat{y} = b_0 + b_1x$. The smaller the total residual error (equation 5.16), the better the fit of the straight-line to the data

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6.1: Analytical Standards

5.1: Analytical Standards

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6.2: Calibrating the Signal

5.2: Calibrating the Signal

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6.3: Determining the Sensitivity

5.3: Determining the Sensitivity

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6.4: Linear Regression and Calibration Curves

5.4: Linear Regression and Calibration Curves

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6.5: Blank Corrections

5.5: Blank Corrections

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6.6: Using Excel and R for a Regression Analysis

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6.7: Standardizing Analytical Methods (Exercises)

5.E: Standardizing Analytical Methods (Exercises)

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6.8: Standardizing Analytical Methods (Summary)

5.S: Standardizing Analytical Methods (Summary)

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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.

- 7.1: Reversible Reactions and Chemical Equilibria
- 7.2: Thermodynamics and Equilibrium Chemistry
- 7.3: Manipulating Equilibrium Constants
- 7.4: Equilibrium Constants for Chemical Reactions
- 7.5: Le Châtelier’s Principle
- 7.6: Ladder Diagrams
- 7.7: Solving Equilibrium Problems
- 7.8: Buffer Solutions
- 7.9: Activity Effects
- 7.10: Using Excel and R to Solve Equilibrium Problems
- 7.11: Some Final Thoughts on Equilibrium Calculations
- 7.12: Equilibrium Chemistry (Exercises)
- 7.13: Equilibrium Chemistry (Summary)

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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7.1: Reversible Reactions and Chemical Equilibria

6.01: Reversible Reactions and Chemical Equilibria

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7.2: Thermodynamics and Equilibrium Chemistry

6.02: Thermodynamics and Equilibrium Chemistry

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7.3: Manipulating Equilibrium Constants

6.03: Manipulating Equilibrium Constants

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7.4: Equilibrium Constants for Chemical Reactions

6.04: Equilibrium Constants for Chemical Reactions

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7.5: Le Châtelier's Principle

6.05: Le Châtelier's Principle

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7.6: Ladder Diagrams

6.06: Ladder Diagrams

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7.7: Solving Equilibrium Problems

6.07: Solving Equilibrium Problems

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7.8: Buffer Solutions

6.08: Buffer Solutions

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7.9: Activity Effects

[6.09: Activity Effects](#)

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7.10: Using Excel and R to Solve Equilibrium Problems

6.10: Using Excel and R to Solve Equilibrium Problems

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7.11: Some Final Thoughts on Equilibrium Calculations

6.11: Some Final Thoughts on Equilibrium Calculations

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7.12: Equilibrium Chemistry (Exercises)

6.E: Equilibrium Chemistry (Exercises)

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7.13: Equilibrium Chemistry (Summary)

[6.S: Equilibrium Chemistry \(Summary\)](#)

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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.

- [8.1: The Importance of Sampling](#)
- [8.2: Designing a Sampling Plan](#)
- [8.3: Implementing the Sampling Plan](#)
- [8.4: Separating the Analyte from Interferents](#)
- [8.5: General Theory of Separation Efficiency](#)
- [8.6: Classifying Separation Techniques](#)
- [8.7: Liquid–Liquid Extractions](#)
- [8.8: Separation Versus Preconcentration](#)
- [8.9: Collecting and Preparing Samples \(Exercises\)](#)
- [8.10: Collecting and Preparing Samples \(Summary\)](#)

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

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8.1: The Importance of Sampling

7.1: The Importance of Sampling

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8.2: Designing a Sampling Plan

7.2: Designing a Sampling Plan

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8.3: Implementing the Sampling Plan

7.3: Implementing the Sampling Plan

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8.4: Separating the Analyte from Interferents

7.4: Separating the Analyte from Interferents

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8.5: General Theory of Separation Efficiency

7.5: General Theory of Separation Efficiency

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8.6: Classifying Separation Techniques

7.6: Classifying Separation Techniques

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8.7: Liquid–Liquid Extractions

7.7: Liquid–Liquid Extractions

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8.8: Separation Versus Preconcentration

7.8: Separation Versus Preconcentration

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8.9: Collecting and Preparing Samples (Exercises)

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8.10: Collecting and Preparing Samples (Summary)

[7.S: Collecting and Preparing Samples \(Summary\)](#)

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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.

[9.1: Overview of Gravimetric Methods](#)

[9.2: Precipitation Gravimetry](#)

[9.3: Volatilization Gravimetry](#)

[9.4: Particulate Gravimetry](#)

[9.5: Gravimetric Methods \(Exercises\)](#)

[9.6: Gravimetric Methods \(Summary\)](#)

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

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9.1: Overview of Gravimetric Methods

8.1: Overview of Gravimetric Methods

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9.2: Precipitation Gravimetry

8.2: Precipitation Gravimetry

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9.3: Volatilization Gravimetry

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9.4: Particulate Gravimetry

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9.5: Gravimetric Methods (Exercises)

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9.6: Gravimetric Methods (Summary)

8.S: Gravimetric Methods (Summary)

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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.

[10.1: Overview of Titrimetry](#)

[10.2: Acid–Base Titrations](#)

[10.3: Complexation Titrations](#)

[10.4: Redox Titrations](#)

[10.5: Precipitation Titrations](#)

[10.6: Titrimetric Methods \(Exercises\)](#)

[10.7: Titrimetric Methods \(Summary\)](#)

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)).

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10.1: Overview of Titrimetry

9.1: Overview of Titrimetry

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10.2: Acid–Base Titrations

9.2: Acid–Base Titrations

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10.3: Complexation Titrations

9.3: Complexation Titrations

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10.4: Redox Titrations

9.4: Redox Titrations

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10.5: Precipitation Titrations

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10.6: Titrimetric Methods (Exercises)

9.E: Titrimetric Methods (Exercises)

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10.7: Titrimetric Methods (Summary)

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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.

[11.1: Overview of Spectroscopy](#)

[11.2: Spectroscopy Based on Absorption](#)

[11.03: UV](#)

[11.3: UV/Vis and IR Spectroscopy](#)

[11.4: Atomic Absorption Spectroscopy](#)

[11.5: Emission Spectroscopy](#)

[11.6: Photoluminescence Spectroscopy](#)

[11.7: Atomic Emission Spectroscopy](#)

[11.8: Spectroscopy Based on Scattering](#)

[11.9: Spectroscopic Methods \(Exercises\)](#)

[11.10: Spectroscopic Methods \(Summary\)](#)

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11.1: Overview of Spectroscopy

10.1: Overview of Spectroscopy

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11.2: Spectroscopy Based on Absorption

10.2: Spectroscopy Based on Absorption

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11.03: UV

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11.3: UV/Vis and IR Spectroscopy

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11.4: Atomic Absorption Spectroscopy

[10.4: Atomic Absorption Spectroscopy](#)

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11.5: Emission Spectroscopy

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11.6: Photoluminescence Spectroscopy

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11.7: Atomic Emission Spectroscopy

10.7: Atomic Emission Spectroscopy

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11.8: Spectroscopy Based on Scattering

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11.9: Spectroscopic Methods (Exercises)

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11.10: Spectroscopic Methods (Summary)

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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.

[12.1: Overview of Electrochemistry](#)

[12.2: Potentiometric Methods](#)

[12.3: Coulometric Methods](#)

[12.4: Voltammetric Methods](#)

[12.5: Electrochemical Methods \(Exercises\)](#)

[12.6: Electrochemical Methods \(Summary\)](#)

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).

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12.1: Overview of Electrochemistry

[11.1: Overview of Electrochemistry](#)

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12.2: Potentiometric Methods

[11.2: Potentiometric Methods](#)

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12.3: Coulometric Methods

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12.4: Voltammetric Methods

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12.5: Electrochemical Methods (Exercises)

[11.E: Electrochemical Methods \(Exercises\)](#)

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12.6: Electrochemical Methods (Summary)

[11.S: Electrochemical Methods \(Summary\)](#)

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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.

[13.1: 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](#)

[13.6: Other Forms of Liquid Chromatography](#)

[13.7: Electrophoresis](#)

[13.8: Chromatographic and Electrophoretic Methods \(Exercises\)](#)

[13.9: Chromatographic and Electrophoretic Methods \(Summary\)](#)

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

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13.1: Overview of Analytical Separations

12.1: Overview of Analytical Separations

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13.2: General Theory of Column Chromatography

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13.3: Optimizing Chromatographic Separations

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13.4: Gas Chromatography

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13.5: High-Performance Liquid Chromatography

[12.5: High-Performance Liquid Chromatography](#)

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13.6: Other Forms of Liquid Chromatography

[12.6: Other Forms of Liquid Chromatography](#)

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13.7: Electrophoresis

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13.8: Chromatographic and Electrophoretic Methods (Exercises)

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13.9: Chromatographic and Electrophoretic Methods (Summary)

[12.S: Chromatographic and Electrophoretic Methods \(Summary\)](#)

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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.

[14.1: Kinetic Methods Versus Equilibrium Methods](#)

[14.2: Chemical Kinetics](#)

[14.3: Radiochemistry](#)

[14.4: Flow Injection Analysis](#)

[14.5: Kinetic Methods \(Exercises\)](#)

[14.6: Kinetic Methods \(Summary\)](#)

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.

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14.1: Kinetic Methods Versus Equilibrium Methods

13.1: Kinetic Methods Versus Equilibrium Methods

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14.2: Chemical Kinetics

[13.2: Chemical Kinetics](#)

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14.3: Radiochemistry

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14.4: Flow Injection Analysis

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14.5: Kinetic Methods (Exercises)

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14.6: Kinetic Methods (Summary)

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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.

[15.1: 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](#)

[15.5: Developing a Standard Method \(Exercises\)](#)

[15.6: Developing a Standard Method \(Summary\)](#)

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15.1: Optimizing the Experimental Procedure

14.1: Optimizing the Experimental Procedure

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15.2: Verifying the Method

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15.3: Validating the Method as a Standard Method

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15.4: Using Excel and R for an Analysis of Variance

14.4: Using Excel and R for an Analysis of Variance

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15.5: Developing a Standard Method (Exercises)

[14.E: Developing a Standard Method \(Exercises\)](#)

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15.6: Developing a Standard Method (Summary)

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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.

