Additional Resources

Resource Overview

Gathered here are three types of resources: additional readings from the analytical literature that extend and supplement topics covered in the textbook; suggested experiments, mostly from the *Journal of Chemical Education* and *The Chemical Educator*, that provide practical examples of concepts in the textbook; and electronic resources that help illustrate concepts from the textbook. Although primarily intended for the use of instructors, these resources also benefit students who wish to pursue a topic at more depth. Materials are organized by chapter with the exception of the last heading, which catalogs active learning materials developed by and made available through the Analytical Sciences Digital Library.

Chapter 1

The role of analytical chemistry within the broader discipline of chemistry has been discussed by many prominent analytical chemists; several notable examples are listed here.

- Baiulescu, G. E.; Patroescu, C; Chalmers, R. A. *Education and Teaching in Analytical Chemistry*, Ellis Horwood: Chichester, 1982.
- de Haseth, J. "What is Analytical Chemistry?," *Spectroscopy* **1990**, *5*, 19–21.
- Heiftje, G. M. "The Two Sides of Analytical Chemistry," *Anal. Chem.* **1985**, *57*, 256A–267A.
- Heiftje, G. M. "But is it analytical chemistry?," *Am. Lab.* **1993**, October, 53–61.
- Kissinger, P. T. "Analytical Chemistry—What is It? Why Teach It?," *Trends Anal. Chem.* **1992**, *11*, 57–57.
- Laitinen, H. A.; Ewing, G. (eds.) *A History of Analytical Chemistry*, The Division of Analytical Chemistry of the American Chemical Society: Washington, D. C., 1972.
- Laitinen, H. A. "Analytical Chemistry in a Changing World," *Anal. Chem.* **1980**, *52*, 605A–609A.
- Laitinen, H. A. "History of Analytical Chemistry in the U. S. A.," *Talanta*, **1989**, *36*, 1–9.
- McLafferty, F. W. "Analytical Chemistry: Historic and Modern," *Acc. Chem. Res.* **1990**, *23*, 63–64.
- Mottola, H. A. "The Interdisciplinary and Multidisciplinary Nature of Contemporary Analytical Chemistry and its Core Components," *Anal. Chim. Acta* **1991**, *242*, 1–3.
- Noble, D. "From Wet Chemistry to Instrumental Analysis: A Perspective on Analytical Sciences," *Anal. Chem.* **1994**, *66*, 251A–263A.
- Tyson, J. *Analysis: What Analytical Chemists Do*, Royal Society of Chemistry: Cambridge, England 1988.

For additional discussion of clinical assays based on paper-based microfluidic devices, see the following papers.

- Ellerbee, A. K.; Phillips, S. T.; Siegel, A. C.; Mirica, K. A.; Martinez, A. W.; Striehl, P.; Jain, N.; Prentiss, M.; Whitesides, G. M. "Quantifying Colorimetric Assays in Paper-Based Microfluidic Devices by Measuring the Transmission of Light Through Paper," *Anal. Chem.* **2009**, *81*, 8447–8452.
- Martinez, A. W.; Phillips, S. T.; Whitesides, G. M. "Diagnostics for the Developing World: Microfluidic Paper-Based Analytical Devices," *Anal. Chem.* **2010**, *82*, 3–10.

This textbook provides one introduction to the discipline of analytical chemistry. There are other textbooks for introductory courses in analytical chemistry and you may find it useful to consult them when you encounter a difficult concept; often a fresh perspective will help crystallize your understanding. The textbooks listed here are excellent resources.

- Enke, C. *The Art and Science of Chemical Analysis*, Wiley: New York.
- Christian, G. D.; Dasgupta, P, K.; Schug; K. A. *Analytical Chemistry*, Wiley: New York.
- Harris, D. *Quantitative Chemical Analysis*, W. H. Freeman and Company: New York.
- Kellner, R.; Mermet, J.-M.; Otto, M.; Valcárcel, M.; Widmer, H. M. *Analytical Chemistry*, Wiley-VCH: Weinheim, Germany.

- Rubinson, J. F.; Rubinson, K. A. *Contemporary Chemical Analysis*, Prentice Hall: Upper Saddle River, NJ.
- Skoog, D. A.; West, D. M.; Holler, F. J. *Fundamentals of Analytical Chemistry*, Saunders: Philadelphia.

To explore the practice of modern analytical chemistry there is no better resource than the primary literature. The following journals publish broadly in the area of analytical chemistry.

- Analytical and Bioanalytical Chemistry
- Analytical Chemistry
- Analytical Chimica Acta
- Analyst
- Talanta

Chapter 2

The following two web sites contain useful information about the SI system of units.

- http://www.bipm.org/en/home/ – The home page for the Bureau International des Poids and Measures.
- http://physics.nist.gov/cuu/Units/index.html – The National Institute of Standards and Technology's introduction to SI units.

For a chemist's perspective on the SI units for mass and amount, consult the following papers.

- Davis, R. S. "What is a Kilogram in the Revised International System of Units (SI)?", *J. Chem. Educ.* **2015**, *92*, 1604–1609.
- Freeman, R. D. "SI for Chemists: Persistent Problems, Solid Solutions," *J. Chem. Educ.* **2003**, *80*, 16–20.
- Gorin, G. "Mole, Mole per Liter, and Molar: A Primer on SI and Related Units for Chemistry Students," *J. Chem. Educ.* **2003**, *80*, 103–104.

Discussions regarding possible changes in the SI base units are reviewed in this article.

