

# MODERN METHODS OF FOOD ANALYSIS

Edited by

Kent K. Stewart

Department of Food Science  
and Technology  
Virginia Polytechnic Institute  
and State University  
Blacksburg, Virginia

John R. Whitaker

Department of Food Science  
and Technology  
University of California  
Davis, California

**avi**

AVI PUBLISHING COMPANY, INC.  
Westport, Connecticut

Copyright © 1984 by  
THE AVI PUBLISHING COMPANY, INC.  
Westport, Connecticut

All rights reserved. No part of this work covered by the copyright hereon may be reproduced or used in any form or by any means—graphic, electronic, or mechanical, including photocopying, recording, taping, or information storage and retrieval systems—without written permission of the publisher.

### **Library of Congress Cataloging in Publication Data**

Main entry under title:

Modern methods of food analysis.

Papers presented at Symposium on Modern Methods of Food Analysis held June 17 and 18, 1983 in New Orleans; sponsored jointly by the Institute of Food Technologists and the International Union of Food Science and Technology.

Bibliography: p.

Includes index.

1. Food—Analysis—Addresses, essays, lectures.

I. Stewart, Kent K. II. Whitaker, John R. III. Symposium on Modern Methods of Food Analysis (1983 : New Orleans, La.) IV. Institute Food Technologists. V. International Union of Food Science and Technology.

TX541.M56 1984 664'.07

84-14584

ISBN 0-87055-462-X

ABCDE 3210987654

Printed in the United States of America

# MODERN METHODS OF FOOD ANALYSIS

# **ift** Basic Symposium Series

Edited by  
INSTITUTE OF FOOD TECHNOLOGISTS  
221 N. LaSalle St.  
Chicago, Illinois

## **Other Books in This Series**

**FOOD PROTEINS**

*Whitaker and Tannenbaum*

**POSTHARVEST BIOLOGY AND BIOTECHNOLOGY**

*Hultin and Milner*

**IMPACT OF TOXICOLOGY ON FOOD PROCESSING**

*Ayres and Kirschman*

**FOOD CARBOHYDRATES**

*Lineback and Inglett*

**PHYSICAL PROPERTIES OF FOODS**

*Peleg and Bagley*

# Contributors

- ALVAREZ, R. (81).<sup>1</sup> Office of Standard Reference Materials, National Bureau of Standards, Gaithersburg, MD 20899
- BEECHER, G.R. (29). Nutrient Composition Laboratory, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Beltsville, MD 20705
- DESSY, R.E. (57). Chemistry Department, Virginia Polytechnic Institute and State University, Blacksburg, VA 24061
- HARNLY, J.M. (101). Nutrient Composition Laboratory, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Building 161, BARC East, Beltsville, MD 20705
- IHNAT, M. (129). Chemistry and Biology Research Institute, Agriculture Canada, Ottawa, Ontario, Canada K1A 0C6
- JAY, J.M. (227). Department of Biological Sciences, Wayne State University, Detroit, MI 48202
- JENNINGS, W. (319). Department of Food Science and Technology, University of California, Davis, CA 95616
- KIRK, J.R. (381). Research and Development, Campbell Soup Company, Camden, NJ 08101
- LENTO, H.G. (71). Corporate Analytical Laboratory, Campbell Institute for Research and Technology, Campbell Place, Camden, NJ 08101
- MILLER-IHLI, N.J. (101). Nutrient Composition Laboratory, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Building 161, BARC East, Beltsville, MD 20705
- PANGBORN, R.M. (265). Department of Food Science and Technology, University of California, Davis, CA 95616
- PARDUE, H.L. (1). Department of Chemistry, Purdue University, West Lafayette, IN 47907
- REINECCIUS, G.A. (293). Department of Food Science and Nutrition, University of Minnesota, St. Paul, MN 55108
- SCHWEIGERT, B.S. (xvii). Department of Food Science and Technology, University of California, Davis, CA 95616
- STEWART, K.K. (369). Department of Food Science and Technology, Virginia Polytechnic Institute and State University, Blacksburg, Virginia 24061
- VANDERSLICE, J.T. (29). Nutrient Composition Laboratory, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Washington, DC 20705

<sup>1</sup>Numeral in parentheses indicates the page on which the author's contribution begins.

xii CONTRIBUTORS

- WHITAKER, J.R. (187). Department of Food Science and Technology, University of California, Davis, CA 95616
- WOLF, W.R. (101). Nutrient Composition Laboratory, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Building 161, BARC East, Beltsville, MD 20705
- ZWEIG, G.J. (339). Environmental Protection Agency, Office of Pesticide Programs, Washington, DC 20460

<sup>1</sup> Present address: School of Public Health, 322 Warren Hall, University of California, Berkeley, CA 94720

# Preface

This Symposium on Modern Methods of Food Analysis was the seventh in a series of basic symposia, begun in 1976, on topics of major importance to food scientists and food technologists. The Symposium, sponsored jointly by the Institute of Food Technologists and the International Union of Food Science and Technology, was held June 17 and 18, 1983, in New Orleans immediately prior to the 43rd annual IFT meeting. Like the other six basic symposia, the program brought together outstanding speakers, from biochemistry, chemistry, food science, microbiology and nutrition, who are at the cutting edge of their specialty, and provided a setting where they could interact with each other and with the participants.