[16.1: The Analytical Perspective—Revisited](#)

[16.2: Quality Control](#)

[16.3: Quality Assessment](#)

[16.4: Evaluating Quality Assurance Data](#)

[16.5: Quality Assurance \(Exercises\)](#)

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

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16.1: The Analytical Perspective—Revisited

15.1: The Analytical Perspective—Revisited

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16.2: Quality Control

15.2: Quality Control

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16.3: Quality Assessment

15.3: Quality Assessment

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16.4: Evaluating Quality Assurance Data

15.4: Evaluating Quality Assurance Data

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16.5: Quality Assurance (Exercises)

15.E: Quality Assurance (Exercises)

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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.

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- 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.
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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.
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- 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.
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- 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, Intercepts, 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 from '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 from '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 discuss 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 $y = \alpha x$ 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 discuss 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 their 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.; Høim "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.; Høim "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 K_{sp}-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 $\text{pH} \approx (\text{pK}_{\text{a}1} + \text{pK}_{\text{a}2})/2$ 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.
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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](#)).
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- "Guide to Solid Phase Extraction," Bulletin 910, Sigma-Aldrich, 1998.
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- *Microwave-Enhanced Chemistry: Fundamentals, Sample Preparation, and Applications*, Kingston, H. M.; Haswell, S. J., eds.; American Chemical Society: Washington, D.C., 1997.
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Chapter 8

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

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Chapter 9

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Acid–base titrimetry

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Complexation Titrimetry

- Ceretti, H.; Hughes, E. A.; Zalts, A. "The Softening of Hard Water and Complexometric Titrations," *J. Chem. Educ.* **1999**, 76, 1420–1421.
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Precipitation Titrimetry

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

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- 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.

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- 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

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Chapter 11

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Chapter 12

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Chapter 13

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

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Contributors

- [David Harvey \(DePauw University\)](#)

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CHAPTER OVERVIEW

18: Back Matter

Index

A

abbreviation

[3.1: Measurements in Analytical Chemistry](#)

absorbance

[4.3: Classifying Analytical Techniques](#)
[11.1: Overview of Spectroscopy](#)
[11.2: Spectroscopy Based on Absorption](#)
[15.1: Optimizing the Experimental Procedure](#)

Absorbance Spectra

[11.2: Spectroscopy Based on Absorption](#)

absorbance spectrum

[11.1: Overview of Spectroscopy](#)

absorption

[11.2: Spectroscopy Based on Absorption](#)
[11.6: Photoluminescence Spectroscopy](#)

absorption spectrophotometers

[11.4: Atomic Absorption Spectroscopy](#)

absorption spectroscopy

[11.2: Spectroscopy Based on Absorption](#)
[11.4: Atomic Absorption Spectroscopy](#)

absorption spectrum

[13.5: High-Performance Liquid Chromatography](#)

absorptivity

[11.2: Spectroscopy Based on Absorption](#)
[15.1: Optimizing the Experimental Procedure](#)

accuracy

[4.4: Selecting an Analytical Method](#)
[5.2: Characterizing Experimental Errors](#)
[11.3: UV/Vis and IR Spectroscopy](#)
[11.4: Atomic Absorption Spectroscopy](#)
[11.7: Atomic Emission Spectroscopy](#)
[15.2: Verifying the Method](#)
[15.3: Validating the Method as a Standard Method](#)

acetic acid

[7.6: Ladder Diagrams](#)

acid

[7.4: Equilibrium Constants for Chemical Reactions](#)
[7.7: Solving Equilibrium Problems](#)
[7.8: Buffer Solutions](#)

acid–base buffer

[7.8: Buffer Solutions](#)

acid–base titrations

[10.2: Acid–Base Titrations](#)

acid–base titrimetry

[10.2: Acid–Base Titrations](#)
[10.4: Redox Titrations](#)

acidity

[10.2: Acid–Base Titrations](#)

activity

[7.9: Activity Effects](#)
[12.2: Potentiometric Methods](#)

activity coefficients

[7.9: Activity Effects](#)

activity effects

[7.9: Activity Effects](#)

adjusted retention time

[13.2: General Theory of Column Chromatography](#)

adsorption chromatography

[13.1: Overview of Analytical Separations](#)

algorithm

[15.1: Optimizing the Experimental Procedure](#)

Alkalinity

[10.2: Acid–Base Titrations](#)

alpha particle

[14.3: Radiochemistry](#)

alternative hypotheses

[5.5: Statistical Analysis of Data](#)

alternative hypothesis

[5.5: Statistical Analysis of Data](#)

amalgam

[12.4: Voltammetric Methods](#)

amperometry

[12.1: Overview of Electrochemistry](#)
[12.4: Voltammetric Methods](#)
[13.5: High-Performance Liquid Chromatography](#)

amphiprotic

[7.4: Equilibrium Constants for Chemical Reactions](#)

Amphiprotic Species

[7.4: Equilibrium Constants for Chemical Reactions](#)

amplitude

[11.1: Overview of Spectroscopy](#)

analysis

[4.1: Analysis, Determination, and Measurement](#)
[14.1: Kinetic Methods Versus Equilibrium Methods](#)

analysis of standards

[16.3: Quality Assessment](#)

analysis of variance

[15.3: Validating the Method as a Standard Method](#)
[15.4: Using Excel and R for an Analysis of Variance](#)

analyst

[15.2: Verifying the Method](#)

analysts

[15.3: Validating the Method as a Standard Method](#)

analyte

[4.1: Analysis, Determination, and Measurement](#)
[8.5: General Theory of Separation Efficiency](#)
[9.2: Precipitation Gravimetry](#)
[9.4: Particulate Gravimetry](#)
[10.1: Overview of Titrimetry](#)
[11.3: UV/Vis and IR Spectroscopy](#)
[11.4: Atomic Absorption Spectroscopy](#)
[11.5: Emission Spectroscopy](#)
[12.1: Overview of Electrochemistry](#)
[12.2: Potentiometric Methods](#)
[12.3: Coulometric Methods](#)
[13.1: Overview of Analytical Separations](#)
[13.4: Gas Chromatography](#)
[15.1: Optimizing the Experimental Procedure](#)
[16.3: Quality Assessment](#)

Analyte's Concentration

[13.4: Gas Chromatography](#)

analytes

[11.6: Photoluminescence Spectroscopy](#)
[13.7: Electrophoresis](#)

analytical

[14.1: Kinetic Methods Versus Equilibrium Methods](#)

Analytical Approach

[2.2: The Analytical Perspective](#)

analytical balance

[3.4: Basic Equipment](#)

analytical calculations

[3.3: Stoichiometric Calculations](#)

analytical chemistry

[2.1: What is Analytical Chemistry?](#)
[3.1: Measurements in Analytical Chemistry](#)
[12.1: Overview of Electrochemistry](#)

analytical chemists

[2.2: The Analytical Perspective](#)

Analytical Columns

[13.5: High-Performance Liquid Chromatography](#)

analytical data

[16.1: The Analytical Perspective—Revisited](#)

analytical method

[2.1: What is Analytical Chemistry?](#)
[4.4: Selecting an Analytical Method](#)
[5.5: Statistical Analysis of Data](#)
[6.1: Analytical Standards](#)
[6.3: Determining the Sensitivity](#)
[7.6: Ladder Diagrams](#)
[8.4: Separating the Analyte from Interferents](#)
[11.3: UV/Vis and IR Spectroscopy](#)
[15.3: Validating the Method as a Standard Method](#)
[16.4: Evaluating Quality Assurance Data](#)

analytical methodology

[4.7: The Importance of Analytical Methodology](#)

analytical methods

[4.4: Selecting an Analytical Method](#)
[4.7: The Importance of Analytical Methodology](#)
[6.5: Blank Corrections](#)

Analytical Perspective

[16.1: The Analytical Perspective—Revisited](#)

analytical separation

[8.5: General Theory of Separation Efficiency](#)

Analytical Separations

[13.1: Overview of Analytical Separations](#)

analytical signal

[9.1: Overview of Gravimetric Methods](#)
[13.5: High-Performance Liquid Chromatography](#)
[14.1: Kinetic Methods Versus Equilibrium Methods](#)

Analytical titrations

[10.4: Redox Titrations](#)

Analyze Data

[5.8: Using Excel and R to Analyze Data](#)

anodic current

[12.4: Voltammetric Methods](#)

ANOVA

[15.3: Validating the Method as a Standard Method](#)

Applied Potential

[12.4: Voltammetric Methods](#)

aqueous buffer

[13.7: Electrophoresis](#)

argentometric titration

[10.5: Precipitation Titrations](#)

asymmetric equivalence point

[10.4: Redox Titrations](#)

asymmetric peaks

[13.2: General Theory of Column Chromatography](#)

atomic emission

[11.7: Atomic Emission Spectroscopy](#)

atomic emission spectra

[11.7: Atomic Emission Spectroscopy](#)

atomic emission spectroscopy

[11.7: Atomic Emission Spectroscopy](#)

atomic number

[14.3: Radiochemistry](#)

atomization

[11.4: Atomic Absorption Spectroscopy](#)
[11.7: Atomic Emission Spectroscopy](#)

atomization methods

[11.4: Atomic Absorption Spectroscopy](#)

attenuated total reflectance

[11.3: UV/Vis and IR Spectroscopy](#)

auxiliary complexing agent

[10.3: Complexation Titrations](#)

auxiliary electrode

- 12.1: Overview of Electrochemistry
- 13.5: High-Performance Liquid Chromatography

