ANALYTICAL CHEMISTRY

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Analytical Chemistry

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CHAPTER OVERVIEW

1: Front Matter

DePauw University

Analytical Chemistry 2.1

David Harvey

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Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation nor the US Department of Education.

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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.

- 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.

- 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.

- 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.

CHAPTER OVERVIEW

2: Introduction to Analytical Chemistry

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

Topic hierarchy

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

Contributors

- David Harvey (DePauw University)

2.1: What is Analytical Chemistry?

1.1: What is Analytical Chemistry?

2.2: The Analytical Perspective

1.2: The Analytical Perspective

2.3: Common Analytical Problems

1.3: Common Analytical Problems

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1.E: Introduction to Analytical Chemistry (Exercises)

2.5: Introduction to Analytical Chemistry (Summary)

1.S: Introduction to Analytical Chemistry (Summary)

CHAPTER OVERVIEW

3: Basic Tools of Analytical Chemistry

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

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

Contributors

- David Harvey (DePauw University)

3.1: Measurements in Analytical Chemistry

2.1: Measurements in Analytical Chemistry

3.2: Concentration

2.2: Concentration

3.3: Stoichiometric Calculations

2.3: Stoichiometric Calculations

3.4: Basic Equipment

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2.5: Preparing Solutions

3.6: Spreadsheets and Computational Software

2.6: Spreadsheets and Computational Software

3.7: The Laboratory Notebook

2.7: The Laboratory Notebook

3.8: Basic Tools of Analytical Chemistry (Exercises)

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

3.9: Basic Tools of Analytical Chemistry (Summary)

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

CHAPTER OVERVIEW

4: The Vocabulary of Analytical Chemistry

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

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

Contributors

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4.1: Analysis, Determination, and Measurement

3.1: Analysis, Determination, and Measurement

4.2: Techniques, Methods, Procedures, and Protocols

3.2: Techniques, Methods, Procedures, and Protocols

4.3: Classifying Analytical Techniques

4.4: Selecting an Analytical Method

4.5: Developing the Procedure

4.6: Protocols

3.6: Protocols

4.7: The Importance of Analytical Methodology

4.8: The Vocabulary of Analytical Chemistry (Exercises)

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

4.9: The Vocabulary of Analytical Chemistry (Summary)

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

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.

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

Contributors

- David Harvey (DePauw University)

5.1: Characterizing Measurements and Results

5.2: Characterizing Experimental Errors

4.2: Characterizing Experimental Errors

5.3: Propagation of Uncertainty

5.4: The Distribution of Measurements and Results

4.4: The Distribution of Measurements and Results

5.5: Statistical Analysis of Data

5.6: Statistical Methods for Normal Distributions

4.6: Statistical Methods for Normal Distributions

5.7: Detection Limits

4.7: Detection Limits

5.8: Using Excel and R to Analyze Data

5.9: Evaluating Analytical Data (Exercises)

5.10: Evaluating Analytical Data (Summary)

4.S: Evaluating Analytical Data (Summary)

CHAPTER OVERVIEW

6: Standardizing Analytical Methods

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

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

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

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

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6.1: Analytical Standards

5.1: Analytical Standards

6.2: Calibrating the Signal

5.2: Calibrating the Signal

6.3: Determining the Sensitivity

5.3: Determining the Sensitivity

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5.4: Linear Regression and Calibration Curves

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5.5: Blank Corrections

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5.E: Standardizing Analytical Methods (Exercises)

6.8: Standardizing Analytical Methods (Summary)

5.S: Standardizing Analytical Methods (Summary)

CHAPTER OVERVIEW

7: Equilibrium Chemistry

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

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

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- David Harvey (DePauw University)

7.1: Reversible Reactions and Chemical Equilibria

6.01: Reversible Reactions and Chemical Equilibria

7.2: Thermodynamics and Equilibrium Chemistry

6.02: Thermodynamics and Equilibrium Chemistry

7.3: Manipulating Equilibrium Constants

6.03: Manipulating Equilibrium Constants

7.4: Equilibrium Constants for Chemical Reactions

6.04: Equilibrium Constants for Chemical Reactions

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6.05: Le Châtelier’s Principle

