# FOOD ANALYSIS: THEORY AND PRACTICE

# **Food Analysis:**

# Theory and Practice

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THE AVI PUBLISHING COMPANY, INC.

1971

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REGISTERED AT STATIONER'S HALL LONDON, ENGLAND 1971

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Library of Congress Catalog Card Number: 73-156799 ISBN-0-87055-100-0

Printed in the United States of America BY MACK PRINTING COMPANY, EASTON, PENNSYLVANIA

# Food Analysis: Theory and Practice

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1.

This book has been designed for use as a text by undergraduate students majoring in food science and technology, and as a survey of modern analytical techniques and instruments for the worker and researcher in the field of food analysis.

In preparation of the manuscript, we followed the recommendations for subject coverage in a textbook of food analysis for students of food science and technology, as contained in the report of the Task Force on Food Analysis of the Institute of Food Technologists' Council Committee on Education.

Basic principles are stressed, rather than details of analytical methods. Although the emphasis has been on modern and sophisticated instruments and methods, we have described also the classical procedures that have been in use for many years. In chapters devoted to instrumentation, we have attempted to provide the background theory that is required for understanding the principles of each instrumental assay. Included are diagrams and descriptions of typical instruments, information on their application and precision, and sample problems with detailed solutions. Whenever applicable, we have compared the instrumental and assay procedures to evaluate their usefulness and limitations. The chapters on instrumentation end with problem questions (and answers) to further develop the subject matter covered in the text.

In the sections devoted to applications, qualitative and quantitative aspects of the basic instruments and procedures are discussed in terms of their numerous analytical and instrumental procedures, and discussions of their use in solving specific problems are followed by comprehensive and up-to-date bibliographic lists.

This book is not meant to replace standard methods of analysis; its main purpose is to explain the background and principles of those methods. In writing this book, we were faced with several major questions. The rapid advances in analytical instrumentation may make some of the sections out of date. We hope by stressing fundamental principles, rather than details of methodology, that the book will be for some time a useful source of information both for the student and for the experienced researcher. For understanding most of the material, the reader would be expected to have studied general, organic, analytical, and food chemistry, and to have an appre-

ciation of biochemistry. To allow for his limited training in physical chemistry, we have included the physicochemical principles, terminology, and detailed computations.

A major problem concerned the selection of methods and techniques that should be included. While we realize that some of the instrumental techniques that require expensive equipment or more sophisticated biological methods may not be within the reach of many food chemists, we included them to stimulate the thinking of the searching student and the experienced researcher. We hope that these new procedures will contribute to an appreciation of the scopes and potentialities of food analysis. It might be worth pointing out that some of these techniques and procedures can yield excellent results with simplified adaptations—provided the principles are understood. We hope most readers will agree with our selection of methods, techniques, and approaches, and would greatly appreciate comments from teachers, researchers, food analysts, and—most important—students in food science and technology.

We wish to thank the authors of articles and books, industrial companies, and publishers for permission to reproduce material. Special appreciation is expressed to Dr. D. K. Tressler and to Mr. J. J. O'Neil for their many helpful suggestions in preparation of the book.

Y. Pomeranz C. E. Meloan

March 1971

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General

CHAPTER 1

# Searching the Literature

### INTRODUCTION

Keeping informed of current developments in his field is important to any professional worker. It is, however, particularly essential to scientists in a rapidly expanding area such as analytical chemistry. While no analyst can be familiar with all of the recent advances, he can learn how and where to look most effectively for the needed information. An attempt to survey the vast quantity of scientific literature may so frustrate the inexperienced worker that he decides to ignore it altogether. Alternatively, he may sacrifice bench work to do practically endless reading and searching the literature (Bottle 1962).

Several books dealing with the use of the chemical literature have been published (Becker and Hayes 1964; Bottle 1962; Burman 1965; Crane *et al.* 1957; Dyson 1951; Gould 1961; Mellon 1965). Those books show how and where to find information; analyze the various sources; and describe the tools, elements, and theories of information storage and retrieval.