auxiliary oxidizing agents

- 10.4: Redox Titrations

auxiliary reducing agent

- 10.4: Redox Titrations

average

- 5.6: Statistical Methods for Normal Distributions

B**background correction**

- 11.4: Atomic Absorption Spectroscopy

balanced reaction

- 3.3: Stoichiometric Calculations

band broadening

- 13.2: General Theory of Column Chromatography

base

- 7.4: Equilibrium Constants for Chemical Reactions

baseline width

- 13.2: General Theory of Column Chromatography

Beer's law

- 11.2: Spectroscopy Based on Absorption

Berthollet

- 7.1: Reversible Reactions and Chemical Equilibria

beta particle

- 14.3: Radiochemistry

binomial distribution

- 5.4: The Distribution of Measurements and Results

blank

- 6.5: Blank Corrections

blind analysis

- 15.2: Verifying the Method

Bonded stationary phases

- 13.5: High-Performance Liquid Chromatography

box plot

- 5.8: Using Excel and R to Analyze Data

buffer

- 7.8: Buffer Solutions
- 13.7: Electrophoresis

buffer capacity

- 7.8: Buffer Solutions

buffer solutions

- 7.8: Buffer Solutions

buffer viscosity

- 13.7: Electrophoresis

buret

- 10.1: Overview of Titrimetry

C**calculations**

- 3.3: Stoichiometric Calculations

calibration

- 4.5: Developing the Procedure

calibration curve

- 14.2: Chemical Kinetics
- 15.1: Optimizing the Experimental Procedure

calibration curves

- 6.4: Linear Regression and Calibration Curves

Calomel

- 12.2: Potentiometric Methods

calomel electrodes

- 12.2: Potentiometric Methods

capillary

- 13.5: High-Performance Liquid Chromatography
- 13.7: Electrophoresis

capillary columns

- 13.3: Optimizing Chromatographic Separations
- 13.4: Gas Chromatography

capillary electrochromatography

- 13.7: Electrophoresis

capillary electrophoresis

- 13.7: Electrophoresis

capillary gel electrophoresis

- 13.7: Electrophoresis

capillary tubing

- 13.7: Electrophoresis

capillary zone electrophoresis

- 13.7: Electrophoresis

capillary's radius

- 13.7: Electrophoresis

catalytic activity

- 14.2: Chemical Kinetics

cathode

- 12.2: Potentiometric Methods

cathodic current

- 12.4: Voltammetric Methods

CCB

- 4.6: Protocols

CCV

- 4.6: Protocols

CEC

- 13.7: Electrophoresis

central composite design

- 15.1: Optimizing the Experimental Procedure

central limit theorem

- 5.4: The Distribution of Measurements and Results

central tendency

- 5.1: Characterizing Measurements and Results

centrifugal analyzer

- 14.2: Chemical Kinetics

centrifugation

- 8.6: Classifying Separation Techniques

CGE

- 13.7: Electrophoresis

characteristic decay

- 14.3: Radiochemistry

Characterization

- 10.2: Acid-Base Titrations
- 11.3: UV/Vis and IR Spectroscopy
- 12.3: Coulometric Methods
- 12.4: Voltammetric Methods
- 14.2: Chemical Kinetics
- 14.3: Radiochemistry

characterization analysis

- 2.3: Common Analytical Problems

charge

- 12.3: Coulometric Methods

charge balance

- 7.7: Solving Equilibrium Problems

charge balance equation

- 7.7: Solving Equilibrium Problems

charging current

- 12.4: Voltammetric Methods

Chauvenet's criterion

- 5.6: Statistical Methods for Normal Distributions

chemical analysis

- 2.1: What is Analytical Chemistry?

chemical interferences

- 11.4: Atomic Absorption Spectroscopy
- 11.7: Atomic Emission Spectroscopy

Chemical kinetic

- 14.2: Chemical Kinetics
- 14.4: Flow Injection Analysis

chemical kinetic methods

- 14.2: Chemical Kinetics
- 14.4: Flow Injection Analysis

chemical kinetics

- 14.2: Chemical Kinetics

chemical properties

- 8.6: Classifying Separation Techniques

chemical reaction

- 7.2: Thermodynamics and Equilibrium Chemistry
- 14.2: Chemical Kinetics

chemiluminescence

- 11.1: Overview of Spectroscopy

Chesapeake Bay Monitoring Program

- 4.7: The Importance of Analytical Methodology

chromatogram

- 13.4: Gas Chromatography

Chromatographic

- 13.2: General Theory of Column Chromatography

chromatographic columns

- 13.4: Gas Chromatography

chromatographic resolution

- 13.2: General Theory of Column Chromatography

chromatography

- 8.6: Classifying Separation Techniques
- 13.1: Overview of Analytical Separations
- 13.2: General Theory of Column Chromatography
- 13.4: Gas Chromatography
- 13.5: High-Performance Liquid Chromatography
- 13.6: Other Forms of Liquid Chromatography

Claude Berthollet

- 7.1: Reversible Reactions and Chemical Equilibria

Clinical

- 11.3: UV/Vis and IR Spectroscopy

coagulation

- 9.2: Precipitation Gravimetry

Collaborative Study

- 15.3: Validating the Method as a Standard Method

collaborative test

- 15.3: Validating the Method as a Standard Method

collaborative testing

- 15.3: Validating the Method as a Standard Method

column

- 13.6: Other Forms of Liquid Chromatography

column chromatography

- 13.1: Overview of Analytical Separations
- 13.2: General Theory of Column Chromatography

column efficiency

- 13.2: General Theory of Column Chromatography
- 13.3: Optimizing Chromatographic Separations

Columns

- 13.5: High-Performance Liquid Chromatography

common ion effect

- 7.7: Solving Equilibrium Problems

competitive inhibition

- 14.2: Chemical Kinetics

complexation

- 7.4: Equilibrium Constants for Chemical Reactions
- 7.6: Ladder Diagrams

complexation reactions

- 7.4: Equilibrium Constants for Chemical Reactions

complexation titrations[10.3: Complexation Titrations](#)**Complexation Titrimetry**[10.3: Complexation Titrations](#)**composite sample**[8.2: Designing a Sampling Plan](#)**concentration**[3.2: Concentration](#)[3.4: Basic Equipment](#)[5.7: Detection Limits](#)[6.4: Linear Regression and Calibration Curves](#)[7.7: Solving Equilibrium Problems](#)[7.9: Activity Effects](#)[7.10: Using Excel and R to Solve Equilibrium Problems](#)[8.5: General Theory of Separation Efficiency](#)[9.2: Precipitation Gravimetry](#)[10.2: Acid-Base Titrations](#)[11.8: Spectroscopy Based on Scattering](#)[12.2: Potentiometric Methods](#)[12.3: Coulometric Methods](#)[12.4: Voltammetric Methods](#)[13.1: Overview of Analytical Separations](#)[13.4: Gas Chromatography](#)[13.7: Electrophoresis](#)[14.2: Chemical Kinetics](#)[14.4: Flow Injection Analysis](#)[15.1: Optimizing the Experimental Procedure](#)[15.2: Verifying the Method](#)[16.3: Quality Assessment](#)**concentration gradient**[14.4: Flow Injection Analysis](#)**concentration techniques**[4.3: Classifying Analytical Techniques](#)**concentrations**[10.2: Acid-Base Titrations](#)[10.4: Redox Titrations](#)[12.1: Overview of Electrochemistry](#)[16.4: Evaluating Quality Assurance Data](#)**conditional formation constant**[10.3: Complexation Titrations](#)**conductivity**[13.5: High-Performance Liquid Chromatography](#)**confidence interval**[5.5: Statistical Analysis of Data](#)[15.3: Validating the Method as a Standard Method](#)**confidence intervals**[5.4: The Distribution of Measurements and Results](#)**coning**[8.3: Implementing the Sampling Plan](#)**conservation of mass**[9.1: Overview of Gravimetric Methods](#)**constant determinate error**[5.2: Characterizing Experimental Errors](#)**Constant Potential**[12.3: Coulometric Methods](#)**continuing calibration blank**[4.6: Protocols](#)**continuing calibration verification**[4.6: Protocols](#)**contour map**[15.1: Optimizing the Experimental Procedure](#)**Contract Laboratory Program**[4.6: Protocols](#)**control chart**[16.4: Evaluating Quality Assurance Data](#)**convection**[12.4: Voltammetric Methods](#)[14.4: Flow Injection Analysis](#)**convenience sampling**[8.2: Designing a Sampling Plan](#)**coprecipitates**[9.2: Precipitation Gravimetry](#)**corresponding**[7.8: Buffer Solutions](#)**cost**[4.4: Selecting an Analytical Method](#)**coulombs**[12.3: Coulometric Methods](#)**coulometric titration**[12.3: Coulometric Methods](#)**Coulometric Titrations**[12.3: Coulometric Methods](#)**coulometry**[12.3: Coulometric Methods](#)[13.5: High-Performance Liquid Chromatography](#)**counter electrode**[12.1: Overview of Electrochemistry](#)**countercurrent extraction**[13.1: Overview of Analytical Separations](#)**cryogenic focusing**[13.4: Gas Chromatography](#)**current**[12.1: Overview of Electrochemistry](#)[12.4: Voltammetric Methods](#)**current efficiency**[12.3: Coulometric Methods](#)**curve fitting**[14.2: Chemical Kinetics](#)**curvilinear regression**[6.4: Linear Regression and Calibration Curves](#)**cyclic voltammetry**[12.4: Voltammetric Methods](#)**D****dark current**[11.1: Overview of Spectroscopy](#)**data**[5.5: Statistical Analysis of Data](#)[5.8: Using Excel and R to Analyze Data](#)**data analysis**[5.8: Using Excel and R to Analyze Data](#)[6.6: Using Excel and R for a Regression Analysis](#)[15.4: Using Excel and R for an Analysis of Variance](#)**definitive techniques**[9.1: Overview of Gravimetric Methods](#)**degrees of freedom**[5.4: The Distribution of Measurements and Results](#)[15.3: Validating the Method as a Standard Method](#)**density**[8.6: Classifying Separation Techniques](#)**density gradient centrifugation**[8.6: Classifying Separation Techniques](#)**dependent**[15.1: Optimizing the Experimental Procedure](#)**dependent factors**[15.1: Optimizing the Experimental Procedure](#)**descriptive statistics**[5.8: Using Excel and R to Analyze Data](#)**desiccant**[3.4: Basic Equipment](#)**desiccator**[3.4: Basic Equipment](#)**detection**[15.2: Verifying the Method](#)**detection limit**[4.4: Selecting an Analytical Method](#)[5.7: Detection Limits](#)[15.2: Verifying the Method](#)[16.4: Evaluating Quality Assurance Data](#)**detector**[14.4: Flow Injection Analysis](#)**detectors**[13.5: High-Performance Liquid Chromatography](#)[13.7: Electrophoresis](#)**determinate error**[5.2: Characterizing Experimental Errors](#)**determination**[4.1: Analysis, Determination, and Measurement](#)[5.4: The Distribution of Measurements and Results](#)**dialysis**[8.6: Classifying Separation Techniques](#)[14.4: Flow Injection Analysis](#)**diffusion**[12.4: Voltammetric Methods](#)[14.4: Flow Injection Analysis](#)**diffusion layer**[12.4: Voltammetric Methods](#)**digestion**[9.2: Precipitation Gravimetry](#)**dilution**[3.5: Preparing Solutions](#)**Diode Array Spectrometer**[11.3: UV/Vis and IR Spectroscopy](#)**direct analysis**[9.1: Overview of Gravimetric Methods](#)**direct titration**[10.1: Overview of Titrimetry](#)**dispersion**[14.4: Flow Injection Analysis](#)**displacement titration**[10.1: Overview of Titrimetry](#)**dissociation**[7.4: Equilibrium Constants for Chemical Reactions](#)[7.8: Buffer Solutions](#)**dissociation constant**[7.4: Equilibrium Constants for Chemical Reactions](#)[7.8: Buffer Solutions](#)**distillation**[8.6: Classifying Separation Techniques](#)**Distribution of Measurements**[5.4: The Distribution of Measurements and Results](#)**distribution ratios**[8.7: Liquid-Liquid Extractions](#)**Dixon's Q-test**[5.6: Statistical Methods for Normal Distributions](#)**DME**[12.4: Voltammetric Methods](#)**dot chart**[5.8: Using Excel and R to Analyze Data](#)**dropping mercury electrode**[12.4: Voltammetric Methods](#)**Drying**[3.4: Basic Equipment](#)**duplicate samples**[16.3: Quality Assessment](#)