7.6: Ladder Diagrams

6.06: Ladder Diagrams

7.7: Solving Equilibrium Problems

6.07: Solving Equilibrium Problems

7.8: Buffer Solutions

6.08: Buffer Solutions

7.9: Activity Effects

6.09: Activity Effects

7.10: Using Excel and R to Solve Equilibrium Problems

6.10: Using Excel and R to Solve Equilibrium Problems

7.11: Some Final Thoughts on Equilibrium Calculations

6.11: Some Final Thoughts on Equilibrium Calculations

7.12: Equilibrium Chemistry (Exercises)

6.E: Equilibrium Chemistry (Exercises)

7.13: Equilibrium Chemistry (Summary)

6.S: Equilibrium Chemistry (Summary)

CHAPTER OVERVIEW

8: Collecting and Preparing Samples

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

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

Contributors

- David Harvey (DePauw University)

8.1: The Importance of Sampling

7.1: The Importance of Sampling

8.2: Designing a Sampling Plan

7.2: Designing a Sampling Plan

8.3: Implementing the Sampling Plan

7.3: Implementing the Sampling Plan

8.4: Separating the Analyte from Interferents

7.4: Separating the Analyte from Interferents

8.5: General Theory of Separation Efficiency

7.5: General Theory of Separation Efficiency

8.6: Classifying Separation Techniques

7.6: Classifying Separation Techniques

8.7: Liquid–Liquid Extractions

7.7: Liquid–Liquid Extractions

8.8: Separation Versus Preconcentration

7.8: Separation Versus Preconcentration

8.9: Collecting and Preparing Samples (Exercises)

8.10: Collecting and Preparing Samples (Summary)

7.S: Collecting and Preparing Samples (Summary)

CHAPTER OVERVIEW

9: Gravimetric Methods

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

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

Contributors

- David Harvey (DePauw University)

9.1: Overview of Gravimetric Methods

8.1: Overview of Gravimetric Methods

9.2: Precipitation Gravimetry

8.2: Precipitation Gravimetry

9.3: Volatilization Gravimetry

8.3: Volatilization Gravimetry

9.4: Particulate Gravimetry

8.4: Particulate Gravimetry

9.5: Gravimetric Methods (Exercises)

9.6: Gravimetric Methods (Summary)

8.S: Gravimetric Methods (Summary)

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10: Titrimetric Methods

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

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

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

Contributors

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10.1: Overview of Titrimetry

9.1: Overview of Titrimetry

10.2: Acid–Base Titrations

9.2: Acid–Base Titrations

10.3: Complexation Titrations

9.3: Complexation Titrations

10.4: Redox Titrations

9.4: Redox Titrations

10.5: Precipitation Titrations

9.5: Precipitation Titrations

10.6: Titrimetric Methods (Exercises)

9.E: Titrimetric Methods (Exercises)

10.7: Titrimetric Methods (Summary)

9.S: Titrimetric Methods (Summary)

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11: Spectroscopic Methods

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

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

Contributors

- David Harvey (DePauw University)

11.1: Overview of Spectroscopy

10.1: Overview of Spectroscopy

11.2: Spectroscopy Based on Absorption

10.2: Spectroscopy Based on Absorption

11.03: UV

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11.3: UV/Vis and IR Spectroscopy

11.4: Atomic Absorption Spectroscopy

10.4: Atomic Absorption Spectroscopy

11.5: Emission Spectroscopy

11.6: Photoluminescence Spectroscopy

10.6: Photoluminescence Spectroscopy

11.7: Atomic Emission Spectroscopy

10.7: Atomic Emission Spectroscopy

11.8: Spectroscopy Based on Scattering

10.8: Spectroscopy Based on Scattering

11.9: Spectroscopic Methods (Exercises)

11.10: Spectroscopic Methods (Summary)

CHAPTER OVERVIEW

12: Electrochemical Methods

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

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

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

Contributors

- David Harvey (DePauw University)

12.1: Overview of Electrochemistry

11.1: Overview of Electrochemistry

12.2: Potentiometric Methods

11.2: Potentiometric Methods

12.3: Coulometric Methods

12.4: Voltammetric Methods

12.5: Electrochemical Methods (Exercises)

11.E: Electrochemical Methods (Exercises)

12.6: Electrochemical Methods (Summary)

11.S: Electrochemical Methods (Summary)

