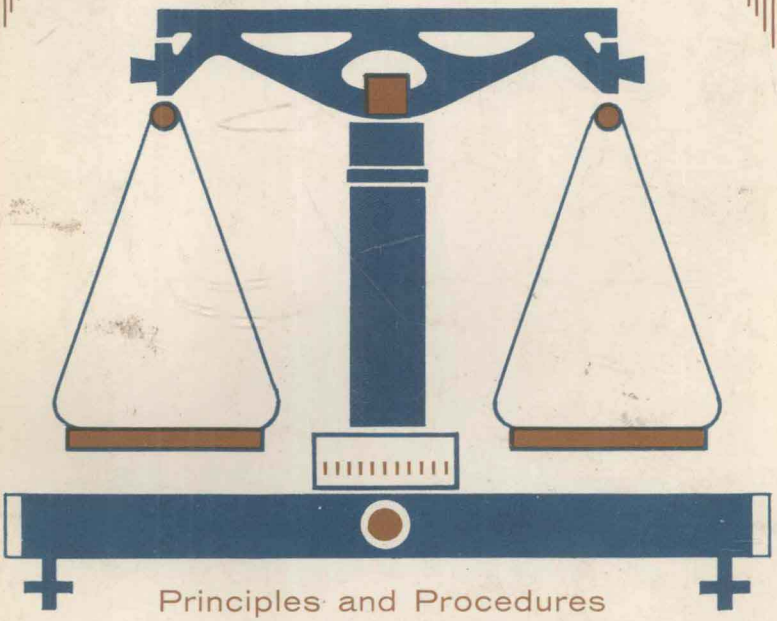
Quantitative Analysis

by

RAY U. BRUMBLAY



Principles and Procedures
Examples, Problems with Answers

QUANTITATIVE ANALYSIS

Ray U. Brumblay
Professor of Chemistry
University of Wisconsin

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Preface

This book is intended to provide in brief form the information usually contained in a one-semester course in quantitative analysis. In addition, a few well-established ideas and methods are included. Activities are given relatively more space than is usual in most textbooks because they are fundamental and should be applied to problems in analytical chemistry more frequently than they are. Thermogravimetric analysis is a basic method of evaluating a standard procedure, the heating of a substance to constant weight. It deserves some mention. The over-all intention is to state the fundamentals of analytical chemistry, and whenever possible, illustrate them with problems. The solutions to problems are broken down into simple steps to aid the student in following the reasoning involved.

In order for the student to study the subject matter in this book profitably an acquaintance with simple algebra and elementary chemistry is essential. Other books in the "College Outline Series" that can be of aid in understanding quantitative analysis principles are:

College Algebra
First Year College Chemistry
Chemistry Problems and How to Solve Them
Physical Chemistry

Physical Chemistry treats much of the theoretical material applied in analytical chemistry from a different angle and usually with a more rigorous mathematical approach. The different viewpoint it provides can be quite helpful in giving the student a better insight into ideas encountered in analytical chemistry.

Tabulated Bibliography of Standard Textbooks

This *College Outline* is keyed to standard textbooks in two ways.

1. If you are studying one of the following textbooks, consult the cross references here listed to find which pages of this *Outline* summarize the appropriate chapter of your text. (Roman numerals refer to the textbook chapters; Arabic figures refer to the corresponding pages of this *Outline*.)

2. If you are using this *Outline* as your basis for study and need a fuller treatment of a topic, consult the pages of any of the standard textbooks as indicated in the Quick Reference Table on pp. xiv–xvii.

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Introduction

Analytical chemistry is concerned with identifying the constituents of a substance and finding how much of each constituent is in the substance. Identifying the constituents is called qualitative analysis. Finding how much of one or more constituents is present in a given amount of the substance is called quantitative analysis. Quantities are usually measured in weight and are calculated as per cent by weight of the original material, although, in dealing with liquids, volumes are frequently employed.

Progress in scientific research depends almost entirely on quantitative measurements. This is indicated by the fact that chemistry made little advancement until quantitative weight experiments produced knowledge of the nature of combustion and the laws of chemical action. Then chemistry as a science advanced rapidly, and it continues to advance only as rapidly as quantitative measurements can furnish answers to research problems. Advances in instrumental methods have accelerated analytical work greatly but have not replaced strictly chemical methods. This is true because each instrument requires calibration and checking, made possible only by using established analytical methods.