E

ECD

13.4: Gas Chromatography

EDTA

10.3: Complexation Titrations

effective

15.1: Optimizing the Experimental Procedure

effective bandwidth

11.1: Overview of Spectroscopy

effective electric

13.7: Electrophoresis

effective electric field

13.7: Electrophoresis

effectiveness

15.1: Optimizing the Experimental Procedure

efficiency

13.7: Electrophoresis

15.1: Optimizing the Experimental Procedure

electrochemical cell

12.3: Coulometric Methods

12.4: Voltammetric Methods

electrochemical cells

12.2: Potentiometric Methods

electrochemical detectors

13.5: High-Performance Liquid Chromatography

electrochemical measurements

13.5: High-Performance Liquid Chromatography

electrochemical techniques

12.1: Overview of Electrochemistry

electrochemically irreversible

12.4: Voltammetric Methods

electrochemically reversible

12.4: Voltammetric Methods

electrochemistry

4.3: Classifying Analytical Techniques

12.1: Overview of Electrochemistry

electrode

12.1: Overview of Electrochemistry

12.2: Potentiometric Methods

12.3: Coulometric Methods

electrode's potential

12.3: Coulometric Methods

electrodes

12.2: Potentiometric Methods

electrogravimetry

9.1: Overview of Gravimetric Methods

12.3: Coulometric Methods

electrokinetic injection

13.7: Electrophoresis

electrolysis

12.3: Coulometric Methods

electrolysis time

12.3: Coulometric Methods

electromagnetic radiation

11.1: Overview of Spectroscopy

11.2: Spectroscopy Based on Absorption

15.1: Optimizing the Experimental Procedure

electromagnetic spectrum

11.1: Overview of Spectroscopy

electron capture detector

13.4: Gas Chromatography

electroosmotic

13.7: Electrophoresis

electroosmotic flow

13.7: Electrophoresis

electroosmotic flow velocity

13.7: Electrophoresis

electropherogram

13.7: Electrophoresis

electrophoresis

13.7: Electrophoresis

Electrophoretic

13.1: Overview of Analytical Separations

electrophoretic mobility

13.7: Electrophoresis

Electrophoretic Separations

13.1: Overview of Analytical Separations

electrophoretic velocity

13.7: Electrophoresis

emission

11.1: Overview of Spectroscopy

14.3: Radiochemistry

emission spectra

11.6: Photoluminescence Spectroscopy

11.7: Atomic Emission Spectroscopy

emission spectroscopy

11.7: Atomic Emission Spectroscopy

emission spectrum

11.6: Photoluminescence Spectroscopy

empirical model

15.1: Optimizing the Experimental Procedure

end point

10.2: Acid–Base Titrations

10.3: Complexation Titrations

10.5: Precipitation Titrations

End points

10.1: Overview of Titrimetry

Endpoint

12.3: Coulometric Methods

enthalpy

7.2: Thermodynamics and Equilibrium Chemistry

entropy

7.2: Thermodynamics and Equilibrium Chemistry

environmental monitoring programs

4.7: The Importance of Analytical Methodology

enzyme catalysis

14.2: Chemical Kinetics

enzyme electrodes

12.2: Potentiometric Methods

enzymes

14.2: Chemical Kinetics

Equilibria

7.6: Ladder Diagrams

equilibrium

7.1: Reversible Reactions and Chemical Equilibria

7.3: Manipulating Equilibrium Constants

7.5: Le Châtelier's Principle

7.6: Ladder Diagrams

7.7: Solving Equilibrium Problems

7.9: Activity Effects

7.10: Using Excel and R to Solve Equilibrium

Problems

8.7: Liquid–Liquid Extractions

11.3: UV/Vis and IR Spectroscopy

12.1: Overview of Electrochemistry

equilibrium chemistry

7.6: Ladder Diagrams

equilibrium concentration

7.7: Solving Equilibrium Problems

7.10: Using Excel and R to Solve Equilibrium

Problems

equilibrium constan

7.3: Manipulating Equilibrium Constants

equilibrium constant

7.2: Thermodynamics and Equilibrium Chemistry

7.3: Manipulating Equilibrium Constants

12.2: Potentiometric Methods

equilibrium constants

7.3: Manipulating Equilibrium Constants

10.2: Acid–Base Titrations

11.3: UV/Vis and IR Spectroscopy

equilibrium method

14.1: Kinetic Methods Versus Equilibrium Methods

equilibrium reaction

8.7: Liquid–Liquid Extractions

14.1: Kinetic Methods Versus Equilibrium Methods

equipment

4.4: Selecting an Analytical Method

equivalence

10.1: Overview of Titrimetry

10.2: Acid–Base Titrations

equivalence point

10.1: Overview of Titrimetry

10.2: Acid–Base Titrations

10.4: Redox Titrations

Equivalence Points

10.1: Overview of Titrimetry

equivalency testing

15.2: Verifying the Method

Equivalent Weights

10.2: Acid–Base Titrations

error

5.2: Characterizing Experimental Errors

15.3: Validating the Method as a Standard Method

errors

16.4: Evaluating Quality Assurance Data

Ethylenediaminetetraacetic

10.3: Complexation Titrations

ethylenediaminetetraacetic acid

10.3: Complexation Titrations

Excel

3.6: Spreadsheets and Computational Software

5.8: Using Excel and R to Analyze Data

6.6: Using Excel and R for a Regression Analysis

15.4: Using Excel and R for an Analysis of Variance

excitation

11.2: Spectroscopy Based on Absorption

11.7: Atomic Emission Spectroscopy

excitation spectrum

11.6: Photoluminescence Spectroscopy

exclusion limit

13.6: Other Forms of Liquid Chromatography

experimental errors

5.2: Characterizing Experimental Errors

external conversion

11.6: Photoluminescence Spectroscopy

external standards

6.3: Determining the Sensitivity

12.2: Potentiometric Methods

extraction

8.6: Classifying Separation Techniques

8.7: Liquid–Liquid Extractions

extraction efficiency

8.7: Liquid–Liquid Extractions

F

factor levels

[15.1: Optimizing the Experimental Procedure](#)

Factorial

[15.1: Optimizing the Experimental Procedure](#)

factorial designs

[15.1: Optimizing the Experimental Procedure](#)

factors

[15.1: Optimizing the Experimental Procedure](#)

[15.2: Verifying the Method](#)

Fajans method

[10.5: Precipitation Titrations](#)

faradaic current

[12.4: Voltammetric Methods](#)

Faraday's constant

[12.3: Coulometric Methods](#)

Faraday's law

[12.3: Coulometric Methods](#)

fiagram

[14.4: Flow Injection Analysis](#)

FID

[13.4: Gas Chromatography](#)

field blank

[16.3: Quality Assessment](#)

[16.4: Evaluating Quality Assurance Data](#)

filter

[11.1: Overview of Spectroscopy](#)

Filter Photometer

[11.3: UV/Vis and IR Spectroscopy](#)

filtrate

[8.6: Classifying Separation Techniques](#)

filtration

[8.6: Classifying Separation Techniques](#)

Flame Atomizer

[11.4: Atomic Absorption Spectroscopy](#)

flame ionization detector

[13.4: Gas Chromatography](#)

flow injection

[14.4: Flow Injection Analysis](#)

flow injection analysis

[14.4: Flow Injection Analysis](#)

flow injection analyzer

[14.4: Flow Injection Analysis](#)

fluorescence

[11.6: Photoluminescence Spectroscopy](#)

Fluorescence spectra

[11.6: Photoluminescence Spectroscopy](#)

fluorescent

[11.6: Photoluminescence Spectroscopy](#)

[13.5: High-Performance Liquid Chromatography](#)

fluorescent quantum yield

[11.6: Photoluminescence Spectroscopy](#)

fluorimeter

[11.6: Photoluminescence Spectroscopy](#)

Forensic

[11.3: UV/Vis and IR Spectroscopy](#)

formality

[3.2: Concentration](#)

formation constant

[7.4: Equilibrium Constants for Chemical Reactions](#)

[14.1: Kinetic Methods Versus Equilibrium Methods](#)

formation constants

[10.3: Complexation Titrations](#)

Fourier transform spectrometer

[11.3: UV/Vis and IR Spectroscopy](#)

frequency

[11.1: Overview of Spectroscopy](#)

fundamental analysis

[2.3: Common Analytical Problems](#)

G

galvanostat

[12.1: Overview of Electrochemistry](#)

Galvanostats

[12.1: Overview of Electrochemistry](#)

gamma ray

[14.3: Radiochemistry](#)

gas

[8.3: Implementing the Sampling Plan](#)

[13.4: Gas Chromatography](#)

gas chromatography

[13.3: Optimizing Chromatographic Separations](#)

[13.4: Gas Chromatography](#)

Gas-Liquid Chromatography

[13.4: Gas Chromatography](#)

gaseous diffusion

[14.4: Flow Injection Analysis](#)

gases

[8.3: Implementing the Sampling Plan](#)

Gaussian

[13.2: General Theory of Column Chromatography](#)

GC detectors

[13.6: Other Forms of Liquid Chromatography](#)

Geiger counter

[14.3: Radiochemistry](#)

general elution problem

[13.3: Optimizing Chromatographic Separations](#)

Gibb's free energy

[7.2: Thermodynamics and Equilibrium Chemistry](#)

glass electrodes

[12.2: Potentiometric Methods](#)