CHAPTER OVERVIEW

13: Chromatographic

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

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

Contributors

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13.1: Overview of Analytical Separations

12.1: Overview of Analytical Separations

13.2: General Theory of Column Chromatography

13.3: Optimizing Chromatographic Separations

13.4: Gas Chromatography

13.5: High-Performance Liquid Chromatography

12.5: High-Performance Liquid Chromatography

13.6: Other Forms of Liquid Chromatography

12.6: Other Forms of Liquid Chromatography

13.7: Electrophoresis

12.7: Electrophoresis

13.8: Chromatographic and Electrophoretic Methods (Exercises)

13.9: Chromatographic and Electrophoretic Methods (Summary)

12.S: Chromatographic and Electrophoretic Methods (Summary)

CHAPTER OVERVIEW

14: Kinetic Methods

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

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

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

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

Contributors

- David Harvey (DePauw University)

14.1: Kinetic Methods Versus Equilibrium Methods

13.1: Kinetic Methods Versus Equilibrium Methods

14.2: Chemical Kinetics

13.2: Chemical Kinetics

14.3: Radiochemistry

14.4: Flow Injection Analysis

14.5: Kinetic Methods (Exercises)

14.6: Kinetic Methods (Summary)

CHAPTER OVERVIEW

15: Developing a Standard Method

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

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15.1: Optimizing the Experimental Procedure

14.1: Optimizing the Experimental Procedure

15.2: Verifying the Method

15.3: Validating the Method as a Standard Method

15.4: Using Excel and R for an Analysis of Variance

14.4: Using Excel and R for an Analysis of Variance

15.5: Developing a Standard Method (Exercises)

14.E: Developing a Standard Method (Exercises)

15.6: Developing a Standard Method (Summary)

CHAPTER OVERVIEW

16: Quality Assurance

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

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

Contributors

- David Harvey (DePauw University)

16.1: The Analytical Perspective—Revisited

15.1: The Analytical Perspective—Revisited

16.2: Quality Control

15.2: Quality Control

16.3: Quality Assessment

15.3: Quality Assessment

16.4: Evaluating Quality Assurance Data

16.5: Quality Assurance (Exercises)

15.E: Quality Assurance (Exercises)

17: Additional Resources

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

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

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- Stone, C. A.; Mumaw, L. D. “Practical Experiments in Statistics,” *J. Chem. Educ.* **1995**, *72*, 518–524.
- Thomasson, K.; Lofthus-Merschman, S.; Humbert, M.; Kulevsky, N. “Applying Statistics in the Undergraduate Chemistry Laboratory: Experiments with Food Dyes,” *J. Chem. Educ.* **1998**, *75*, 231–233.
- Vitha, M. F.; Carr, P. W. “A Laboratory Exercise in Statistical Analysis of Data,” *J. Chem. Educ.* **1997**, *74*, 998–1000.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

The following papers provide insight into visualizing data.

- Analytical Methods Committee “Representing data distributions with kernel density estimates,” AMC Technical Brief, March 2006 (http://www.rsc.org/lap/rsccom/amc/amc_index.htm).

- Frigge, M.; Hoaglin, D. C.; Iglewicz, B. “Some Implementations of the Boxplot,” *The American Statistician* **1989**, *43*, 50–54.

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

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

Chapter 5

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

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

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

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

The following articles providing more details about linear regression.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Chapter 6

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

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

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

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

The following texts provide additional coverage of equilibrium chemistry.

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

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

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

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

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

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

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

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

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

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

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

- Clark, R. W.; Bonicamp, J. M. "The Ksp-Solubility Conundrum," *J. Chem. Educ.* **1998**, *75*, 1182–1185.
- de Levie, R. "On Teaching Ionic Activity Effects: What, When, and Where?" *J. Chem. Educ.* **2005**, *82*, 878–884.
- McCarty, C. G.; Vitz, E. "pH Paradoxes: Demonstrating That It Is Not True That pH = –log[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 pH ≈ (pK_{a1} + pK_{a2})/2 Using Autoprotolysis of HA^-: Doubtful Value of the Supposedly More Rigorous Equation," *J. Chem. Educ.* **2000**, *77*, 1183–1184. See, also, an exchange of letters between J. J. Roberts and S. J. Hawkes, *J. Chem. Educ.* **2002**, *79*, 161–162.