It is assumed that the analyst is "literature-conscious" and it is the purpose of this short chapter to outline the main sources of information and general approaches to a literature search.

For over 300 yr periodicals were the principal means for the exchange of scientific information. There are two basic types of scientific journals (1) primary sources which report original work and provide detailed experimental procedures, and data derived from them, and (2) secondary sources which process the original data into abstracts and reviews, and provide a condensed source of information.

Keys to the contents of periodicals are provided by abstracts and indexes. Abstracts which are short summaries of published articles keep the readers informed of current research; indexes, which may list subjects, authors or formulas, assist in the search of published information. Most journals issue an index to each volume, generally annually, and some issue cumulative indexes covering several years. The most important compilations of abstracts and indexes cover a number of periodicals in a specific area, such as *Chemical Abstracts* (10,000 periodicals) in the area of chemistry.

To keep abreast of the current information in a familiar field, one generally reads a number of journals and then scans the abstracts for a wider coverage.

#### SEARCHING A NEW FIELD

## General Background

In a new field, the first source is an advanced textbook. For general background information, several voluminous encyclopedia devoted to chemistry and technology can be consulted. These include the technical and detailed Kirk-Othmer's Encyclopedia of Chemical Technology (Interscience Publishers) and Ullmann's Enzyklopedia der Technischen Chemie (Urban and Schwarzenberg Publishing). The Encyclopedia of Science and Technology (McGraw-Hill Book Co.) is less detailed but covers a wider field.

An excellent source of information is the single-volume *Merck Index of Chemicals and Drugs*. This compilation revised every several years contains formulas, preparations, and properties of over 10,000 chemicals.

In the identification and determination of food composition, the analyst often has to determine the physical properties of the substance. The reference data can be obtained from various standard tables, handbooks, and some of the newer encyclopedias and dictionaries. Information on physical properties is often published by manufacturers of chemicals. In addition to industrial laboratories, government agencies (i.e. the U.S. National Bureau of Standards) publish authoritative compilations of physicochemical data.

The Handbuch der Organischen Chemie, initiated by F. K. Beilstein, is the largest and most comprehensive source of information in organic chemistry. The twenty-seven volumes of the main work published by the German Chemical Society included 200,000 entries covering the literature prior to 1910. Supplements are published periodically. The "Handbuch" deals with well-defined organic compounds and with natural materials of unknown structure. The coverage varies with the significance of the compound and the available information. Included are (whenever available) names, formu-

las, structures, history, occurrence, preparation, properties, technology, analysis, and reactions. In recent years, several new references on organic chemistry have been published but the "Handbuch" continues to maintain its prominent position.

#### Reviews

The next stage is a survey or a critical review of the current knowledge in the particular field. Monographs are basically comprehensive surveys of current knowledge on a specific subject. One of the best known is the *Advances in Chemistry Series* published by the American Chemical Society. Another series is published in *Annals of the New York Academy of Science*. Both series are largely based on symposia organized by scientific societies. There are several sources of review articles: annual reviews, special review periodicals, and special issues or selected parts of regular periodicals. Since 1959, *Chemical Abstracts* of the American Chemical Society publishes annually a *Bibliography of Chemical Reviews*. The number of such reviews is around 7,000 each year.

Since 1904, the Chemical Society (England) publishes authoritative summaries of the previous years' important papers in the form of Annual Reports on the Progress of Chemistry. A parallel series is published by the Society of Chemical Industry in the form of Reports on the Progress of Applied Chemistry. The reviews on foods are subdivided into the major areas and each section provides 100 to 200 references to original research papers, reviews, and proceedings of scientific conferences. Each year, the April issue of Analytical Chemistry is devoted to review papers. Various aspects of food analysis are covered every second year. Additional background information is provided in Analytical Chemistry by reviews in other areas, such as biochemistry, clinical chemistry, water analysis, and various areas of inorganic, physical, and organic analyses.

