Preface to Second Edition

Since its introduction in the early part of this century, microanalysis has become a much used tool, particularly as an aid to research where the amounts of material to be analyzed are both scarce and costly. Many articles on the subject have been published with the purpose of making it possible for others than those trained by the "old masters" to satisfactorily perform the work. These articles have been reviewed both for individual determinations over long periods of time and for all phases over short periods. For the benefit of the experienced analyst who wishes to use variations of old procedures or to put into use newly published methods, a large number of articles, representing a high percentage of all of those published through 1959, and some in 1960, are referred to in tabular form at the end of each chapter. The above-mentioned reviews have been most helpful in collecting this data.

To be successful the beginner, as well as the experienced microanalyst, must have correctly proportioned apparatus and, in addition, the former must have specific directions for performing the work—methods known to be successful in the hands of those with comparatively little experience. Several groups in the United States have worked toward this goal, namely, the following:

(a) Committee on Microchemical Apparatus of the Division of the Analytical Chemistry of the American Chemical Society.

(b) Subcommittee No. 29 on Microchemical Apparatus under Committee E-1 on Methods of Testing of the American Society for Testing Materials.

(c) The Association of Official Agricultural Chemists.

(d) Commission on Microchemical Techniques of the Section of Analytical Chemistry of the International Union of Pure and Applied Chemistry.

Extensive work on the standardization of apparatus has also been in progress for a number of years in Great Britain.

The first edition of this book* included the various pieces for which the American Chemical Society group had recommended specifications and methods adopted by the Association of Official Agricultural Chemists up to that time. Since the writing of the first edition, additional material has been published by the above two organizations and the American Society for Testing Materials has come into the picture reviewing and changing, if necessary, the recommendations of the American Chemical Society group. The International Union of Pure and Applied Chemistry group also has been active since the days of the first edition. There is close contact between these groups—in fact certain individuals are associated with three of the groups and the author with all of them. The second edition is up to date on all of the latest recommendations

* Published in 1951 by The Blakiston Company.

of the American (or American participating) groups, since the author firmly believes that, through the use of these, best results can be obtained by both the beginner and the experienced analyst.

In the first edition, *only* those procedures were described in detail which had been used extensively by the author and his assistants in the microchemical laboratories of their company during the preceding twelve years. These laboratories act as a service department for the company's large research division. During the past ten years, a number of new procedures have been developed for various determinations; however, in this second edition, the author presents, for the most part, basically the same procedures contained in the first edition since these, after nearly twenty-two years of use by him and his assistants, are still considered to be the best guide for the beginner as well as time-tested reliable methods for the experienced analyst. Where more than one method for a determination has met these qualifications, a choice is included and the beginner will do well to become acquainted with each inasmuch as experience has shown the importance of having available referee methods for proving the reliability of analytical data, particularly in connection with the extensive research programs in progress in many organizations.

Considerable material has been added to that which was presented in the first edition. This includes discussions of the following: test samples, blank tests, description of a second type of efficient vibration-absorbing balance table, enlargement of the section on microchemical balances, new Kjeldahl procedures to determine nitrogen in compounds in which nitrogen is connected to nitrogen or to oxygen, oxygen flask combustions, determination of fluorine, and micro-hydrogenation.

The procedure for the determination of oxygen has been modified to a gravimetric one, which eliminates the errors due to interfering elements. One of the alkoxyl procedures has been changed to coincide with that developed through the joint efforts of the Committee on Microchemical Apparatus of the Division of Analytical Chemistry of the American Chemical Society and the Association of Official Agricultural Chemists.

Long experience has shown that the procedures give best results when the pieces of apparatus are in constant use. In some cases, after a determination has been giving reliable results for months and is then suddenly not used for a period of time, trouble will be experienced on trying to resume the work. The encountered difficulties are not always easily corrected and at times even appear to correct themselves. Naturally, the more experience the operator has had, the fewer will be these instances.

The order in which the various chapters are presented is the same order in which the work has been taught to new members of the author's department.

During the past years, chemists holding bachelor's degrees, but without previous knowledge of microchemistry, have been employed and given a training period, the length of which depends upon the ability of the individual.* On the average, after a few weeks, the newcomer is able to accept research samples for the determination of moisture, metals, nitrogen, and, in some cases, halogens and sulfur. After about a month's experience with the above, the novice is ready to begin work on the carbon-hydrogen determination, which is considered to be the most important of all. After a few more weeks he is able to accept research samples and perform the carbon-hydrogen analyses with the desired accuracy. Within the following months the new analyst is taught all of the other determinations in this book, as the author is of the opinion that each member of the staff should be able to do all of the various analyses. Of course, the above schedule is not a practical one for teaching a college course, but it must be remembered that the author uses it to train microanalysts who must, within as short a time as possible, perform the work in an industrial laboratory with the apparent responsibilities involved. Naturally, this text is equally adaptable to either the one-semester or one-year courses offered in colleges to advanced students.

In closing, the author wishes to emphasize that few microanalysts carry out the determinations in exactly the same manner, each having his own modification. Despite this, the beginner must have reliable methods as a guide, particularly those known to give good results in the hands of many analysts. This book presents such procedures, and its use, as a college text, as well as a guide for industry, is suggested.

In general, the apparatus required is commercially available from the various scientific apparatus dealers. Suggestion by the author of a certain source of supply should *not* be interpreted as an endorsement of the company in question. The same applies to the various instruments used. The author is merely attempting to be helpful by informing the readers of some sources of supply and, in certain cases, the type of instruments used.

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* The late Rev. F. W. Power of Fordham University once stated that "microchemistry is an art as well as a science." This statement is proved by the fact that many well-trained chemists with considerable experience have been failures at this work, while others, seemingly less equipped, have been very successful. The author has found that persons who have developed the use of their hands through piano playing, typing, art, mechanics, etc., are best suited for this work.