Chapter 7

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

- Bauer, C. F. "Sampling Error Lecture Demonstration," *J. Chem. Educ.* **1985**, *62*, 253.
- Canaes, L. S.; Brancalion, M. L.; Rossi, A. V.; Rath, S. "Using Candy Samples to Learn About Sampling Techniques and Statistical Evaluation of Data," *J. Chem. Educ.* **2008**, *85*, 1083–1088.
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- “Extract-Clean™ SPE Sample Preparation Guide Volume 1”, Bulletin No. 83, Alltech Associates, Inc. Deerfield, IL.
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- “Terminology—The key to understanding analytical science. Part 2: Sampling and sample preparation,” AMC Technical Brief No. 19, March 2005 (link).
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- “What is uncertainty from sampling, and why is it important?” AMC Technical Brief No. 16A, June 2004 (link).
- “Analytical and sampling strategy, fitness for purpose, and computer games,” AMC Technical Brief No. 20, August 2005 (link).
- “Measurement uncertainty arising from sampling: the new Eurachem Guide,” AMC Technical Brief No. 31, July 2008 (link).
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- Brittain, H. G. "Particle-Size Distribution II: The Problem of Sampling Powdered Solids," *Pharm. Technol.* July **2002**, 67–73.
- Ramsey, M. H. "Measurement Uncertainty Arising from Sampling: Implications for the Objectives of Geoanalysis," *Analyst*, **1997**, *122*, 1255–1260.
- Seiler, T-B; Schulze, T.; Hollert, H. "The risk of altering soil and sediment samples upon extract preparation for analytical and bio-analytical investigations—a review," *Anal. Bioanal. Chem.* **2008**, 390, 1975–1985.

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- "Guide to Solid Phase Extraction," Bulletin 910, Sigma-Aldrich, 1998.
- "Solid Phase Microextraction: Theory and Optimization of Conditions," Bulletin 923, Sigma-Aldrich, 1998.
- *Microwave-Enhanced Chemistry: Fundamentals, Sample Preparation, and Applications*, Kingston, H. M.; Haswell, S. J., eds.; American Chemical Society: Washington, D.C., 1997.
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Chapter 8

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

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- Bassett, J.; Denney, R. C.; Jeffery, G. H.; Mendham, J. *Vogel's Textbook of Quantitative Inorganic Analysis*, Longman: London, 4th Ed., 1981.
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Chapter 9

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

Acid–base titrimetry

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Complexation Titrimetry

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- Fulton, R.; Ross, M.; Schroeder, K. “Spectrophotometric Titration of a Mixture of Calcium and Magnesium,” *J. Chem. Educ.* **1986**, *63*, 721–723.
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Precipitation Titrimetry

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

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

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

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

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

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

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

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

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

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

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

Chapter 10

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

UV/Vis Spectroscopy

- Abney, J. R.; Scalettar, B. A. “Saving Your Students’ Skin. Undergraduate Experiments That Probe UV Protection by Sunscreens and Sunglasses,” *J. Chem. Educ.* **1998**, *75*, 757–760.
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- Domínguez, A., Fernández, A.; González, N.; Iglesias, E.; Montenegro, L. “Determination of Critical Micelle Concentration of Some Surfactants by Three Techniques,” *J. Chem. Educ.* **1997**, *74*, 1227–1231.
- Gilbert, D. D. “Determining Optimum Spectral Bandwidth,” *J. Chem. Educ.* **1991**, *68*, A278–A281.
- Han, J.; Story, T.; Han, G. “A Spectrophotometric Method for Quantitative Determination of Bromine Using Tris(2-carboxyethyl)phophine,” *J. Chem. Educ.* **1999**, *76*, 976–977.
- Higginbotham, C.; Pike, C. F.; Rice, J, K. “Spectroscopy in Sol-Gel Matricies,” *J. Chem. Educ.* **1998**, *75*, 461–464.
- Hill, Z. D.; MacCarthy, P. “Novel Approach to Job’s Method,” *J. Chem. Educ.* **1986**, *63*, 162–167.
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- Tello-Solis, S. R. "Thermal Unfolding of Lysozyme Studied by UV Difference Spectroscopy," *Chem. Educator* **2008**, *13*, 16–18.
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Chapter 11

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Chapter 12

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