One of the most useful sources of current information, for the reader with sufficient background knowledge, are articles in annual publications in the form of Advances in . . . , Annual Review of . . ., Progress in . . . , and Methods in . . . . Those pertinent to food analyses are listed at the end of this chapter.

#### Theses

Theses and dissertations contain comprehensive reviews, generally in a limited area. While most of the new information in theses is published in scientific journals, the literature reviews are shortened to reduce cost of publication. Many countries publish lists of higher education degree theses. Theses from U.S. universities are processed since 1938 for microfilming by University Microfilms of Ann Arbor, Michigan. Abstracts of up to 600 words of such theses are published by *Dissertation Abstracts*. Theses of a chemical nature are listed in Chemical Abstracts, and the entire theses can be purchased from University Microfilms. An annual list of *U.S. Masters'* Theses in the Pure and Applied Sciences has been published since 1955–1956 by the Thermophysical Properties' Research Center, Purdue University, Lafayette, Indiana.

## Symposia

Programs of conferences and symposia are published in several journals. A comprehensive list is found in *Science*; the coverage of *Food Technology* is more selective and limited. In the area of food science, they include proceedings of meetings sponsored by government departments (such as various agencies of the U.S. Department of Agriculture). University Presses (i.e. MIT Press), professional groups, commercial publishing houses (i.e. Pergamon Press), and private companies (i.e. Campbell Soup Co.) frequently reproduce the lectures and stimulating discussions of meetings in which prominent scientists present invited review papers in the area of their competence. Several renowned Symposia on Foods held at Oregon State University have been published as hard cover books by the Avi Publishing Co.

Abstracts of papers presented at scientific meetings are published for members in the form of books (i.e. by the American Chemical Society or Federation of Biological Sciences) or are included in periodicals (i.e. Cereal Science Today for AACC, and J. American Oil Chemists' Society for AOCS). The abstracts are particularly useful in learning of the most recent developments.

#### **Trade Publications**

The purpose of most house organs and trade publications is to sell products. Most such advertisements contain valuable information. The publications of many manufacturers are on a high scientific level. Some of these publications are the best sources on properties and applications of specialized equipment and chemicals. Several manufacturers issue periodically bibliographies and abstracts of technical-scientific articles in a specific area; some publish periodicals that reproduce pertinent articles from regular scientific journals; and some prepare detailed handbooks giving specifications, properties, and details of analytical procedures in selected areas (micro-

biology, enzymatic assay, electrophoresis, immunochemistry, automation in analytical chemistry). Industrial manuals are, of course, indispensable in installing, using, and servicing equipment. Several scientific journals prepare periodically lists of major commercial supply houses. A Comprehensive Guide to Scientific Instruments is published annually in *Science*.

#### **Translations**

Scientific journals continue to be the most important source of detailed information. Searching the literature becomes more difficult with the increased quantity of available literature and more publications in foreign languages. Many of the latter have a summary in English. In addition, active programs for translation from Slavic, Japanese, and Chinese literature are available.

#### Abstracts

Information on important publications is available in bibliographical lists or abstracting journals. *Chemical Abstracts* publishes over 200,000 abstracts a year, selected from about 10,000 journals in over 50 languages. The section on biochemistry covers various aspects of biology and chemistry of materials of plant, animal, and microbial origin. Included are informative abstracts of scientific original papers, patents, and some reviews; as well as lists of theses, monographs, books, reviews, and proceedings.

Several periodicals publish abstracts in a more restricted field. Analytical Abstracts and the abstract section of Z. Analytische Chemie are concerned primarily with analytical procedures. The abstract sections in J. Science Food and Agriculture and Z. Lebensmitteluntersuchung Forschung cover production, processing, storage, chemistry, and analyses of foods and agricultural products. The abstract sections in J. American Oil Chemists' Society, J. Institute of Brewing, Wallerstein Laboratories Communications, and many others cover in a broad sense the respective areas. Food patents are abstracted in Food Technology. Some research organizations prepare excellent abstracts for member companies (Corn Industries Research Foundation) or for the general public (Washington State University). Information on cereals is abstracted in Germany by Documenta Cerealia and published as a monthly addition to the weekly periodical Die Muhle.