The analytical chemist reaches out into all fields of knowledge for principles which he can apply to improve his work or accomplish his objective. One of his most useful tools is mathematics. Although higher mathematics is often required to establish the validity of an equation, or to derive it, the calculations made by the student of analytical chemistry seldom require more than a knowledge of simple algebra. It is largely through calculations, the working of problems, that one becomes familiar with most of the basic principles of analytical chemistry.

Not everyone who studies analytical chemistry becomes an analytical chemist. For most people, study of the subject is for the

purpose of becoming familiar with the possibilities the subject offers in helping to solve problems that one may face in related fields.

Types of Chemical Analysis

There are several ways of classifying the methods applied in analytical chemistry. The two largest groups are organic analysis and inorganic analysis. The former is quite specialized; the latter is applicable to a wide variety of elements and their compounds. It is inorganic analysis with which this book is generally concerned.

Complete Analysis. If all elements in a material, even those in trace quantities, are determined quantitatively, the analysis is said to be complete.

Partial Analysis. A partial analysis is the quantitative determination of one or more constituents in a sample, such as the determination of the per cent of phosphorus in phosphate rock or the per cent of iron in iron ore.

Proximate Analysis. The determination of the per cent of one or more constituents in a sample all of which react alike with a certain treatment is called a proximate analysis. The separation and determination of the per cent of R_2O_3 in a limestone (p. 215) is a common example of a proximate analysis.

Ultimate Analysis. If the per cent of each element in a substance is determined, the process is called an ultimate or elemental analysis. This type is usually applied to the analysis of organic compounds in order to aid in establishing the formula of the substance. It seldom includes determination of the per cent of trace elements.

Types by Sample Size. Analytical methods are sometimes labelled according to the size of the sample taken for analysis.

Macro methods apply to samples 0.1 g or larger.

Semimicro methods apply to samples approximately 0.01 g to 0.1 g.

Micro methods apply to samples approximately 0.001 g to 0.01 g (1–10 mg).

Ultramicro methods apply to samples approximately 1 to 10 micrograms, designated by the symbols μg or γ (gamma). A microgram is 0.001 mg.

Types of Methods of Determination

There are almost an unlimited number of types of methods used in making determinations, but only a few are general enough to deserve mention here.

Gravimetric (Weight-Measuring) Methods. A constituent or some compound of it is weighed to find the per cent of the constituent. Several general procedures are applied in gravimetric methods.

Precipitation of a solid, filtering it off, drying it, and weighing it can in many instances give a value which is related to the weight of the constituent sought.

Volatilization of a material which has a relatively high vapor pressure or low decomposition temperature may be useful in measuring the portion of a sample that can be driven off by heat.

Electrolysis is often applied to separating an element or a compound in a weighable form.

Volumetric (Volume-Measuring) Methods. The measurement of a volume of a substance, or of a reagent, is often made to determine the per cent of a constituent. The volume of reagent as a solution added is usually measured in an instrument called a buret (p. 12). The process of adding the reagent from a buret is called titration. A method involving a titration is therefore called a titrimetric method. The analysis of gases is also generally a volumetric method.

Instrumental Methods. Methods of determining a constituent by measuring some physical property with an instrument are called instrumental methods. Some are based on light measurements and are called optical methods, and some are based on measurement of electrical phenomena and are called by various names depending on what electrical property is measured. Some of the better known electrical methods are conductometric (measurement of conductance), polarography (measurement of polarization due to concentration), and potentiometric (measurement of voltage or potential at an electrode, as in a pH meter).

It would be ideal for the student to become familiar with all the types of methods in common use. However, this becomes increasingly difficult with the rapid introduction of new methods that has taken place in recent years. In addition, some of the newer instrumental methods are so technical and specialized as to be beyond the scope of an elementary course in analytical chemistry. It is necessary therefore to limit this book to the most widely used methods and to those which seem to be based on general rather than on specific principles.