- Chao, L. S.; Schlamminger, S.; Newell, D. B.; Pratt, J. R.; Seifert, F.; Zhang, X.; Sineriz, M. L.; Haddad, D. "A LEGO Watt Balance: An Apparatus to Determine a Mass Based on the New SI," arXiv:1412.1699 [physics.ins-det].
- Fraundorf, P. "A Multiple of 12 for Avogadro," arXiv:1201.5537 [physics.gen-ph].
- Kemsley, J. "Rethinking the Mole and Kilogram," *C&E News*, August 25, 2014, p. 25.

The following are useful resources for maintaining a laboratory notebook and for preparing laboratory reports.

- Coghill, A. M.; Garson, L. M. (eds) *The ACS Style Guide: Effective Communication of Scientific Information*, 3rd Edition, American Chemical Society: Washington, D. C.; 2006.
- Kanare, H. M. *Writing the Laboratory Notebook*, American Chemical Society: Washington, D. C.; 1985.

The following texts provide instructions for using spreadsheets in analytical chemistry.

- de Levie, R. *How to Use Excel in Analytical Chemistry and in General Scientific Data Analysis*, Cambridge University Press: Cambridge, UK, 2001.
- Diamond, D.; Hanratty, V. C. A., *Spreadsheet Applications in Chemistry*, Wiley-Interscience: New York, 1997.
- Feiser, H. *Concepts and Calculations in Analytical Chemistry: A Spreadsheet Approach*, CRC Press: Boca Raton, FL, 1992.

The following classic textbook emphasizes the application of intuitive thinking to the solving of problems.

- Harte, J. *Consider a Spherical Cow: A Course in Environmental Problem Solving*, University Science Books: Sausalito, CA, 1988.

Chapter 3

The International Union of Pure and Applied Chemistry (IUPAC) maintains a web-based compendium of analytical terminology. You can find it at the following web site.

- http://old.iupac.org/publications/analytical_compendium/

The following papers provide alternative schemes for classifying analytical methods.

- Booksh, K. S.; Kowalski, B. R. “Theory of Analytical Chemistry,” *Anal. Chem.* **1994**, *66*, 782A–791A.
- Phillips, J. B. “Classification of Analytical Methods,” *Anal. Chem.* **1981**, *53*, 1463A–1470A.
- Valcárcel, M.; Luque de Castro, M. D. “A Hierarchical Approach to Analytical Chemistry,” *Trends Anal. Chem.* **1995**, *14*, 242–250.
- Valcárcel, M.; Simonet, B. M. “Types of Analytical Information and Their Mutual Relationships,” *Trends Anal. Chem.* **1995**, *14*, 490–495.

Further details on criteria for evaluating analytical methods are found in the following series of papers.

- Wilson, A. L. “The Performance-Characteristics of Analytical Methods”, Part I-*Talanta*, **1970**, *17*, 21–29; Part II-*Talanta*, **1970**, *17*, 31–44; Part III-*Talanta*, **1973**, *20*, 725–732; Part IV-*Talanta*, **1974**, *21*, 1109–1121.

For a point/counterpoint debate on the meaning of sensitivity consult the following two papers and two letters of response.

- Ekins, R.; Edwards, P. “On the Meaning of ‘Sensitivity’,” *Clin. Chem.* **1997**, *43*, 1824–1831.
- Ekins, R.; Edwards, P. “On the Meaning of ‘Sensitivity:’ A Rejoinder,” *Clin. Chem.* **1998**, *44*, 1773–1776.
- Pardue, H. L. “The Inseparable Triangle: Analytical Sensitivity, Measurement Uncertainty, and Quantitative Resolution,” *Clin. Chem.* **1997**, *43*, 1831–1837.
- Pardue, H. L. “Reply to ‘On the Meaning of ‘Sensitivity:’ A Rejoinder’,” *Clin. Chem.* **1998**, *44*, 1776–1778.

Several texts provide analytical procedures for specific analytes in well-defined matrices.

- Basset, J.; Denney, R. C.; Jeffery, G. H.; Mendham, J. *Vogel’s Textbook of Quantitative Inorganic Analysis*, 4th Edition; Longman: London, 1981.
- Csuros, M. *Environmental Sampling and Analysis for Technicians*, Lewis: Boca Raton, 1994.
- Keith, L. H. (ed) *Compilation of EPA’s Sampling and Analysis Methods*, Lewis: Boca Raton, 1996
- Rump, H. H.; Krist, H. *Laboratory Methods for the Examination of Water, Wastewater and Soil*, VCH Publishers: NY, 1988.
- *Standard Methods for the Analysis of Waters and Wastewaters*, 21st Edition, American Public Health Association: Washington, D. C.; 2005.

For a review of the importance of analytical methodology in today’s regulatory environment, consult the following text.

- Miller, J. M.; Crowther, J. B. (eds) *Analytical Chemistry in a GMP Environment*, John Wiley & Sons: New York, 2000.