Abstracts on current work (including theses) in East-European countries may be found as an appendix to the journal *Die Nahrung*. Abstracts in the *Zentr. Dok. Dienst Sozialist. Lander-Nahrung und* 

Ernahrung—are an excellent source of information on scientific work in foods and nutrition. Although some western periodicals are abstracted, the emphasis is on East-European publications. Some of the latter journals are not abstracted by *Chemical Abstracts* and are generally unavailable to the English reader.

The journal Food Science and Technology—Abstracts has been published since 1969 by the International Food Information Service. The Institute of Food Technologists in the United States; the Commonwealth Agricultural Bureau, headquartered in England; and the Institut fur Dokumentationswesen of the German Democratic Republic are the sponsors.

In addition to the comprehensive *Chemical Titles*, published by the American Chemical Society, several publications provide more limited bibliographical lists. Selective lists are included in some periodicals such as *J. Chromatography* and *J. Lipid Research*.

Food analysts use primarily methods approved by various associations such as the Association of Official Analytical Chemists, American Association of Cereal Chemists, American Oil Chemists' Society, American Public Health Association, and American Society of Brewing Chemists (and their national or international counterparts). Most of the methods recommended by the United States and other organizations have been developed after years of collaborative testing and are considered reliable and official. To understand the background, limitations, and significance of analytical findings the analyst must acquaint himself with the current developments and periodically survey the pertinent sources.

A selected list of such sources is given at the end of this chapter.

#### BIBLIOGRAPHY

#### General Literature References

Anon. 1970. The IFT World Directory and Guide. Institute of Food Technologists, Chicago, Ill.

Becker, J., and Hayes, R. M. 1964. Information Storage and Retrieval: Tools, Elements, Theories. J. Wiley & Sons, New York.

BOTTLE, R. T. (Editor). 1962. Use of the Chemical Literature. Butterworths, London.

Burman, C. R. 1965. How to Find Out in Chemistry. Pergamon Press, New York.

Crane, E. J., Patterson, A. M., and Marr, E. B. 1957. A Guide to the Literature of Chemistry. J. Wiley & Sons, New York.

Dyson, G. M. 1951. Chemical Literature. Longmans and Green, London.Gould, R. F. (Editor). 1961. Searching the Chemical Literature. Advan.Chem. Ser. 30. Am. Chem. Soc., Washington, D.C.

Mellon, M. G. 1965. Chemical Publications: Their Nature and Use. McGraw-Hill Book Co., New York.

#### Handbooks, Dictionaries, General References

ABERHALDEN, E. 1920. Handbuch der Biologischen Arbeitsmethoden. Urban and Schwarzenberg, Berlin.

ABERHALDEN, E. (Editor). 1910–1933. Biochemisches Handlexikon. Springer-Verlag, Berlin.

Adams, R. (Editor). 1942. Organic Reactions. Chapman and Hall, London. Anon. 1964. Heinz Nutritional Data. H. J. Heinz Co., Pittsburgh, Penna.

Borgstrom, G. 1968. Principles of Food Science. The MacMillan Co., New York.

Burton, B. T. (Editor). 1968. The Heinz Handbook of Nutrition. McGraw Hill Book Co., New York.

CAMERON, E. J., and ESTY, J. R. 1950. Canned Foods in Human Nutrition. National Canners Association, Washington, D.C.

DIEMAIR, W. (Editor). 1965. Handbuch der Lebensmittelchemie. Springer-Verlag, Berlin.

Furia, T. E. (Editor). 1968. Handbook of Food Additives. Chemical Rubber Co., Cleveland.

JACOBS, M. B. (Editor). 1951. The Chemistry and Technology of Food and Food Products, 3 Vols. Interscience Publishers, New York.