GLC

[13.4: Gas Chromatography](#)

global optimum

[15.1: Optimizing the Experimental Procedure](#)

GMP

[16.2: Quality Control](#)

GMPs

[16.2: Quality Control](#)

good laboratory practices

[16.2: Quality Control](#)

good measurement practices

[16.2: Quality Control](#)

grab sample

[8.2: Designing a Sampling Plan](#)

gradient elutions

[13.5: High-Performance Liquid Chromatography](#)

graphite furnace

[11.4: Atomic Absorption Spectroscopy](#)

gravimetric analysis

[9: Gravimetric Methods](#)

gravimetric method

[4.4: Selecting an Analytical Method](#)

gravimetry

[4.3: Classifying Analytical Techniques](#)

[9: Gravimetric Methods](#)

[9.1: Overview of Gravimetric Methods](#)

[9.2: Precipitation Gravimetry](#)

[9.3: Volatilization Gravimetry](#)

[9.4: Particulate Gravimetry](#)

gross sample

[8.3: Implementing the Sampling Plan](#)

Grubb's test

[5.6: Statistical Methods for Normal Distributions](#)

GSC

[13.4: Gas Chromatography](#)

guard columns

[13.5: High-Performance Liquid Chromatography](#)

H

hanging mercury drop electrode

[12.4: Voltammetric Methods](#)

headspace sampling

[13.4: Gas Chromatography](#)

Henderson-Hasselbalch approximation

[7.8: Buffer Solutions](#)

heterogeneous

[8.2: Designing a Sampling Plan](#)

histogram

[5.4: The Distribution of Measurements and Results](#)

HMDE

[12.4: Voltammetric Methods](#)

homogeneous

[8.2: Designing a Sampling Plan](#)

homogeneous precipitation

[9.2: Precipitation Gravimetry](#)

HPLC

[13.5: High-Performance Liquid Chromatography](#)

HPLC column

[13.6: Other Forms of Liquid Chromatography](#)

HPLC detectors

[13.5: High-Performance Liquid Chromatography](#)

HPLC Plumbing

[13.5: High-Performance Liquid Chromatography](#)

hydrodynamic injection

[13.7: Electrophoresis](#)

hydrodynamic voltammetry

[12.4: Voltammetric Methods](#)

I

ICB

[4.6: Protocols](#)

ICV

[4.6: Protocols](#)

ICV and

[4.6: Protocols](#)

ignition

[9.2: Precipitation Gravimetry](#)

Ilkovic equation

[12.4: Voltammetric Methods](#)

in situ sampling

[8.2: Designing a Sampling Plan](#)

Incident Radiation

[11.8: Spectroscopy Based on Scattering](#)

inclusion

[9.2: Precipitation Gravimetry](#)