Chapter 4

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

- Bularzik, J. "The Penny Experiment Revisited: An Illustration of Significant Figures, Accuracy, Precision, and Data Analysis," *J. Chem. Educ.* **2007**, *84*, 1456–1458.
- Columbia, M. R. "The Statistics of Coffee: 1. Evaluation of Trace Metals for Establishing a Coffee's Country of Origin Based on a Means Comparison," *Chem. Educator* **2007**, *12*, 260–262.
- Cunningham, C. C.; Brown, G. R.; St Pierre, L. E. "Evaluation of Experimental Data," *J. Chem. Educ.* **1981**, *58*, 509–511.
- Edminston, P. L.; Williams, T. R. "An Analytical Laboratory Experiment in Error Analysis: Repeated Determination of Glucose Using Commercial Glucometers," *J. Chem. Educ.* **2000**, *77*, 377–379.
- Gordus, A. A. "Statistical Evaluation of Class Data for Two Buret Readings," *J. Chem. Educ.* **1987**, *64*, 376–377.
- Harvey, D. T. "Statistical Evaluation of Acid/Base Indicators," *J. Chem. Educ.* **1991**, *68*, 329–331.
- Hibbert, D. B. "Teaching modern data analysis with The Royal Austrian Chemical Institute's titration competition," *Aust. J. Ed. Chem.* **2006**, *66*, 5–11.
- Johll, M. E.; Poister, D.; Ferguson, J. "Statistical Comparison of Multiple Methods for the Determination of Dissolved Oxygen Levels in Natural Water," *Chem. Educator* **2002**, *7*, 146–148.
- Jordon, A. D. "Which Method is Most Precise; Which is Most Accurate?," *J. Chem. Educ.* **2007**, *84*, 1459–1460.
- Olsen, R. J. "Using Pooled Data and Data Visualization To Introduce Statistical Concepts in the General Chemistry Laboratory," *J. Chem. Educ.* **2008**, *85*, 544–545.
- O'Reilley, J. E. "The Length of a Pestle," *J. Chem. Educ.* **1986**, *63*, 894–896.
- Overway, K. "Population versus Sampling Statistics: A Spreadsheet Exercise," *J. Chem. Educ.* **2008** *85*, 749.
- Paselk, R. A. "An Experiment for Introducing Statistics to Students of Analytical and Clinical Chemistry," *J. Chem. Educ.* **1985**, *62*, 536.
- Puignou, L.; Llauradó, M. "An Experimental Introduction to Interlaboratory Exercises in Analytical Chemistry," *J. Chem. Educ.* **2005**, *82*, 1079–1081.
- Quintar, S. E.; Santagata, J. P.; Villegas, O. I.; Cortinez, V. A. "Detection of Method Effects on Quality of Analytical Data," *J. Chem. Educ.* **2003**, *80*, 326–329.
- Richardson, T. H. "Reproducible Bad Data for Instruction in Statistical Methods," *J. Chem. Educ.* **1991**, *68*, 310–311.
- Salzsieder, J. C. "Statistical Analysis Experiment for Freshman Chemistry Lab," *J. Chem. Educ.* **1995**, *72*, 623.
- Samide, M. J. "Statistical Comparison of Data in the Analytical Laboratory," *J. Chem. Educ.* **2004**, *81*, 1641–1643.
- Sheeran, D. "Copper Content in Synthetic Copper Carbonate: A Statistical Comparison of Experimental and Expected Results," *J. Chem. Educ.* **1998**, *75*, 453–456.

- Spencer, R. D. "The Dependence of Strength in Plastics upon Polymer Chain Length and Chain Orientation," *J. Chem. Educ.* **1984**, *61*, 555–563.
- Stolzberg, R. J. "Do New Pennies Lose Their Shells? Hypothesis Testing in the Sophomore Analytical Chemistry Laboratory," *J. Chem. Educ.* **1998**, *75*, 1453–1455.
- Stone, C. A.; Mumaw, L. D. "Practical Experiments in Statistics," *J. Chem. Educ.* **1995**, *72*, 518–524.
- Thomasson, K.; Lofthus-Merschman, S.; Humbert, M.; Kulevsky, N. "Applying Statistics in the Undergraduate Chemistry Laboratory: Experiments with Food Dyes," *J. Chem. Educ.* **1998**, *75*, 231–233.
- Vitha, M. F.; Carr, P. W. "A Laboratory Exercise in Statistical Analysis of Data," *J. Chem. Educ.* **1997**, *74*, 998–1000.

A more comprehensive discussion of the analysis of data, which includes all topics considered in this chapter as well as additional material, are found in many 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.
- 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 "Rogues and Suspects: How to Tackle Outliers," AMCTB 39, 2009.
- Analytical Methods Committee "Robust statistics: a method of coping with outliers," AMCTB 6, 2001.
- Analytical Methods Committee "Using the Grubbs and Cochran tests to identify outliers," *Anal. Methods*, **2015**, *7*, 7948–7950.
- 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.

- Analytical Methods Committee "Optimizing your uncertainty—a case study," AMCTB 32, 2008.
- Analytical Methods Committee "Dark Uncertainty," AMCTB 53, 2012.
- Analytical Methods Committee "What causes most errors in chemical analysis?" AMCTB 56, 2013.
- Andraos, J. "On the Propagation of Statistical Errors for a Function of Several Variables," *J. Chem. Educ*. **1996**, *73*, 150–154.
- Donato, H.; Metz, C. "A Direct Method for the Propagation of Error Using a Personal Computer Spreadsheet Program," *J. Chem. Educ.* **1988**, *65*, 867–868.
- Gordon, R.; Pickering, M.; Bisson, D. "Uncertainty Analysis by the 'Worst Case' Method," *J. Chem. Educ.* **1984**, *61*, 780–781.
- Guare, C. J. "Error, Precision and Uncertainty," *J. Chem. Educ.* **1991**, *68*, 649–652.
- Guedens, W. J.; Yperman, J.; Mullens, J.; Van Poucke, L. C.; Pauwels, E. J. "Statistical Analysis of Errors: A Practical Approach for an Undergraduate Chemistry Lab Part 1. The Concept," *J. Chem. Educ.* **1993**, *70*, 776–779
- Guedens, W. J.; Yperman, J.; Mullens, J.; Van Poucke, L. C.; Pauwels, E. J. "Statistical Analysis of Errors: A Practical Approach for an Undergraduate Chemistry Lab Part 2. Some Worked Examples," *J. Chem. Educ.* **1993**, *70*, 838–841.
- Heydorn, K. "Detecting Errors in Micro and Trace Analysis by Using Statistics," *Anal. Chim. Acta* **1993**, *283*, 494–499.
- Hund, E.; Massart, D. L.; Smeyers-Verbeke, J. "Operational definitions of uncertainty," *Trends Anal. Chem.* **2001**, *20*, 394–406.
- Kragten, J. "Calculating Standard Deviations and Confidence Intervals with a Universally Applicable Spreadsheet Technique," *Analyst* **1994**, *119*, 2161–2165.
- Taylor, B. N.; Kuyatt, C. E. "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, 1994.

