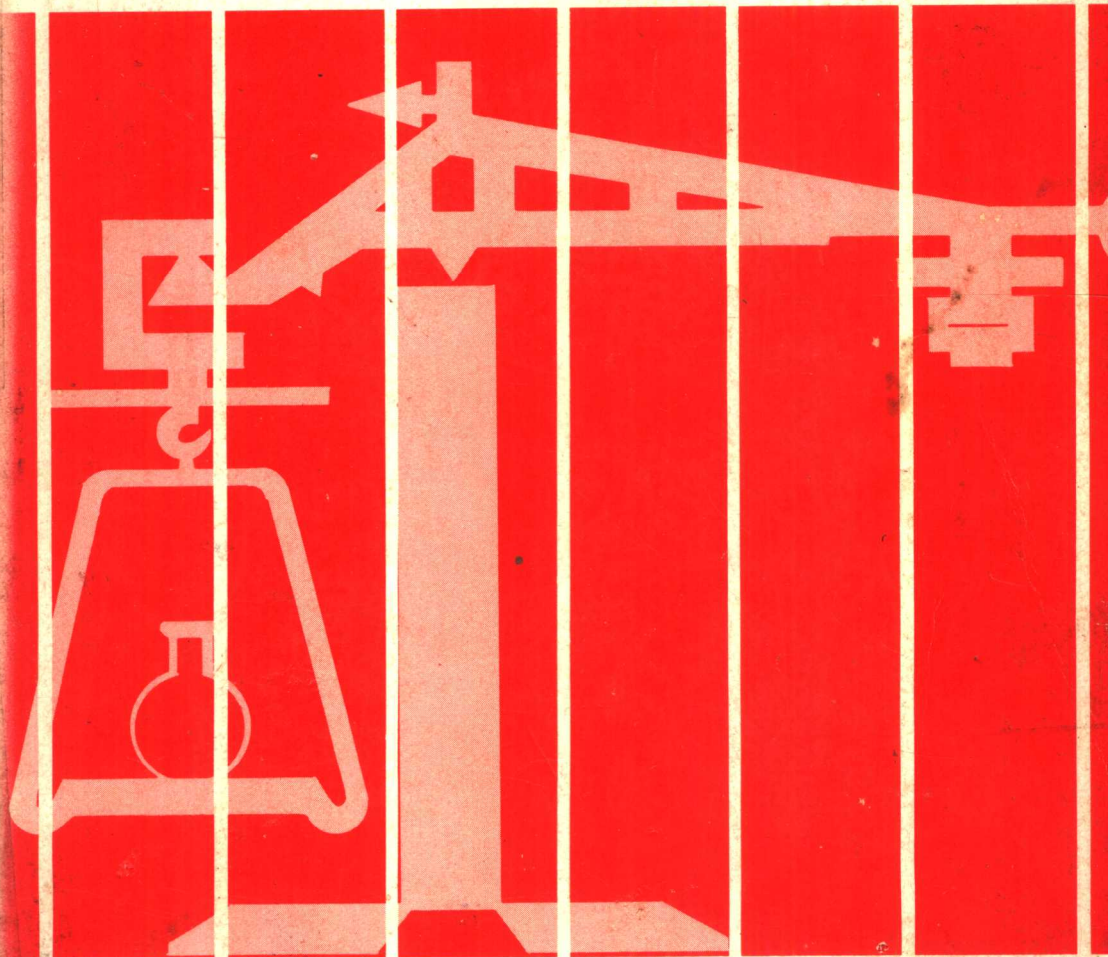


**Douglas A. Skoog  
Donald M. West**

# **Fundamentals of Analytical Chemistry**

**Third Edition**



**Holt-Saunders International Editions**

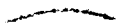


# FUNDAMENTALS OF ANALYTICAL CHEMISTRY

THIRD EDITION

DOUGLAS A. SKOOG  
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# PREFACE

This edition of *Fundamentals of Analytical Chemistry* contains several innovations. Chapter 3 is a new chapter which has been added to review equilibrium concepts in the same way that Chapter 2 reviews stoichiometric relationships. All specific laboratory directions are collected in a single chapter (31). Chapters concerned with the evaluation of data, potentiometry, spectroscopy, and separations have been revised and expanded. New problems have been prepared; the answers to nearly half the problems in the text are given at the back of the book. A solutions manual is also available. To make room for new material, discussions concerned with gravimetric analysis have been substantially condensed and the chapter on conductometric titration deleted.