inclusion limit

13.6: Other Forms of Liquid Chromatography

Independent

15.1: Optimizing the Experimental Procedure

independent factors

15.1: Optimizing the Experimental Procedure

indeterminate errors

5.2: Characterizing Experimental Errors

indicator

10.1: Overview of Titrimetry

10.2: Acid–Base Titrations

10.5: Precipitation Titrations

12.2: Potentiometric Methods

indicator electrode

12.1: Overview of Electrochemistry

indicator electrodes

12.2: Potentiometric Methods

indirect analysis

9.1: Overview of Gravimetric Methods

infrared spectra

11.2: Spectroscopy Based on Absorption

inhibitor

14.2: Chemical Kinetics

initial calibration blank

4.6: Protocols

initial calibration verification

4.6: Protocols

initial rate

14.2: Chemical Kinetics

inorganic analysis

9.3: Volatilization Gravimetry

instrumentation

11.3: UV/Vis and IR Spectroscopy

11.6: Photoluminescence Spectroscopy

12.3: Coulometric Methods

14.3: Radiochemistry

14.4: Flow Injection Analysis

integrated rate law

14.2: Chemical Kinetics

Interfacial

12.1: Overview of Electrochemistry

Interfacial Concentrations

12.1: Overview of Electrochemistry

interferences

4.5: Developing the Procedure

interferent

4.4: Selecting an Analytical Method

interferogram

11.1: Overview of Spectroscopy

interferometer

11.1: Overview of Spectroscopy

Interferometers

11.1: Overview of Spectroscopy

internal conversion

11.6: Photoluminescence Spectroscopy

internal standard

6.3: Determining the Sensitivity

intersystem crossing

11.6: Photoluminescence Spectroscopy

ionic strength

7.9: Activity Effects

ionization suppressor

11.4: Atomic Absorption Spectroscopy

IR spectra

11.2: Spectroscopy Based on Absorption

IR spectroscopy

11.3: UV/Vis and IR Spectroscopy

isocratic elution

13.5: High-Performance Liquid Chromatography

isothermal

13.4: Gas Chromatography

isotope dilution

14.3: Radiochemistry

isotopes

14.3: Radiochemistry

IUPAC

5.7: Detection Limits

J

Jones reductor

10.4: Redox Titrations

Joule heating

13.7: Electrophoresis

judgmental sampling

8.2: Designing a Sampling Plan

junction potential

12.2: Potentiometric Methods

K

Ka

4.4: Selecting an Analytical Method

6.4: Linear Regression and Calibration Curves

7.4: Equilibrium Constants for Chemical Reactions

Kb

7.4: Equilibrium Constants for Chemical Reactions

KD

8.6: Classifying Separation Techniques

kernel density plot

5.8: Using Excel and R to Analyze Data

kinetic method

14.1: Kinetic Methods Versus Equilibrium Methods

Kjeldahl analysis

10.2: Acid–Base Titrations

L

laboratory

3.7: The Laboratory Notebook

laboratory notebook

3.7: The Laboratory Notebook

laboratory sample

8.3: Implementing the Sampling Plan

ladder diagram

7.6: Ladder Diagrams

12.3: Coulometric Methods

ladder diagrams

7.8: Buffer Solutions

laminar flow

14.4: Flow Injection Analysis

Le Châtelier's principle

7.5: Le Châtelier's Principle

7.6: Ladder Diagrams

Le Chatelier's principle

7.5: Le Châtelier's Principle

leveling

10.2: Acid–Base Titrations

lifetime

11.5: Emission Spectroscopy

ligand

7.4: Equilibrium Constants for Chemical Reactions

limit of identification

5.7: Detection Limits

limiting current

12.4: Voltammetric Methods

linear regression

6.4: Linear Regression and Calibration Curves

liquid chromatography

13.5: High-Performance Liquid Chromatography

liquid mobile phase

13.5: High-Performance Liquid Chromatography

Liquid–Liquid Extractions

13.1: Overview of Analytical Separations

liquid–solid adsorption chromatography

13.6: Other Forms of Liquid Chromatography

local optimum

15.1: Optimizing the Experimental Procedure

longitudinal diffusion

13.3: Optimizing Chromatographic Separations

loop injector

13.5: High-Performance Liquid Chromatography

LSC

13.6: Other Forms of Liquid Chromatography

M

manifold

14.4: Flow Injection Analysis

mass

3.4: Basic Equipment

4.3: Classifying Analytical Techniques

5.1: Characterizing Measurements and Results

8.6: Classifying Separation Techniques

9.1: Overview of Gravimetric Methods

mass spectrometer

13.4: Gas Chromatography

mass spectrum

13.4: Gas Chromatography

Mass Transfer

13.3: Optimizing Chromatographic Separations

mass transport

12.4: Voltammetric Methods

Mathematical

15.1: Optimizing the Experimental Procedure

mathematical models

15.1: Optimizing the Experimental Procedure

matrix

4.1: Analysis, Determination, and Measurement

matrix matching

6.3: Determining the Sensitivity

mean

5.1: Characterizing Measurements and Results

5.6: Statistical Methods for Normal Distributions

15.3: Validating the Method as a Standard Method

measurement

4.1: Analysis, Determination, and Measurement

5.1: Characterizing Measurements and Results

5.2: Characterizing Experimental Errors

measurement errors

5.2: Characterizing Experimental Errors

measurements

3.1: Measurements in Analytical Chemistry

5.4: The Distribution of Measurements and Results

12.2: Potentiometric Methods

15.1: Optimizing the Experimental Procedure

Measuring Volume

3.4: Basic Equipment

median

5.1: Characterizing Measurements and Results

medication

5.5: Statistical Analysis of Data

MEKC

13.7: Electrophoresis

membrane electrodes

12.2: Potentiometric Methods

membrane potential

12.2: Potentiometric Methods

Membrane Potentials

12.2: Potentiometric Methods

Membranes

12.2: Potentiometric Methods

meniscus

3.4: Basic Equipment

mercury film electrode

12.4: Voltammetric Methods

metallochromic indicators

10.3: Complexation Titrations

method

4.2: Techniques, Methods, Procedures, and Protocols

4.4: Selecting an Analytical Method

6.3: Determining the Sensitivity

method blank

4.5: Developing the Procedure

16.3: Quality Assessment

method error

5.2: Characterizing Experimental Errors

method errors

5.2: Characterizing Experimental Errors

method of continuous variations

11.3: UV/Vis and IR Spectroscopy

method of standard additions

6.3: Determining the Sensitivity

micellar electrokinetic capillary

chromatography

13.7: Electrophoresis

Micelle

13.7: Electrophoresis

Michaelis constant

14.2: Chemical Kinetics

microextraction

13.4: Gas Chromatography

Microsoft Excel

3.6: Spreadsheets and Computational Software

migration

12.4: Voltammetric Methods

migration time

13.7: Electrophoresis

mobile phase

13.1: Overview of Analytical Separations

13.4: Gas Chromatography

13.5: High-Performance Liquid Chromatography

13.6: Other Forms of Liquid Chromatography

Mobile Phases

13.1: Overview of Analytical Separations

Mobility

13.7: Electrophoresis

Mohr method

10.5: Precipitation Titrations

molar absorptivity

11.2: Spectroscopy Based on Absorption

15.1: Optimizing the Experimental Procedure

molarity

3.2: Concentration

moles

3.3: Stoichiometric Calculations

Monitoring Absorbance

10.3: Complexation Titrations

monitoring program

4.7: The Importance of Analytical Methodology

monochromatic

11.1: Overview of Spectroscopy

11.8: Spectroscopy Based on Scattering

monochromator

11.1: Overview of Spectroscopy

monolithic columns

13.5: High-Performance Liquid Chromatography

Monoprotic

7.4: Equilibrium Constants for Chemical Reactions

7.7: Solving Equilibrium Problems

MS

13.4: Gas Chromatography

Multicomponent Analysis

12.4: Voltammetric Methods

multielemental analysis

11.7: Atomic Emission Spectroscopy

multiple paths

13.3: Optimizing Chromatographic Separations

multivariate regression

6.4: Linear Regression and Calibration Curves

N

negatron

14.3: Radiochemistry

nephelometry

11.8: Spectroscopy Based on Scattering

Nernst equation

7.4: Equilibrium Constants for Chemical Reactions

12.2: Potentiometric Methods

Nessler

11.1: Overview of Spectroscopy

neutron

14.3: Radiochemistry

neutron activation

14.3: Radiochemistry

neutron activation analysis

14.3: Radiochemistry

neutron flux

14.3: Radiochemistry

nominal wavelength

11.1: Overview of Spectroscopy

noncatalytic reactions

14.2: Chemical Kinetics

Noncompetitive Inhibition

14.2: Chemical Kinetics

nonfaradaic current

12.4: Voltammetric Methods

nonretained solutes

13.2: General Theory of Column Chromatography

normal calibration curve

6.3: Determining the Sensitivity

normal distribution

5.4: The Distribution of Measurements and Results

5.5: Statistical Analysis of Data

5.6: Statistical Methods for Normal Distributions

Normal Distributions

5.6: Statistical Methods for Normal Distributions

normality

3.2: Concentration

notebook

3.7: The Laboratory Notebook

Nuclear Reactor

14.3: Radiochemistry

Nyquist theorem

8.2: Designing a Sampling Plan

O

Occlusions

9.2: Precipitation Gravimetry

Ohm's law

12.1: Overview of Electrochemistry

open tubular column

13.4: Gas Chromatography

optimization

15.1: Optimizing the Experimental Procedure

Optimize Resolution

13.3: Optimizing Chromatographic Separations

organic analysis

9.3: Volatilization Gravimetry

outliers

5.6: Statistical Methods for Normal Distributions

overpotential

12.3: Coulometric Methods

Oxidation

7.4: Equilibrium Constants for Chemical Reactions

7.6: Ladder Diagrams

oxidation reaction

12.2: Potentiometric Methods

oxidizing agent

7.4: Equilibrium Constants for Chemical Reactions

P

packed columns

13.4: Gas Chromatography

paired data

5.6: Statistical Methods for Normal Distributions

Participate

12.1: Overview of Electrochemistry

particulate gravimetry

9.1: Overview of Gravimetric Methods

9.4: Particulate Gravimetry

particulates

9.4: Particulate Gravimetry

partition coefficient

8.6: Classifying Separation Techniques

partition coefficients

8.7: Liquid-Liquid Extractions

parts per billion

3.2: Concentration

parts per million

3.2: Concentration

pathlength

15.1: Optimizing the Experimental Procedure

peak capacity

13.2: General Theory of Column Chromatography

peak current

12.4: Voltammetric Methods

Peak height

14.4: Flow Injection Analysis

peak shape

13.2: General Theory of Column Chromatography

peptization

9.2: Precipitation Gravimetry

personal error

5.2: Characterizing Experimental Errors

pH

7.4: Equilibrium Constants for Chemical Reactions

7.10: Using Excel and R to Solve Equilibrium

Problems

10.2: Acid–Base Titrations

12.2: Potentiometric Methods

pH scale

7.4: Equilibrium Constants for Chemical Reactions

phase angle

11.1: Overview of Spectroscopy

phosphorescence

11.6: Photoluminescence Spectroscopy

Phosphorescence Spectra

11.6: Photoluminescence Spectroscopy

phosphorescent

11.6: Photoluminescence Spectroscopy

phosphorescent quantum yield

11.6: Photoluminescence Spectroscopy

photodiode array

11.1: Overview of Spectroscopy

Photoluminescence

11.1: Overview of Spectroscopy

11.5: Emission Spectroscopy

11.6: Photoluminescence Spectroscopy

Photoluminescence spectroscopy

11.6: Photoluminescence Spectroscopy

photon

11.1: Overview of Spectroscopy

11.2: Spectroscopy Based on Absorption

photon transducers

11.1: Overview of Spectroscopy

photons

11.1: Overview of Spectroscopy

physical properties

8.6: Classifying Separation Techniques

pipet

3.4: Basic Equipment

planar chromatography

13.1: Overview of Analytical Separations

plasma

11.7: Atomic Emission Spectroscopy

PLOT

6.6: Using Excel and R for a Regression Analysis

13.4: Gas Chromatography

Plumbing

13.5: High-Performance Liquid Chromatography

pnorm

5.8: Using Excel and R to Analyze Data

point

10.2: Acid–Base Titrations

polarity index

13.5: High-Performance Liquid Chromatography

polarography

12.4: Voltammetric Methods

polychromatic

11.1: Overview of Spectroscopy

Polyprotic

7.4: Equilibrium Constants for Chemical Reactions

7.7: Solving Equilibrium Problems

population

5.4: The Distribution of Measurements and Results

8.1: The Importance of Sampling

8.2: Designing a Sampling Plan

Populations

5.4: The Distribution of Measurements and Results

Positron

14.3: Radiochemistry

potential

12.1: Overview of Electrochemistry

12.2: Potentiometric Methods

12.4: Voltammetric Methods

potential interferents

13.1: Overview of Analytical Separations

potentiometer

12.1: Overview of Electrochemistry

Potentiometric

12.2: Potentiometric Methods

potentiometric biosensors

12.2: Potentiometric Methods

potentiometric measurements

12.2: Potentiometric Methods

potentiometric method

12.3: Coulometric Methods

Potentiometric Methods

12.2: Potentiometric Methods

potentiometric titration curve

10.2: Acid–Base Titrations

potentiometry

12.2: Potentiometric Methods

potentiostat

12.1: Overview of Electrochemistry

Potentiostats

12.1: Overview of Electrochemistry

ppb

3.2: Concentration

ppm

3.2: Concentration

precipitant

9.2: Precipitation Gravimetry

precipitate

7.4: Equilibrium Constants for Chemical Reactions

9.2: Precipitation Gravimetry