 KLEIN, G. 1932. Handbuch der Pflanzenanalyse. Springer-Verlag, Berlin.
 LEES, R. (Editor). 1968. The Laboratory Handbook of Methods of Food Analysis. Chemical Rubber Co., Cleveland.

LONG, C. (Editor). 1961. Biochemist's Handbook. E. and F. Spon. Publishing Co., London.

McCance, R. A., and Widdowson, E. M. 1960. Composition of Foods. Medical Research Council, Special Rept. 297. H.M.S.O., London.

MEYER, L. H. 1960. Food Chemistry. Reinhold Publishing Corp., New York.

MILLER, J. (Editor). 1953. Organic Analysis. Interscience Publishers, New York.

Muller, E. (Editor). 1958. Methoden der Organischen Chemie. G. Thierme, Stuttgart, Germany.

PAECH, K., and TRACEY, M. V. (Editors). 1956. Modern Methods of Plant Analysis. Springer-Verlag, Berlin.

POTTER, N. N. 1968. Food Science. AVI Publishing Co., Westport, Conn.

Radt, F. (Editor). 1940. Elseviers' Encyclopedia of Organic Chemistry. Elsevier Press, Amsterdam.

RAUEN, v. H. M. 1956. Biochemisches Taschenbuch. Springer-Verlag, Berlin.

RAPPOPORT, Z. (Editor). 1968. Handbook of Tables for Organic Compound Identification. Chemical Rubber Co., Cleveland.

Sober, H. A. (Editor). 1968. Handbook of Biochemistry. Chemical Rubber Co., Cleveland.

STANDEN, A. (Editor). 1967. Kirk-Othmer's Encyclopedia of Chemical Technology. J. Wiley & Sons, New York.

STECHER, P. G. (Editor). 1960. The Merck Index of Chemicals and Drugs. Merck and Co., Rahway, N.J.

Watt, B. K., and Merrill, A. L. 1963. Composition of Foods, Agriculture Handbook. 8. U.S. Dept. Agr., Washington, D.C.

Weast, R. C. (Editor). 1968. Handbook of Chemistry and Physics, 49th Edition. Chemical Rubber Co., Cleveland.

Weissberger, A. (Editor). 1948. Physical Methods of Organic Chemistry. Interscience Publishers, New York.

WILLIAMS, R. J., and LANSFORD, E. M. (Editors). 1967. Encyclopedia of Biochemistry. Reinhold Publishing Corp., New York. Winton, A. L., and Winton, K. B. 1935-1939. Structure and Composition of Foods. J. Wiley & Sons, New York.

#### Reviews

Advances in Agronomy

Analytical Chemistry and Instrumentation

Carbohydrate Chemistry

Chemistry Series

Chromatography

Clinical Chemistry

Colloid Science

Comparative Biochemistry and Physiology

Enzymology and Related Subjects of Biochemistry

Food Research

Lipid Research

Protein Chemistry

Annual Reports on Progress of Chemistry

Annual Review of Biochemistry
Microbiology

Physiology

Bacteriological Reviews

Biological Reviews

Chemical Reviews

Chromatographic Reviews

Methods in Medical Research

Methods of Biochemical Analysis

Methods in Enzymology

Nutrition Abstracts and Reviews

Physiological Reviews

Progress in the Chemistry of Fats and Other Lipids

Medicinal Chemistry

Recent Advances in Food Science

Reports on the Progress of Applied Chemistry

Vitamins and Hormones

Yearbook of Agriculture—U.S. Dept. Agr.

# Abstracts, Bibliography, Indexes

Agricultural Index

Analytical Abstracts

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Bibliographic Index

Bibliographic Current List of Papers, Reports, and Proceedings of International

Meetings

Bibliography of Agriculture

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Chemical Abstracts

Chemical Titles

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Current Chemical Papers

Current Contents

World Bibliography of Bibliographies

#### Periodicals

Acta Chemica Scandinavia

Agricultural and Biological Chemistry (Tokyo)