Although it is tempting to emphasize changes, it is also important to note that the principal aims of this edition are the same as those of earlier editions. We remain convinced that a thorough grounding in theory and—insofar as it can be considered in an elementary text—an appreciation for the entire analytical process are of prime importance. The student with a clear understanding of the chemical background for an analysis is most likely to identify the experimental details that are crucial to success and to distinguish them from those which require little or no attention.

With the exception of the changes noted at the outset, we have retained the format of the earlier editions. Although gravimetric analysis is covered before volumetric methods, the order of presentation can be reversed if the instructor so chooses. Sufficient material concerned with instrumental methods for completion of an analysis is presented to allow coverage of those topics that are deemed to be most important; of particular importance for a one-semester course are Chapter 17, in which ion-selective membrane electrodes are discussed, and Chapters 23 and 24, which are concerned with methods based on the absorption of electromagnetic radiation.

Many colleagues have generously shared suggestions for improvement of the presentation. As before, we are particularly grateful to Professor Alfred Armstrong of The College of William and Mary who read the entire manuscript and contributed many incisive criticisms. We also acknowledge with gratitude the comments of M. R. Bacon, University of Nevada; R. G. Bates, University of Florida; D. G. Berge, University of Wisconsin; E. J. Billo, Boston College; R. L. Birke, University of South Florida; G. L. Blackmer, Texas Technological University; J. Q. Chambers, University of Tennessee; J. A. Cox, Southern Illinois University; V. P. Guinn, University of California/Irvine; T. P. Hadjiioannou, University of Illinois/Urbana-Champaign; T. J. Hauptert, California State University/Sacramento; J. W. Knoeck, North Dakota State University; R. C. Legendre, University of Southern Alabama; D. E. Leyden, University of Georgia; C. K. Mann, Florida State University; H. B. Mark, Jr., University of Cincinnati; R. S. Mitchell, Arkansas State University; J. H. Nelson, University of Nevada; R. T. O'Donnell, State University of New York/Oswego; G. K. Pagenkopf, Montana State University; F. W. Plankey, University of Pittsburgh; D. S. Polcyn, University of Wisconsin; M. W. Rowe, Texas A&M University; J. T. Stock, University of Connecticut/Storrs; W. E. Swartz, Jr., University of South Florida; P. J. Taylor, Wright State University; C. E. Wilson, Indiana/Purdue University; D. C. Young, Oakland University; S. T. Zenchelsky, Rutgers University.

*December, 1975  
Stanford, California  
San Jose, California*

*D.A.S.  
D.M.W.*

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# INTRODUCTION

Analytical chemistry deals with methods for the identification of one or more of the components in a sample of matter and the determination of the relative amounts of each. The identification process is called a *qualitative analysis* while the determination of amount is termed a *quantitative analysis*. In this text, we will deal largely with the latter.

The results of a quantitative analysis are expressed in such relative terms as the percent of the *analyte* (the substance being determined) in the sample, the parts of analyte per thousand, per million, or even per billion parts of sample, the grams or milliliters of analyte per liter of sample, the pounds of analyte per ton of sample, or the mole fraction of the analyte in the sample.

## Applications of Quantitative Analysis

The results of chemical analyses have widespread practical applications. To cite just a few examples of the way in which quantitative data influence the life of modern man, consider the following. Information as to the parts per million of hydrocarbons, nitrogen oxides, and carbon monoxide in exhaust gases serves as a means of defining the quality of smog-control devices for the automobile.