precipitation

9.2: Precipitation Gravimetry

9.4: Particulate Gravimetry

10.1: Overview of Titrimetry

10.5: Precipitation Titrations

precipitation gravimetry

9.1: Overview of Gravimetric Methods

9.2: Precipitation Gravimetry

precipitation reactions

7.4: Equilibrium Constants for Chemical Reactions

precipitation titration

10.5: Precipitation Titrations

Precipitation Titrimetry

10.5: Precipitation Titrations

precision

4.4: Selecting an Analytical Method

5.2: Characterizing Experimental Errors

9.2: Precipitation Gravimetry

10.2: Acid–Base Titrations

11.3: UV/Vis and IR Spectroscopy

11.4: Atomic Absorption Spectroscopy

11.7: Atomic Emission Spectroscopy

15.2: Verifying the Method

15.3: Validating the Method as a Standard Method

16.4: Evaluating Quality Assurance Data

preconcentration

8.8: Separation Versus Preconcentration

prescriptive approach

16.4: Evaluating Quality Assurance Data

pressure

7.2: Thermodynamics and Equilibrium Chemistry

primary standard

6.1: Analytical Standards

probability distributions

5.4: The Distribution of Measurements and Results

5.8: Using Excel and R to Analyze Data

procedure

4.2: Techniques, Methods, Procedures, and Protocols

product

7.5: Le Châtelier's Principle

products

3.3: Stoichiometric Calculations

7.5: Le Châtelier's Principle

proficiency standards

16.3: Quality Assessment

propagation of uncertainty

5.3: Propagation of Uncertainty

property control chart

16.4: Evaluating Quality Assurance Data

proportional determinate error

5.2: Characterizing Experimental Errors

protocol

4.2: Techniques, Methods, Procedures, and Protocols

protocol for a specific purpose

16.2: Quality Control

proton

14.3: Radiochemistry

PSP

16.2: Quality Control

pulse polarography

12.4: Voltammetric Methods

Q

qualitative analysis

2.3: Common Analytical Problems

qualitative applications

9.2: Precipitation Gravimetry

11.3: UV/Vis and IR Spectroscopy

quality assessment

16.1: The Analytical Perspective—Revisited

16.3: Quality Assessment

16.4: Evaluating Quality Assurance Data

quality assurance

4.6: Protocols

quality assurance program

16.1: The Analytical Perspective—Revisited

16.4: Evaluating Quality Assurance Data

quality control

4.6: Protocols

16.1: The Analytical Perspective—Revisited

16.2: Quality Control

quantitative analysis

2.3: Common Analytical Problems

quantitative applications

11.7: Atomic Emission Spectroscopy

12.3: Coulometric Methods

quantitative estimates

16.4: Evaluating Quality Assurance Data

quantitative science

3.1: Measurements in Analytical Chemistry

quantitative transfer

3.5: Preparing Solutions

quartering

8.3: Implementing the Sampling Plan

quartz crystal microbalance

9.4: Particulate Gravimetry

quench

14.2: Chemical Kinetics

R

R

5.8: Using Excel and R to Analyze Data

15.4: Using Excel and R for an Analysis of Variance

R for

15.4: Using Excel and R for an Analysis of Variance

radiation

11.8: Spectroscopy Based on Scattering

radiationless deactivation

11.6: Photoluminescence Spectroscopy

radioactive

14.3: Radiochemistry

Radioactive Analytes

14.3: Radiochemistry

radioactive atoms

14.3: Radiochemistry

radioactive particles

14.3: Radiochemistry

radioactive tracer

14.3: Radiochemistry

radiochemical methods

14.4: Flow Injection Analysis

Radiochemistry

14.3: Radiochemistry

random error

15.3: Validating the Method as a Standard Method

random errors

16.4: Evaluating Quality Assurance Data

random sampling

8.2: Designing a Sampling Plan

range

5.1: Characterizing Measurements and Results

rate

14.2: Chemical Kinetics

rate constant

12.3: Coulometric Methods

14.2: Chemical Kinetics

rate law

14.2: Chemical Kinetics

rate method

14.2: Chemical Kinetics

reactant

7.5: Le Châtelier's Principle

reactants

7.5: Le Châtelier's Principle

reaction rate

14.2: Chemical Kinetics

reagent blank

15.2: Verifying the Method

16.3: Quality Assessment

reagent grade

6.1: Analytical Standards

recovery

8.5: General Theory of Separation Efficiency

redox

10.4: Redox Titrations

12.2: Potentiometric Methods

Redox Electrodes

12.2: Potentiometric Methods

redox indicator

10.4: Redox Titrations

redox titration

10.4: Redox Titrations

Redox Titration Curves

10.4: Redox Titrations

Redox Titrimetry

10.4: Redox Titrations

reducing agent

7.4: Equilibrium Constants for Chemical Reactions

Reduction

7.4: Equilibrium Constants for Chemical Reactions

reference electrode

12.1: Overview of Electrochemistry

reference electrodes

12.2: Potentiometric Methods

Regression

6.6: Using Excel and R for a Regression Analysis

regression analysis

6.6: Using Excel and R for a Regression Analysis

regression equation

6.4: Linear Regression and Calibration Curves

relative supersaturation

9.2: Precipitation Gravimetry

relaxation

11.5: Emission Spectroscopy

repeatability

5.2: Characterizing Experimental Errors

reproducibility

5.2: Characterizing Experimental Errors

Residual Current

12.4: Voltammetric Methods

resolution

11.1: Overview of Spectroscopy

13.2: General Theory of Column Chromatography

13.3: Optimizing Chromatographic Separations

response

15.1: Optimizing the Experimental Procedure

response surface

15.1: Optimizing the Experimental Procedure

retentate

8.6: Classifying Separation Techniques

retention factor

13.2: General Theory of Column Chromatography

13.3: Optimizing Chromatographic Separations

retention factors

13.7: Electrophoresis

retention time

13.2: General Theory of Column Chromatography

robust

4.4: Selecting an Analytical Method

room temperature

7.4: Equilibrium Constants for Chemical Reactions

rugged

4.4: Selecting an Analytical Method

ruggedness testing

15.2: Verifying the Method

S

salt bridge

12.2: Potentiometric Methods

sample

5.4: The Distribution of Measurements and Results

13.4: Gas Chromatography

13.7: Electrophoresis

Sample Cells

11.3: UV/Vis and IR Spectroscopy

sample collection

8.3: Implementing the Sampling Plan

Sample Distributions

5.4: The Distribution of Measurements and Results

sample introduction

11.4: Atomic Absorption Spectroscopy

Sample Means

5.6: Statistical Methods for Normal Distributions

sample preparation

8.3: Implementing the Sampling Plan

sample preservation

8.3: Implementing the Sampling Plan

samples

5.4: The Distribution of Measurements and Results

13.5: High-Performance Liquid Chromatography

sampling

4.5: Developing the Procedure

8.1: The Importance of Sampling

8.2: Designing a Sampling Plan

8.3: Implementing the Sampling Plan

sampling plan

8.2: Designing a Sampling Plan

8.3: Implementing the Sampling Plan

saturated calomel electrode

12.2: Potentiometric Methods

scale of operation

4.4: Selecting an Analytical Method

11.4: Atomic Absorption Spectroscopy

11.6: Photoluminescence Spectroscopy

11.7: Atomic Emission Spectroscopy

Scattering

11.8: Spectroscopy Based on Scattering

scientific notation

3.1: Measurements in Analytical Chemistry

scintillation counter

14.3: Radiochemistry

SCOT

13.4: Gas Chromatography

searching algorithm

15.1: Optimizing the Experimental Procedure

secondary standards

6.1: Analytical Standards

selectivity

4.4: Selecting an Analytical Method

10.2: Acid-Base Titrations

13.3: Optimizing Chromatographic Separations

13.5: High-Performance Liquid Chromatography

13.7: Electrophoresis

14.2: Chemical Kinetics

selectivity coefficient

4.4: Selecting an Analytical Method

8.4: Separating the Analyte from Interferents

selectivity factor

13.2: General Theory of Column Chromatography

Sensitivity

- 4.4: Selecting an Analytical Method
- 6.3: Determining the Sensitivity
- 9.2: Precipitation Gravimetry
- 10.2: Acid–Base Titrations

Separate Mixtures

- 13.1: Overview of Analytical Separations

separation

- 8.5: General Theory of Separation Efficiency
- 8.6: Classifying Separation Techniques
- 8.8: Separation Versus Preconcentration

separation factor

- 8.5: General Theory of Separation Efficiency

Separation Modules

- 14.4: Flow Injection Analysis

SFC

- 13.6: Other Forms of Liquid Chromatography

SHE

- 12.2: Potentiometric Methods

SI units

- 3.1: Measurements in Analytical Chemistry

signal

- 4.3: Classifying Analytical Techniques
- 6.2: Calibrating the Signal
- 10.1: Overview of Titrimetry
- 11.1: Overview of Spectroscopy
- 13.5: High-Performance Liquid Chromatography
- 14.4: Flow Injection Analysis

signal averaging

- 11.3: UV/Vis and IR Spectroscopy

signal processor

- 11.1: Overview of Spectroscopy

significance test

- 5.5: Statistical Analysis of Data

significance testing

- 5.5: Statistical Analysis of Data

significance tests

- 5.8: Using Excel and R to Analyze Data

significant figures

- 3.1: Measurements in Analytical Chemistry

silica

- 13.5: High-Performance Liquid Chromatography

silica particles

- 13.5: High-Performance Liquid Chromatography

silver/silver chloride electrode

- 12.2: Potentiometric Methods

simplex

- 15.1: Optimizing the Experimental Procedure

simplex optimization

- 15.1: Optimizing the Experimental Procedure

Single Components

- 12.4: Voltammetric Methods

Single Operator Characteristics

- 15.2: Verifying the Method

singlet excited state

- 11.6: Photoluminescence Spectroscopy

slope

- 6.4: Linear Regression and Calibration Curves

SMDE

- 12.4: Voltammetric Methods

solid

- 8.3: Implementing the Sampling Plan

solids

- 8.3: Implementing the Sampling Plan

solubility

- 7.7: Solving Equilibrium Problems
- 7.10: Using Excel and R to Solve Equilibrium Problems

Problems

- 9.2: Precipitation Gravimetry

solubility product

- 7.4: Equilibrium Constants for Chemical Reactions

solute

- 13.1: Overview of Analytical Separations

solute's charge

- 13.7: Electrophoresis

solute's radius

- 13.7: Electrophoresis

solutions

- 3.5: Preparing Solutions
- 8.3: Implementing the Sampling Plan

SOP

- 16.2: Quality Control

Soxhlet extractor

- 8.6: Classifying Separation Techniques

specificity

- 4.4: Selecting an Analytical Method

spectral interferences

- 11.7: Atomic Emission Spectroscopy

spectral searching

- 11.3: UV/Vis and IR Spectroscopy

spectrofluorimeter

- 11.6: Photoluminescence Spectroscopy
- 13.5: High-Performance Liquid Chromatography

spectrophotometer

- 11.3: UV/Vis and IR Spectroscopy

spectrophotometric titration curve

- 10.3: Complexation Titrations

spectrophotometry

- 15.1: Optimizing the Experimental Procedure

spectroscopic detectors

- 13.5: High-Performance Liquid Chromatography

Spectroscopic methods

- 11.5: Emission Spectroscopy

spectroscopy

- 4.3: Classifying Analytical Techniques
- 11.1: Overview of Spectroscopy
- 11.2: Spectroscopy Based on Absorption
- 11.8: Spectroscopy Based on Scattering
- 14.4: Flow Injection Analysis

spike recovery

- 16.3: Quality Assessment
- 16.4: Evaluating Quality Assurance Data

splitless injection

- 13.4: Gas Chromatography

SPME

- 13.4: Gas Chromatography

spreadsheets

- 3.6: Spreadsheets and Computational Software

stacking

- 13.7: Electrophoresis

standard

- 6.5: Blank Corrections
- 12.2: Potentiometric Methods
- 15.1: Optimizing the Experimental Procedure

standard additions

- 6.3: Determining the Sensitivity
- 12.2: Potentiometric Methods

standard deviation

- 5.1: Characterizing Measurements and Results
- 15.3: Validating the Method as a Standard Method
- 16.4: Evaluating Quality Assurance Data

standard deviation about the regression

- 6.4: Linear Regression and Calibration Curves

standard error of the mean

- 5.4: The Distribution of Measurements and Results

standard hydrogen electrode

- 12.2: Potentiometric Methods

Standard Method

- 15.1: Optimizing the Experimental Procedure
- 15.2: Verifying the Method
- 15.3: Validating the Method as a Standard Method

standard operations procedure

- 16.2: Quality Control

standard potential

- 7.4: Equilibrium Constants for Chemical Reactions

standard reference materials

- 5.2: Characterizing Experimental Errors

standard sample

- 15.2: Verifying the Method

standard state

- 7.2: Thermodynamics and Equilibrium Chemistry

standardization

- 6.3: Determining the Sensitivity

Standardizing

- 10.2: Acid–Base Titrations
- 10.4: Redox Titrations

standards

- 12.2: Potentiometric Methods

static mercury drop electrode

- 12.4: Voltammetric Methods

stationary phase

- 13.1: Overview of Analytical Separations
- 13.6: Other Forms of Liquid Chromatography

stationary phases

- 13.1: Overview of Analytical Separations
- 13.5: High-Performance Liquid Chromatography

statistical analysis

- 5.5: Statistical Analysis of Data

statistical control

- 16.1: The Analytical Perspective—Revisited

statistical methods

- 5.6: Statistical Methods for Normal Distributions
- 15.3: Validating the Method as a Standard Method