- 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.
- 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.
- Fonollosa, J.; Vergara, A; Huerta, R.; Marco, S. "Estimation of the limit of detection using information theory measures," *Anal. Chim. Acta* **2014**, *810*, 1–9.
- 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 articles provide thoughts on the limitations of statistical analysis based on significance testing.

- Analytical Methods Committee "Significance, importance, and power," AMCTB 38, 2009.
- Analytical Methods Committee "An introduction to non-parametric statistics," AMCTB 57, 2013.
- Berger, J. O.; Berry, D. A. "Statistical Analysis and the Illusion of Objectivity," *Am. Sci.* **1988**, *76*, 159–165.
- Kryzwinski, M. "Importance of being uncertain," *Nat. Methods* **2013**, *10*, 809–810.
- Kryzwinski, M. "Significance, P values, and t-tests," *Nat. Methods* **2013**, *10*, 1041–1042.
- Kryzwinski, M. "Power and sample size," *Nat. Methods* **2013**, *10*, 1139–1140.
- Leek, J. T.; Peng, R. D. "What is the question?," *Science* **2015**, *347*, 1314–1315.

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).
- Kelling, K. B.; Pavur, R. J. "A Comparative Study of the Reliability of Nine Statistical Software Packages," *Comput. Statist. Data Anal.* **2007**, *51*, 3811–3831.

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

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.
- Analytical Methods Committee "Robust regression: an introduction, "AMCTB 50, 2012.
- 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.
- Mashkina, E.; Oldman, K. B. "Linear Regressions to Which the Standard Formulas do not Apply," *ChemTexts*, **2015**, *1*, 1–11.
- Miller, J. N. "Basic Statistical Methods for Analytical Chemistry. Part 2. Calibration and Regression Methods," *Analyst* **1991**, *116*, 3–14.
- Raposo, F. "Evaluation of analytical calibration based on least-squares linear regression for instrumental techniques: A tutorial review," *Trends Anal. Chem.* **2016**, *77*, 167–185.
- 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.
- Meija, J.; Pagliano, E.; Mester, Z. "Coordinate Swapping in Standard Addition Graphs for Analytical Chemistry: A Simplified Path for Uncertainty Calculation in Linear and Nonlinear Plots," Anal. Chem. 2014, 86, 8563–8567.
- Nimura, Y.; Carr, M. R. "Reduction of the Relative Error in the Standard Additions Method," *Analyst* **1990**, *115*, 1589–1595.

Approaches that combine a standard addition with an internal standard are described in the following paper.

- Jones, W. B.; Donati, G. L.; Calloway, C. P.; Jones, B. T. "Standard Dilution Analysis," Anal. Chem. 2015, 87, 2321–2327.

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.
- 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.
- Tellinghuisen, J. "Least Squares in Calibration: Dealing with Uncertainty in *x*," *Analyst*, **2010**, *135*, 1961–1969.

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.

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- Zielinski, T. J.; Allendoerfer, R. D. "Least Squares Fitting of Nonlinear Data in the Undergraduate Laboratory," *J. Chem. Educ.* **1997**, *74*, 1001–1007.

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- 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.
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- 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.
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- Young, S. H.; Wierzbicki, A. "Mathcad in the Chemistry Curriculum. Linear Least-Squares Regression," *J. Chem. Educ.* **2000**, *77*, 669.
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Chapter 6

The following experiments involve the experimental determination of equilibrium constants, the characterization of buffers, and, in some cases, demonstrations of 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.
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- 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.

- Cepría, G.; Salvatella, L. "General Procedure for the Easy Calculation of pH in an Introductory Course of General or Analytical Chemistry," *J. Chem. Educ.* **2014**, *91*, 524–530.
- 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.
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- 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.

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- 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.
- Glaser, R. E. Delarosa, M. A.; Salau, A. O.; Chicone, C. “Dynamical Approach to Multiequilibria Problems for Mixtures of Acids and Their Conjugate Bases,” *J. Chem. Educ.* **2014**, *91*, 1009–1016.
- 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 $pH \approx (pK_{a1} + pK_{a2})/2$ 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.
- Clement, R. E. "Environmental Sampling for Trace Analysis," *Anal. Chem.* **1992**, *64*, 1076A–1081A.
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- Fillman, K. L.; Palkendo, J. A. "Collection, Extraction, and Analysis of Lead in Atmospheric Particles," *J. Chem. Edu.* **2014**, *91*, 590–592.
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- Guy, R. D.; Ramaley, L.; Wentzell, P. D. "An Experiment in the Sampling of Solids for Chemical Analysis", J. Chem. Educ. 1998, 75, 1028–1033.
- Hartman, J. R. "An In-Class Experiment to Illustrate the Importance of Sampling Techniques and Statistical Analysis of Data to Quantitative Analysis Students," *J. Chem. Educ.* **2000**, *77*, 1017–1018.
- Harvey, D. T. "Two Experiments Illustrating the Importance of Sampling in a Quantitative Chemical Analysis," *J. Chem. Educ.* **2002**, *79*, 360–363.
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- Kratochvil, B.; Reid, R. S.; Harris, W. E. "Sampling Error in a Particulate Mixture", *J. Chem. Educ.* **1980**, *57*, 518–520.
- Ross, M. R. "A Classroom Exercise in Sampling Technique," *J. Chem. Educ.* **2000**, *77*, 1015–1016.
- Settle, F. A.; Pleva, M. "The Weakest Link Exercise," *Anal. Chem.* **1999**, *71*, 538A–540A.
- Vitt, J. E.; Engstrom, R. C. "Effect of Sample Size on Sampling Error," *J. Chem. Educ.* **1999**, *76*, 99–100.