Determination of the concentration of ionized calcium in blood serum is an important method for the diagnosis of hyperparathyroidism in human patients. Quantitative data on nitrogen in breakfast cereals and other foods are directly related to their nutritional qualities. Periodic quantitative analyses during the production of a steel permit the manufacture of a product having a desired strength, hardness, ductility, or corrosion resistance. The continuous analysis for mercaptans in the household gas supply assures the presence of an odorant which warns of dangerous leaks in the gas-distribution system. The analysis of soils for phosphorus, nitrogen, sulfur, and moisture throughout the growing season makes it possible for the farmer to tailor fertilization and irrigation schedules to meet plant needs most efficiently; significant reductions in costs for fertilizer and water as well as increases in yield result.

In addition to practical applications of the types just considered, quantitative analytical data are at the heart of research activity in chemistry, biochemistry, biology, geology, and the other sciences. In support of this assertion, consider the following few examples. Much of what the chemist knows about the mechanisms by which chemical reactions occur has been learned from kinetic studies in which the rate of disappearance of reactants or appearance of products is followed by quantitative analyses for these reactants or products. Recognition that the conduction of nerve signals in animals and the contraction or relaxation of muscles involves the transport of sodium and potassium ions across membranes was made possible by quantitative measurements of these species on the two sides of such membranes. Studies concerned with the mechanism by which oxygen and carbon dioxide are transported in blood have required methods for continually monitoring the concentration of these and other compounds within a living organism. The understanding of the behavior of semiconductor devices has required development of methods for the quantitative determination of impurities in pure silicon and germanium in the range of  $1 \times 10^{-6}$  to  $1 \times 10^{-10}$  %. Recognition that the amounts of various minor elemental constituents in obsidian samples permit identification and location of their sources has enabled archeologists to trace prehistoric trade routes for tools and weapons fashioned from these materials. In some instances, analytical data on the composition of surface soils have permitted geologists to detect the presence of major ore bodies at considerable depths. Quantitative analyses of minute samples from works of art have provided historians with important clues as to the materials and technologies employed by artists of the past as well as an important tool for the detection of art forgeries.

For the typical research worker in chemistry, biochemistry, and some of the biological sciences, the acquisition of quantitative information represents a significant fraction of his laboratory efforts. Analytical procedures, then, are among the important tools employed by such a scientist in pursuit of his research goals. The development of an understanding of the basis of the quantitative analytical process and the competence and confidence to perform analyses is therefore a prerequisite for research in many of these fields. The role of analytical chemistry in the education of chemists and biochemists can be viewed as being analogous to that of calculus and matrix algebra for those aspiring toward a

career in theoretical physics or to the role of Greek or other ancient languages in the education of the scholar of classics.

## Performance of Quantitative Analysis

The results of a typical quantitative analysis are based upon two measurements (or sometimes two series of measurements), one of which is related to the quantity of sample taken and the second to the quantity of analyte in that sample. Examples of the quantities measured include weight, volume, light intensity, absorption of radiation, fluorescent intensity, and quantity of electricity. It is important to recognize, however, that these measurements are but a part of the typical quantitative analysis. Indeed, some of the preliminary steps are as important and often more difficult and time-consuming as the measurements themselves.

For the most part, the early chapters of this text are devoted to the final measurement steps, and the other aspects of an analysis are not treated in detail until near the end of the book. Thus, to lend perspective, it is useful at the outset to identify the several steps that make up the analytical process and to indicate their importance.

### SAMPLING

To produce meaningful results, an analysis must be performed on a sample whose composition faithfully reflects that of the bulk of material from which it was taken. Where the bulk is large and inhomogeneous, great effort is required to procure a representative sample. Consider, for example, a railroad car containing 25 tons of silver ore. Buyer and seller must come to agreement regarding the value of the shipment, based primarily upon its silver content. The ore itself is inherently *heterogeneous*, consisting of lumps of varying size and of varying silver content. The actual assay of this shipment will be performed upon a sample that weighs perhaps 1 g; its composition must be representative of the 25 tons (or approximately 22,700,000 g) of ore in the shipment. It is clear that the selection of a small sample for this analysis cannot be a simple one-step operation; in short, a systematic preliminary manipulation of the bulk of material will be required before it becomes possible to select 1 g and have any confidence that its composition is typical of the nearly 23,000,000 g from which it was taken.