stock solution

- 3.5: Preparing Solutions

stoichiometric

- 3.3: Stoichiometric Calculations

stoichiometric relationship

- 3.3: Stoichiometric Calculations

stoichiometry

- 4.4: Selecting an Analytical Method
- 11.3: UV/Vis and IR Spectroscopy

stratified sampling

- 8.2: Designing a Sampling Plan

Stray radiation

- 11.2: Spectroscopy Based on Absorption

stripping voltammetry

- 12.4: Voltammetric Methods

strong acid

- 7.4: Equilibrium Constants for Chemical Reactions
- 7.8: Buffer Solutions

strong acids

- 10.2: Acid–Base Titrations

strong base

- 7.4: Equilibrium Constants for Chemical Reactions
- 7.8: Buffer Solutions

strong bases

10.2: Acid–Base Titrations

sublimation

8.6: Classifying Separation Techniques

subsamples

8.3: Implementing the Sampling Plan

substrate

14.2: Chemical Kinetics

supercritical fluid

8.6: Classifying Separation Techniques

supercritical fluid chromatography

13.6: Other Forms of Liquid Chromatography

supernatant

9.2: Precipitation Gravimetry

surface adsorbates

9.2: Precipitation Gravimetry

symbolic notation

3.2: Concentration

symmetric equivalence point

10.4: Redox Titrations

systematic errors

15.3: Validating the Method as a Standard Method

16.4: Evaluating Quality Assurance Data

systematic sampling

8.2: Designing a Sampling Plan

systematic–judgmental sampling

8.2: Designing a Sampling Plan

T

target population

8.1: The Importance of Sampling

TCD

13.4: Gas Chromatography

technique

4.2: Techniques, Methods, Procedures, and Protocols

temperature

7.2: Thermodynamics and Equilibrium Chemistry

13.4: Gas Chromatography

temperature control

13.4: Gas Chromatography

Temperature programming

13.4: Gas Chromatography

Tempmperature

10.2: Acid–Base Titrations

theoretical model

15.1: Optimizing the Experimental Procedure

theory

9.2: Precipitation Gravimetry

thermal conductivity detector

13.4: Gas Chromatography

thermal excitation

11.5: Emission Spectroscopy

thermodynamically

7.2: Thermodynamics and Equilibrium Chemistry

thermodynamics

7.2: Thermodynamics and Equilibrium Chemistry

thermogram

9.3: Volatilization Gravimetry

thermogravimetry

9.3: Volatilization Gravimetry

thermometric titration curve

10.1: Overview of Titrimetry

time

4.4: Selecting an Analytical Method

TISAB

12.2: Potentiometric Methods

titrand

10.1: Overview of Titrimetry

10.4: Redox Titrations

10.5: Precipitation Titrations

titrant

10.1: Overview of Titrimetry

10.5: Precipitation Titrations

titration

10.2: Acid–Base Titrations

titration curve

10.3: Complexation Titrations

10.5: Precipitation Titrations

titration curves

10.1: Overview of Titrimetry

10.4: Redox Titrations

10.5: Precipitation Titrations

titration error

10.1: Overview of Titrimetry

titrations

10.5: Precipitation Titrations

titrimetry

4.3: Classifying Analytical Techniques

10.1: Overview of Titrimetry

10.2: Acid–Base Titrations

10.3: Complexation Titrations

10.4: Redox Titrations

10.5: Precipitation Titrations

tolerance

5.2: Characterizing Experimental Errors

total analysis technique

4.3: Classifying Analytical Techniques

total charge

12.3: Coulometric Methods

total ionic strength adjustment buffer

12.2: Potentiometric Methods

Total Mobility

13.7: Electrophoresis

total Youden blank

6.5: Blank Corrections

tracer

14.3: Radiochemistry

transducer

11.1: Overview of Spectroscopy

transmittance

11.2: Spectroscopy Based on Absorption

11.3: UV/Vis and IR Spectroscopy

trip blank

16.3: Quality Assessment

triplet excited state

11.6: Photoluminescence Spectroscopy

turbidimetry

11.8: Spectroscopy Based on Scattering

type 1 error

5.5: Statistical Analysis of Data

type 2 error

5.5: Statistical Analysis of Data

U

ultramicro

4.4: Selecting an Analytical Method

uncertainty

3.1: Measurements in Analytical Chemistry

5.2: Characterizing Experimental Errors

5.3: Propagation of Uncertainty

6.4: Linear Regression and Calibration Curves

11.3: UV/Vis and IR Spectroscopy

15.1: Optimizing the Experimental Procedure

Uncompetitive Inhibition

14.2: Chemical Kinetics

unit

3.2: Concentration

unit conversion

3.2: Concentration

units

3.2: Concentration

unpaired data

5.6: Statistical Methods for Normal Distributions

unweighted linear regression

6.4: Linear Regression and Calibration Curves

V

validation

4.5: Developing the Procedure

15.3: Validating the Method as a Standard Method

van Deemter equation

13.3: Optimizing Chromatographic Separations

vanadium

15.1: Optimizing the Experimental Procedure

variance

5.1: Characterizing Measurements and Results

8.2: Designing a Sampling Plan

15.3: Validating the Method as a Standard Method

15.4: Using Excel and R for an Analysis of Variance

Variations

13.3: Optimizing Chromatographic Separations

vibrational energy level

11.6: Photoluminescence Spectroscopy

vibrational relaxation

11.6: Photoluminescence Spectroscopy

Visualizing Data

5.8: Using Excel and R to Analyze Data

Vmax

14.2: Chemical Kinetics

void time

13.2: General Theory of Column Chromatography

volatilization

9.3: Volatilization Gravimetry

volatilization gravimetric

9.4: Particulate Gravimetry

volatilization gravimetry

9.1: Overview of Gravimetric Methods

9.3: Volatilization Gravimetry

Volhard method

10.5: Precipitation Titrations

Voltammetric Measurements

12.4: Voltammetric Methods

voltammetric methods

12.4: Voltammetric Methods

Voltammetric Technique

12.4: Voltammetric Methods

voltammetry

12.4: Voltammetric Methods

13.5: High-Performance Liquid Chromatography

voltammogram

12.4: Voltammetric Methods

Voltammograms[12.4: Voltammetric Methods](#)**volume**[3.4: Basic Equipment](#)[4.3: Classifying Analytical Techniques](#)**volume percent**[3.2: Concentration](#)**volumetric buret**[10.1: Overview of Titrimetry](#)**volumetric flask**[3.4: Basic Equipment](#)**volumetric pipet**[3.4: Basic Equipment](#)**W****wavelength**[11.1: Overview of Spectroscopy](#)[11.7: Atomic Emission Spectroscopy](#)[11.8: Spectroscopy Based on Scattering](#)**wavenumber**[11.1: Overview of Spectroscopy](#)**weak acid**[7.7: Solving Equilibrium Problems](#)[10.3: Complexation Titrations](#)**weak acids**[7.4: Equilibrium Constants for Chemical Reactions](#)**weak base**[7.8: Buffer Solutions](#)**weight percent**[3.2: Concentration](#)**where**[15.1: Optimizing the Experimental Procedure](#)**working electrode**[12.1: Overview of Electrochemistry](#)[12.3: Coulometric Methods](#)[13.5: High-Performance Liquid Chromatography](#)**Z****zeta potential**[13.7: Electrophoresis](#)

Glossary

Sample Word 1 | Sample Definition 1

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- Analytical Chemistry - *Undeclared*
 - Front Matter - *Undeclared*
 - [TitlePage](#) - *Undeclared*
 - [InfoPage](#) - *Undeclared*
 - [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](#)
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 - 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

- 11.8: Spectroscopy Based on Scattering - CC BY-NC-SA 4.0
- 11.9: Spectroscopic Methods (Exercises) - CC BY-NC-SA 4.0
- 11.10: Spectroscopic Methods (Summary) - CC BY-NC-SA 4.0
- 12: Electrochemical Methods - CC BY-NC-SA 4.0
 - 12.1: Overview of Electrochemistry - CC BY-NC-SA 4.0
 - 12.2: Potentiometric Methods - CC BY-NC-SA 4.0
 - 12.3: Coulometric Methods - CC BY-NC-SA 4.0
 - 12.4: Voltammetric Methods - CC BY-NC-SA 4.0
 - 12.5: Electrochemical Methods (Exercises) - CC BY-NC-SA 4.0
 - 12.6: Electrochemical Methods (Summary) - CC BY-NC-SA 4.0
- 13: Chromatographic - CC BY-NC-SA 4.0
 - 13.1: Overview of Analytical Separations - CC BY-NC-SA 4.0
 - 13.2: General Theory of Column Chromatography - CC BY-NC-SA 4.0
 - 13.3: Optimizing Chromatographic Separations - CC BY-NC-SA 4.0
 - 13.4: Gas Chromatography - CC BY-NC-SA 4.0
 - 13.5: High-Performance Liquid Chromatography - CC BY-NC-SA 4.0
 - 13.6: Other Forms of Liquid Chromatography - CC BY-NC-SA 4.0
 - 13.7: Electrophoresis - CC BY-NC-SA 4.0
 - 13.8: Chromatographic and Electrophoretic Methods (Exercises) - CC BY-NC-SA 4.0
 - 13.9: Chromatographic and Electrophoretic Methods (Summary) - CC BY-NC-SA 4.0
- 14: Kinetic Methods - CC BY-NC-SA 4.0
 - 14.1: Kinetic Methods Versus Equilibrium Methods - CC BY-NC-SA 4.0
 - 14.2: Chemical Kinetics - CC BY-NC-SA 4.0
 - 14.3: Radiochemistry - CC BY-NC-SA 4.0
 - 14.4: Flow Injection Analysis - CC BY-NC-SA 4.0
 - 14.5: Kinetic Methods (Exercises) - CC BY-NC-SA 4.0
 - 14.6: Kinetic Methods (Summary) - CC BY-NC-SA 4.0
- 15: Developing a Standard Method - CC BY-NC-SA 4.0
 - 15.1: Optimizing the Experimental Procedure - CC BY-NC-SA 4.0
 - 15.2: Verifying the Method - CC BY-NC-SA 4.0
 - 15.3: Validating the Method as a Standard Method - CC BY-NC-SA 4.0
 - 15.4: Using Excel and R for an Analysis of Variance - CC BY-NC-SA 4.0
 - 15.5: Developing a Standard Method (Exercises) - CC BY-NC-SA 4.0
 - 15.6: Developing a Standard Method (Summary) - CC BY-NC-SA 4.0
- 16: Quality Assurance - CC BY-NC-SA 4.0
 - 16.1: The Analytical Perspective—Revisited - CC BY-NC-SA 4.0
 - 16.2: Quality Control - CC BY-NC-SA 4.0
 - 16.3: Quality Assessment - CC BY-NC-SA 4.0
 - 16.4: Evaluating Quality Assurance Data - CC BY-NC-SA 4.0
 - 16.5: Quality Assurance (Exercises) - CC BY-NC-SA 4.0
- 17: Additional Resources - CC BY-NC-SA 4.0
- 18: Back Matter - *Undeclared*
 - Front Matter - *Undeclared*
 - Table of Contents - *Undeclared*
- Back Matter - *Undeclared*
 - Index - *Undeclared*
 - Glossary - *Undeclared*
 - Detailed Licensing - *Undeclared*