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

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

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

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

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

- "Terminology—The key to understanding analytical science. Part 2: Sampling and sample preparation," AMCTB 19, 2005.
- Majors, R. E. "Nomenclature for Sampling in Analytical Chemistry" *LC•GC* **1992**, *10*, 500–506.

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

- Analytical Methods Committee "What is uncertainty from sampling, and why is it important?" AMCTB 16A, 2004.
- Analytical Methods Committee "Analytical and sampling strategy, fitness for purpose, and computer games," AMCTB 20, 2005.
- Analytical Methods Committee "Measurement uncertainty arising from sampling: the new Eurachem Guide," AMCTB No. 31, 2008.
- Analytical Methods Committee "The importance, for regulation, of uncertainty from sampling," AMCTB 42, 2009.
- Analytical Methods Committee "Estimating sampling uncertainty—how many duplicate samples are needed?" AMCTB 58, 2014.
- Analytical Methods Committee "Random samples," AMCTB 60, 2014.
- Analytical Methods Committee "Sampling theory and sampling uncertainty," AMCTB 71, 2015.
- *Sampling for Analytical Purpose*, Gy, P. ed., Wiley: NY, 1998.
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- Cohen, R. D. "How the Size of a Random Sample Affects How Accurately It Represents a Population," *J. Chem. Educ.* **1992**, *74*, 1130–1132.
- Efstathiou, C. E. "On the sampling variance of ultra-dilute solutions," *Talanta* **2000**, *52*, 711–715.
- Esbensen, K. H.; Wagner, C. "Theory of sampling (TOS) *versus* measurement uncertainty (MU)–A call for integration," *TRAC-Trend. Anal. Chem.* **2014**, *57*, 93–106.
- Gerlach, R. W.; Dobb, D. E.; Raab, G. A.; Nocerino, J. M. *J. Chemom.* **2002**, *16*, 321–328.
- Gy, P. M. *Sampling of Particulate Materials: Theory and Practice*; Elsevier: Amsterdam, 1979.
- Gy, P. M. *Sampling of Heterogeneous and Dynamic Materials: Theories of Heterogeneity, Sampling and Homogenizing*; Elsevier: Amsterdam, 1992.

- Harrington, B.; Nickerson, B.; Guo, M. X.; Barber, M.; Giamalva, D.; Lee, C.; Scrivens, G. "Sample Preparation Composite and Replicate Strategy for Assay of Solid Oral Drug Products," *Anal. Chem.* **2014**, *86*, 11930–11936.
- Kratochvil, B.; Taylor, J. K. "Sampling for Chemical Analysis," *Anal. Chem.* **1981**, *53*, 924A–938A.
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- Meyer, V. R. *LC•GC* **2002**, *20*, 106–112.
- Rohlf, F. J.; Akçakaya, H. R.; Ferraro, S. P. "Optimizing Composite Sampling Protocols," *Environ. Sci. Technol.* **1996**, *30*, 2899–2905.
- Smith, R.; James, G. V. *The Sampling of Bulk Materials*; Royal Society of Chemistry: London, 1981.

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

- Barceló, D.; Hennion, M. C. "Sampling of Polar Pesticides from Water Matrices," *Anal. Chim. Acta* **1997**, *338*, 3–18.
- Batley, G. E.; Gardner, D. "Sampling and Storage of Natural Waters for Trace Metal Analysis," *Wat. Res.* **1977**, *11*, 745–756.
- Benoit, G.; Hunter, K. S.; Rozan, T. F. "Sources of Trace Metal Contamination Artifacts during Collection, Handling, and Analysis of Freshwaters," *Anal. Chem.* **1997**, *69*, 1006–1011
- Brittain, H. G. "Particle-Size Distribution II: The Problem of Sampling Powdered Solids," *Pharm. Technol.* July **2002**, 67–73.
- Ramsey, M. H. "Measurement Uncertainty Arising from Sampling: Implications for the Objectives of Geoanalysis," *Analyst*, **1997**, *122*, 1255–1260.
- Seiler, T-B; Schulze, T.; Hollert, H. "The risk of altering soil and sediment samples upon extract preparation for analytical and bio-analytical investigations—a review," *Anal. Bioanal. Chem.* **2008**, 390, 1975–1985.

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

- "Guide to Solid Phase Extraction," Bulletin 910, Sigma-Aldrich, 1998.
- "Solid Phase Microextraction: Theory and Optimization of Conditions," Bulletin 923, Sigma-Aldrich, 1998.
- *Microwave-Enhanced Chemistry: Fundamentals, Sample Preparation, and Applications*, Kingston, H. M.; Haswell, S. J., eds.; American Chemical Society: Washington, D.C., 1997.
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- Bettiol, C.; Stievano, L.; Bertelle, M.; Delfino, F.; Argese, E. "Evaluation of microwave-assisted acid extraction procedures for the determination of metal content and potential bioavailability in sediments," *Appl. Geochem.* **2008**, *23*, 1140–1151.
- Compton, T. R. *Direct Preconcentration Techniques*, Oxford Science Publications: Oxford, 1993.
- Compton, T. R. *Complex-Formation Preconcentration Techniques*, Oxford Science Publications: Oxford, 1993.
- Hinshaw, J. V. "Solid-Phase Microextraction," *LC•GC Europe* **2003**, *December*, 2–5.