The Symposium and this book are dedicated to the memory of George F. Stewart (1908–1982) who made so many important contributions to the field of food science, including that of food analysis. Bernard S. Schweigert has documented George F. Stewart's contributions in the Dedication of this book.

The field of food analysis touches all of us, whether teacher, scientist, regulator, politician, secretary or consumer. Any time a question of how much of an ingredient or the presence of a compound in food is raised, the answer must always be based upon analysis. For some of us, analysis is our life's work; for others it is only a tool to be used as necessary; for others of us it provides protection and quality assurance. Whatever our role in relation to food analysis, it is important that we all communicate with each other in maximizing the advantages of food analysis.

Selection of Symposia topics and of the Co-Chairs and assistance in planning and executing the program is the responsibility of the Basic Symposium Committee, which includes members from industry, government and academia. The 1983 Basic Symposium Committee members were Darrel E. Goll, chair, Ernest J. Briskey, immediate past chair,

Larry R. Beuchat, John P. Cherry, Richard V. Lechowich, Louis B. Rockland, Richard A. Scanlan and Henry G. Schwartzberg.

The success of the seventh basic symposium was also the result of the expert assistance of Owen Fennema, 1983 President of IFT, Calvert L. Willey, Executive Director of IFT, John B. Klis, Director of Publications, and the IFT staff who provided moral support and publicity and coordinated physical planning including registration, meeting rooms, hotel reservations and the numerous other details of such a two-day symposium.

John Klis coordinated all details of interface with the publisher and Anna May Schenck, JFS Assistant Scientific Editor, served for the seventh time as copy editor for the proceedings. Their capabilities, patience and professionalism in the face of pending deadlines were of immense value.

It is to the authors of the chapters of this book that we owe our deepest gratitude. They heeded the call to teach others—not only at the basic symposium but for many years to come through the written word—the importance of the field of modern food analysis. Their unselfish devotion to knowledge and to the education of others should be an example to all of us.

It is with great humility yet with a strong sense of purpose and pride that we, one the son and the other a junior colleague who learned much through his personal encouragement, join in the dedication of this book to the memory of George F. Stewart.

KENT K. STEWART  
JOHN R. WHITAKER



# Dedication:

## GEORGE F. STEWART

### The Man and the Scientist

*B.S. Schweigert*<sup>1</sup>

It is appropriate to introduce this book with comment on a distinguished colleague Professor George F. Stewart who was keenly interested in methods of analysis as well as in the development of new and modern methods and their applications to food systems.

For perspective, a quote from a resolution adopted by the Executive Committee of the Institute of Food Technologists on March 25, 1982, just a week after his death at age seventy-four, follows:

George F. Stewart was a man who touched the lives of many in the Institute of Food Technologists, who initiated many of the activities and projects we take for granted. In his roles as a charter member of the society, as executive editor of the IFT journals, as winner of the prestigious International and Appert Awards, as Fellow, and finally as president of the Society, there is hardly an area of IFT which has not felt his guidance and direction.

His concerns were wide-ranging: He was as interested in forming the local IFT section at Ames, Iowa, as he was the International Union of Food Science & Technology; in being an IFT Scientific Lecturer as in helping bring about the First International Congress of Food Science and Technology. He was not narrow in his organizational outlook: He was as at home as a consultant to industry as he was as advisor to many government agencies and committees. He found it as rewarding to practice as to supervise, and was as productive in basic research as in the Experiment Station. He made time for his family, and for an active outdoors life, and truly enjoyed his fellow man.

<sup>1</sup> Department of Food Science and Technology, University of California, Davis, California 95616

Dr. Stewart was recognized as a Fellow by three scientific societies and served as President of the Society of Nutrition Education in its early formative years. See the accompanying tabulation.