The sampling problem often is not so formidable as that outlined in the preceding paragraph. Still, the chemist cannot afford to proceed with an analysis until he has convinced himself that the fraction of the material with which he plans to work is truly representative of the whole.

### PREPARATION OF THE LABORATORY SAMPLE FOR ANALYSIS

Often, solid materials must be ground to reduce particle size and then thoroughly mixed to ensure homogeneity. In addition, removal of adsorbed moisture is often

required for solid samples. Adsorption or desorption of water causes the percentage composition of a substance to depend upon the humidity of its surroundings at the time of the analysis. To avoid the problems arising from such variations, it is common practice to base the analysis on a dry sample.

### MEASUREMENT OF THE SAMPLE

Quantitative analytical results are usually reported in relative terms; that is, in some way that expresses the quantity of the desired component present per unit weight or volume of sample. It is therefore necessary to know the weight or volume of the sample upon which the analysis is performed.

### SOLUTION OF THE SAMPLE

Most, but certainly not all, analyses are performed on solutions of the sample. Ideally the solvent should dissolve the entire sample (not just the analyte) rapidly and under sufficiently mild conditions that loss of the analyte cannot occur. Unfortunately, such solvents do not exist for many or perhaps most materials that are of interest to the scientist. Commonly he deals with chemically intractable substances such as an ore sample, a high-molecular-weight polymer, or a piece of animal tissue. Converting the analyte in such material into a soluble form is often a formidable and time-consuming task.

### SEPARATION OF INTERFERING SUBSTANCES

Few, if any, chemical or physical properties of importance in analysis are unique to a single chemical species; instead, the reactions used and the properties measured are characteristic of a number of elements or compounds. This lack of truly specific reactions and properties adds greatly to the difficulties faced by the chemist when undertaking an analysis; it means that a scheme must be devised for isolating the species of interest from all others present in the original material that produce an effect upon the final measurement. Compounds or elements that prevent the direct measurement of the species being determined are called *interferences*; their separation prior to the final measurement constitutes an important step in most analyses. No hard and fast rules can be given for the elimination of interferences; this problem is often the most demanding aspect of the analysis.

### THE COMPLETION OF THE ANALYSIS

All preliminary steps in an analysis are undertaken to make the final measurement a true gauge of the quantity of the species being determined.

The chapters that follow contain descriptions of many types of final measurement, along with discussions of the chemical principles upon which such measurements are based.

## Choice of Methods for an Analysis

The chemist or scientist who has need for analytical data usually finds himself faced with an array of methods which could be used to provide the desired information. His choice among these will be based on such considerations as speed, convenience, accuracy, availability of equipment, number of analyses, amount of sample that can be sacrificed, and concentration range of the analyte. The success or failure of an analysis is often critically dependent upon the proper selection of method. Unfortunately, there are no generally applicable rules that can be applied; the choice of method is thus a matter of judgment. Such judgments are difficult, and the ability to make them well comes only with experience.

This text presents many of the most common unit operations associated with chemical analyses and includes a variety of methods for the final measurement of analytes. Both theory and practical detail are treated. Mastery of this material will permit the student to perform many useful analyses and will provide him with background from which he can develop the judgment needed for the prudent choice of an analytical method.



# A REVIEW OF SOME ELEMENTARY CONCEPTS

Most quantitative analytical measurements are performed on solutions of the sample. The study of analytical chemistry, therefore, makes use of solution concepts with which the student should have considerable familiarity. It is the purpose of this chapter, and the one that follows, to review the most important of these concepts.

## The Chemical Composition of Solutions

Both aqueous and organic solvents find widespread use in chemical analysis. Nonpolar solvents, such as hydrocarbons and halogenated hydrocarbons, are employed when the analyte itself is nonpolar. Organic solvents, such as alcohols, ketones, and ethers, which are intermediate in polarity and which form hydrogen bonds with solutes, are considerably more useful than their less polar counterparts because they dissolve a larger variety of both organic and inorganic species. Aqueous solvents, including solutions of the common inorganic acids and bases, are perhaps the most widely used of all for analytical purposes. Our discussion will therefore focus on the behavior of solutes in water; reactions in nonaqueous polar media will be considered in less detail.