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- Luque de Castro, M. D.; Priego-Capote, F.; Sánchez-Ávila, N. "Is dialysis alive as a membrane-based separation technique?" *Trends Anal. Chem.* **2008**, *27*, 315–326.
- Mary, P.; Studer, V.; Tabeling, P. "Microfluidic Droplet-Based Liquid–Liquid Extraction," *Anal. Chem.* **2008**, *80*, 2680–2687.
- Miller, J. M. *Separation Methods in Chemical Analysis*, Wiley-Interscience: N. Y.; 1975.
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- Pawliszyn, J. *Solid-Phase Microextraction: Theory and Practice*, Wiley: NY, 1997.
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- Sulcek, Z.; Povondra, P. *Methods of Decomposition in Inorganic Analysis*; CRC Press: Boca Raton, FL, 1989.
- Theis, A. L.; Waldack, A. J.; Hansen, S. M.; Jeannot, M. A. "Headspace Solvent Microextraction," *Anal. Chem.* **2001**, *73*, 5651–5654.
- Thurman, E. M.; Mills, M. S. *Solid-Phase Extraction: Principles and Practice*, Wiley: NY, 1998.
- Zhang, Z.; Yang, M.; Pawliszyn, J. "Solid-Phase Microextraction," *Anal. Chem.* **1994**, *66*, 844A–853A.

Chapter 8

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

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

The following resources provide a general history of gravimetry.

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

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

- Bassett, J.; Denney, R. C.; Jeffery, G. H.; Mendham, J. *Vogel's Textbook of Quantitative Inorganic Analysis*, Longman: London, 4th Ed., 1981.
- Erdey, L. *Gravimetric Analysis*, Pergamon: Oxford, 1965.
- Steymark, A. *Quantitative Organic Microanalysis*, The Blakiston Co.: NY, 1951.
- Wendlandt, W. W. T*hermal Methods of Analysis*, 2nd Ed. Wiley: NY. 1986.

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

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

Chapter 9

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

Acid–base titrimetry

- Boiani, J. A. "The Gran Plot Analysis of an Acid Mixture," *J. Chem. Educ.* **1986**, *63*, 724–726.
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Chapter 10

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Chapter 11

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Chapter 12

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Chapter 14

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- Leggett, D. L. "Instrumental Simplex Optimization," *J. Chem. Educ.* **1983**, *60*, 707–710.
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The following texts and articles provide an excellent discussion of optimization methods based on searching algorithms and mathematical modeling use factorial designs, including a discussion of the relevant calculations. A few of these sources discuss other types of experimental designs.

- Analytical Methods Committee "Experimental design and optimisation (1): an introduction to some basic concepts," AMCTB 24, 2006.
- Analytical Methods Committee "Experimental design and optimisation (2): handling uncontrolled factors," AMCTB 26, 2006.
- Analytical Methods Committee "Experimental design and optimisation (3): some fractional factorial designs," AMCTB 36, 2009.

- Analytical Methods Committee "Experimental design and optimisation (4): Plackett–Burman designs," AMCTB 55, 2013.
- Bayne, C. K.; Rubin, I. B. *Practical Experimental Designs and Optimization Methods for Chemists*, VCH Publishers: Deerfield Beach, FL; 1986.
- Bezerra, M. A.; Santelli, R. E.; Oliveira, E. P.; Villar, L. S.; Escaleira, L. A. "Response surface methodology (RSM) as a tool for optimization in analytical chemistry," *Talanta* **2008**, *76*, 965–977.
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- Leardi, R. "Experimental Design: A Tutorial," *Anal. Chim. Acta* **2009**, *652*, 161–172.
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- Morgan, E. *Chemometrics: Experimental Design*, John Wiley and Sons: Chichester, 1991.
- Walters, F. H.; Morgan, S. L.; Parker, L. P., Jr.; Deming, S. N. *Sequential Simplex Optimization*, CRC Press: Boca Raton, FL, 1991.

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

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

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- Gonzalez, A. G.; Herrador, M. A. "A Practical Guide to Analytical Method Validation, Including Measurement Uncertainty and Accuracy Profiles," *Trends Anal. Chem.* **2007**, *26*, 227–238.
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Chapter 15

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

- Bell, S. C.; Moore, J. "Integration of Quality Assurance/Quality Control into Quantitative Analysis," *J. Chem. Educ.* **1998**, *75*, 874–877.
- Cancilla, D. A. "Integration of Environmental Analytical Chemistry with Environmental Law: The Development of a Problem-Based Laboratory," *J. Chem. Educ.* **2001**, *78*, 1652–1660.
- Claycomb, G. D.; Venable, F. A. "Selection, Evaluation, and Modification of a Standard Operating Procedure as a Mechanism for Introducing an Undergraduate Student to Chemical Research: A Case Study," *J. Chem. Educ.* **2015**, *92*, 256–262.
- Laquer, F. C. "Quality Control Charts in the Quantitative Analysis Laboratory Using Conductance Measurement," *J. Chem. Educ.* **1990**, *67*, 900–902.
- Marcos, J.; Ríos, A.; Valcárcel, M. "Practicing Quality Control in a Bioanalytical Experiment," *J. Chem. Educ.* **1995**, *72*, 947–949.

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

- Amore, F. "Good Analytical Practices," *Anal. Chem.* **1979**, *51*, 1105A–1110A.
- Anderson, J. E. T. "On the development of quality assurance," *TRAC-Trend. Anal. Chem.* **2014**, *60*, 16–24.
- Barnard, Jr. A. J.; Mitchell, R. M.; Wolf, G. E. "Good Analytical Practices in Quality Control," *Anal. Chem.* **1978**, *50*, 1079A–1086A.
- Cairns, T.; Rogers, W. M. "Acceptable Analytical Data for Trace Analysis," *Anal. Chem.* **1993**, *55*, 54A–57A.
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Additional information about the construction and use of control charts may be found in the following sources.

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

Active Learning Curricular Materials

The Analytical Sciences Digital Library maintains a suite of curricular materials that are the products of a collaborative NSF Phase I CCLI award to Thomas Wenzel, Bates College (DUE 0816649), and Cynthia Larive, University of California Riverside (DUE-0817595) and an NSF TUES Type 2 award to Tom Wenzel, Bates College, (DUE 1118600). The goal of this project is to develop active learning resources to support instruction in analytical chemistry courses. Gathered here are annotated links to these materials.

Materials for Use in Class

Separation Science: A series of collaborative learning activities and accompanying text that develop the field of separation science, with a particular emphasis on chromatographic separations. These activities are intended to be done in class by students working in groups, but can be modified for use as out-of-class exercises. Learning objectives, an instructor's manual, and out-of-class problems are provided. The instructor's manual provides tips for how to use the in-class exercises, the types of responses that students often provide, and how the instructor can build from these responses to develop the concepts. Ancillary modules that are shorter but address specific topics within the area of separation science (steric exclusion chromatography, affinity chromatography, ion exchange chromatography, ultracentrifugation) are provided as well. (Author: Tom Wenzel)

Molecular and Atomic Spectroscopy: A series of collaborative learning activities and accompanying text that develop the areas of molecular and atomic spectroscopy. Chapters on basics of spectrophotometry, ultraviolet/visible absorption, molecular fluorescence, infrared, Raman and atomic spectroscopy are included. These activities are intended to be done in class by students working in groups, but can be modified for use as out-of-class exercises. Learning objectives and an instructor's manual are provided. The instructor's manual provides tips for how to use the in-class exercises, the types of responses that students often provide, and how the instructor can build from these responses to develop the concepts. (Author: Tom Wenzel)

Chemical Equilibrium: A series of collaborative learning activities and accompanying text that develop chemical equilibrium, including acid-base chemistry, formation of water-soluble complexes, and solubility. These activities are intended to be done in class by students working in groups, but can be modified for use as out-of-class exercises. Learning objectives, an instructor's manual, and out-of-class problems are provided. The instructor's manual provides tips for how to use the in-class exercises, the types of responses that students often provide, and how the instructor can build from these responses to develop the concepts. (Author: Tom Wenzel)

Concentration Calibration: A series of collaborative learning activities and accompanying text that develop the concept of concentration calibration, utilizing external standards, internal standards, and standard additions. The module is based primarily on flavonoids, particularly quercetin, as an example analyte. The activities are designed as in-class, small group exercises. An additional out-of-class activity is also available. (Authors: Sandra L. Barnes and David Thompson)

Interpreting the Primary Literature: These assignments are designed to be capstone activities at the end of units on figures of merit (such as sensitivity and LOD), acid-base equilibria, separations, spectroscopy, mass spectrometry, and electrochemistry. Each assignment consists of an out-of-class reading assignment from the primary literature accompanied by objective questions and a set of open-ended, in-class discussion questions. The assignments are designed to require just one class period and can be used before an exam to review important concepts, examine them from new angles, and apply them to new situations. (Author: Michelle L. Kovarik)

Electrochemical Methods of Analysis: A series of collaborative learning activities and accompanying text that develop fundamental aspects of electrochemistry and electrochemical methods of analysis. These activities are intended to be done in class by students working in groups, but can be modified for use as out-of-class exercises. Learning objectives and an instructor's manual are provided. The instructor's manual provides tips for how to use the in-class exercises, the types of responses that students often provide, and how the instructor can build from these responses to develop the concepts. Analytical methods developed in this unit include ion-selective electrodes, electrodeposition, coulometry, electrochemical titrations, and voltammetric methods including anodic stripping voltammetry, linear sweep voltammetry, differential pulse linear sweep voltammetry, and cyclic voltammetry. (Author: Tom Wenzel)

Introduction to Data Analysis: This module introduces students to ways of thinking about and working with data using, as a case study, the analysis of 1.69-oz packages of plain M&Ms. The module is divided into six parts: Ways to Describe Data; Ways to Visualize Data; Ways to Summarize Data; Ways to Model Data; Ways to Draw Conclusions From Data; and Now It's Your Turn! Interspersed within the module'are a series of investigations, each of which asks students to stop and consider one or more important issues. Many of these investigations draw upon a data set that consists of 30 samples of 1.69-oz packages of plain M&Ms. This case study is meant to serve as an introduction to data and to data analysis and, as with any introduction, it considers a small number of topics; additional resources that provide a deeper introduction to data and to data analysis are listed in Appendix 1 of the case study. (Author: David Harvey)

Materials for Use in Lab

Separation Science–Chromatography Projects: An instructor's manual, including learning objectives, for a set of semester-long chromatography projects that are undertaken by students working in small groups. Information about the proposal that students complete before undertaking the experimental part of the project as well as the final written report is provided. Peer- and self-evaluation forms for students are provided as well. Finally, tips for each of the projects that have been done in the past are included. (Author: Tom Wenzel)

Theme-Based Lab Experience: An instructor's guide to implementing a modular theme-based approach to the advanced analytical chemistry laboratory is provided. The guide provides information on student learning objectives, group dynamics, and grading, and provides examples of themes implemented at Butler University. Sample scenarios, student handouts, student reports, and grading rubrics are included in the online appendices. (Author: Michael Samide)

Quality Control Analysis for a Local Brewery: A laboratory project for instrumental analysis with the theme of quality control for a local microbrewery is described. The analyses of important flavor and aroma compounds can be easily modified to work with a variety of instruments and wet chemical techniques. Learning objectives, an instructor's manual, project calendar, assignments, and grading rubrics are provided. The instructor's manual provides a framework for creating a student-centered learning experience, strategies for implementation, TA guidance, and cost estimates. (Author: Jill Robinson)

Analysis of Phosphorous Concentrations in a Natural Water System: This guided research project explores the chemistry and impact of phosphorous on a fresh water system of lakes connected by a river in south-central Wisconsin. Students begin this project by comparing the detection limits, matrix effects, and linearity of standard curves of two different spectrophotometric methods for measuring phosphorus. Once the method of choice is validated, students work in groups to design and carry out experiments to explore the chemistry of phosphorus and its impact on the environment. They learn to use some of the tools necessary for water quality analysis including a Secchi disk and an Ekman dredge. Visible spectroscopy serves as the primary vehicle for learning, although the research projects sometimes incorporate ICP-AES, HPLC, and ISE measurements if students take their research in that direction. (Author: Pamela Doolittle)

Acid Mine Drainage Project Lab: This laboratory project uses the context of Acid Mine Drainage to teach concepts important to analytical chemistry and quantitative analysis. Students set up experiments that mimic the process of metal sulfide mineral oxidative dissolution. The experiments explore how the rate of dissolution changes with respect to changes in pH, added oxidizing agents, and oxygen rich or oxygen poor environments. Visible spectroscopy is used to initially measure the concentration of complexed iron in solution. ICP-AES is used to verify the stoichiometry of the arsenopyrite sample. Elemental sulfur determination and the speciation of the aqueous sulfur in the solution can be determined using reverse phase and ion pair high performance liquid chromatography. (Authors: Pamela Doolittle and Robert J. Hamers)

Contextual Modules (Case Studies)

Environmetal Analysis–Lake Nakuru Flamingos (Pesticides): Could toxic pesticides like DDT be responsible for the deaths of large numbers of lesser flamingos at Lake Nakuru, Kenya? While many organochlorine pesticides including DDT have been banned for decades in the US due to their adverse effects on bird populations, especially bald eagles, they are still used for mosquito control in tropical regions of Africa where malaria is epidemic. In addition, East Africa has become an international dumping ground for stockpiles of obsolete pesticides. In this section we explore the possible role of organochlorine pesticides in the flamingo deaths and examine the use of gas chromatography – mass spectrometry (GC-MS) to separate, detect and quantify pesticides in Lake Nakuru water samples. (Authors: Heather A. Bullen, Alanah Fitch, Richard S. Kelly, and Cynthia K. Larive)

Environmental Analysis–Lake Nakuru Flamingos (Heavy Metals): Toxic trace metals are possible culprits for the ongoing deaths of large numbers of lesser flamingos at Lake Nakuru, Kenya. In this section we explore the possible role of heavy metals in these deaths and examine instrumental methods utilized to evaluate levels of copper, zinc, lead and chromium present in Lake Nakuru sediment and suspended solid samples. These methods include anodic stripping voltammetry (ASV), atomic spectroscopy, and x-ray fluorescence spectroscopy (XRF). Data sets are provided for each technique so that current levels can be calculated and compared to those contained in a report published in 1998. (Authors: Erin Gross, Richard S. Kelly, and Cynthia Larive)

Lithia Water Springs Project: Can the most prevalent inorganic ions be determined in Lithia water using a representative cross-section of the analytical techniques (e.g. titrimetry, potentiometry, spectroscopy) covered in a typical quantitative analysis course? In this module, we will examine the role of chemical equilibria, stoichiometry, and univariate statistics in the sample preparation and characterization of Lithia water. The discovery of mineral springs in the vicinity of Ashland, Oregon sparked the pursuit of a "spa economy" during the 1910s and 1920s. The lithium concentration in this spring, which is the second highest in the U.S., was a marketing point for town leaders in the early 20th century. Even today, Lithia water plays a visible role in the culture and history of Ashland. These materials may be used as a term-long quantitative analysis laboratory project or as a dry lab using the questions and the data supplied in this module. (Author: Steven Petrovic)

End Creek Spotted Frogs & Aquatic Snails in Wetlands: This module provides a context for introducing fundamental techniques used in chemical analysis (spectrophotometry, atomic absorbance spectroscopy and ion selective electrodes) along with considerations about sampling and sample preservation. Using an active learning approach, the module explores some fundamental water quality parameters such as the concentration of inorganic cations and anions that may aid in understanding why certain ponds provide a more suitable habitat for the Columbia Spotted frogs and aquatic snails. (Authors: Anna Cavinato and Karen Antell)

Developing an Analytical Method for the Analysis of a Medicinal Plant: This module introduces students to the process of developing an analytical method using, as a case study, the quantitative analysis of eight analytes in the medicinal plant Danshen using a combination of a microwave extraction to isolate the analytes and

HPLC with UV detection to determine their concentrations. Interspersed within the module's narrative are a series of investigations, each of which asks students to stop and consider one or more important issues. As students progress through the module they are introduced to chromatographic separations, solvent extractions, response surfaces, one-factor-at-a-time optimizations, central-composite designs, desirability functions, and spike recoveries. (Author: David Harvey)

Effect of Acid Rain on Atlantic Salmon Populations: This module provides a real world context for introducing fundamental quantitative techniques (pH, Ion Selective Electrodes, Ion chromatography, and Titrimery) used in chemical analysis of water samples. Using active learning pedagogy, students explore sampling and analyzing the base inorganic ions and acidity parameters in freshwater samples. The module frames the study using the environmental impacts of acid rain on the habitat of the endangered Atlantic Salmon as a case study. (Author: William Otto)