#### GEORGE F. STEWART—AWARDS AND HONORS

<i>Awards</i>	
International, IFT	1968
Nicholas Appert, IFT	1974
<i>Fellow</i>	
American Association for The Advancement of Science	1963
Institute of Food Technologists (IFT)	1971
Poultry Science Association	1949
<i>President</i>	
Institute of Food Technologists	1968
International Union of Food Science & Technology	1970-1974
Society of Nutrition Education	1973

It is also appropriate to provide a few additional highlights on his contributions to teaching, research, and public service. Professor Stewart was active in teaching the introductory course in food science (FS&T 1, Introduction to Food Science), the food packaging course (FS&T 131, Packaging Processed Foods), and after full retirement in 1975, he volunteered to teach FS&T 109, Principles of Quality Assurance in Food Processing that filled a critical teaching need in the Department that of Food Science and Technology, University of California, Davis. This author had the opportunity to work closely with him in the teaching of FS&T 1, and he and another distinguished colleague Professor Maynard Amerine wrote the text published by Academic Press entitled *Introduction to Food Science and Technology*. It is significant to note that the final edited second edition was completed just before Professor Stewart's illness and death.

The following quote from the preface to the second edition illustrates the thinking that he and Professor Amerine provided to readers of the second edition.

Academic training for technical careers in food science and technology requires a broad, in-depth education both in certain sciences and in selected engineering specialties. It is precisely because of the complex nature of food and its processing and the requirement for a rigorous scientific/technical training that food science and technology offers an exceptional opportunity and a real challenge to the bright applications-oriented science student seeking a rewarding career.

Dr. Stewart's contribution to teaching also included the guidance of graduate students, particularly when he served on the faculty at Iowa State University. A member of the Cooperative Extension Faculty in

Food Science and Technology at the University of California—Davis, Dr. A.W. Brant was one of his graduate students at Iowa State University. Three other faculty members have contributed key treatises to this volume—Professors Walter Jennings, Rose Marie Pangborn, and John Whitaker.

In the area of research and research needs, Professor Stewart was a leader in emphasizing trends occurring in the food and allied industries. This included two relatively new aspects of the interdisciplinary field of food science and technology, namely, food engineering and sensory science. He also highlighted nutrition and food analysis in the address he presented when he was President of the International Union of Food Science & Technology during the Fourth International Congress of Food Science and Technology in 1974. The following is a quote from the paper he developed entitled "Tomorrow's Foods—Obligations and Opportunities for the Food Scientist. Chemical Composition of Processed Foods, Especially Their Nutrient Content."

We are woefully lacking in reliable data about the nutrient composition of our foods. Equally serious is a lack of sensitive, accurate, and reproducible methods of analysis for nutrients. While many scientists will not find analytical studies very challenging or exciting, it is essential that we obtain reliable information about the nutritional value of tomorrow's food. Someone must address himself to this neglected area of research. Perhaps some of you can be induced to do so.

This leadership is clearly exemplified by this symposium, including the contributions made by his son, Dr. Kent Stewart, the cochairman of this symposium, and head of the Department of Food Science and Technology, Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

Dr. Stewart's leadership and public service have already been referred to with respect to participation and leadership in scientific societies. He also served as Executive Editor of the IFT journals, *Food Technology* and *Journal of Food Science* during the period 1960–1965 and as a coeditor with Academic Press, Inc., for two major publication series: (1) *Advances in Food Research*, and (2) *Monograph Series in Food Science and Technology*. An achievement of major importance was his leadership as a cofounder of *Food Science and Technology Abstracts* in 1969.

Dr. Stewart also led other important developments in public service including working closely with Howard Mattson, Director of Public Information of the Institute of Food Technologists, and in developing food advertising guidelines in a paper entitled "Food Advertising and Promotion—A Plea for Change." This paper is highly recommended.

Dr. Stewart's interest in public service extended beyond the imme-

diated area of professional interest in food science, and he was very active in working with various groups on environmental issues, particularly preservation of wild streams in California and Montana and the protection of habitat for fish and wild birds and other animals. His expertise in this area was increased by his keen interest in fly fishing!

In summary, it is most fitting that this book be dedicated to Professor George F. Stewart in view of his key leadership in emphasizing the basic sciences associated with the interdisciplinary field of food science and technology including food chemistry and even more specifically modern methods of food analysis. His qualities as a person and his perception of the important scientific issues provide the basis for noting further advances in this important field such as those presented in the chapters that follow.

## BIBLIOGRAPHY

- STEWART, G.F. 1974. Tomorrow's foods—obligations and opportunities for the food scientist, pp. 127–133. *In* Proc. IV Int. Congr. Food Sci. Technol., Vol. 6. Instituto de Agroquímica y Tecnología de Alimentos, Valencia, Spain.
- STEWART, G.F., and AMERINE, M.A. 1982. *Introduction to Food Science and Technology*. 2nd Edition. Academic Press, New York.
- STEWART, G.F., and MATTSON, H. 1978. Food advertising and promotion—A plea for change. *Food Tech.* 32(11): 30–33.

# Contents

<b>Contributors</b>	xi
<b>Preface</b>	xiii
<b>Dedication: George F. Stewart—The Man and the Scientist</b> B.S. Schweigert	xvii
 <b>1 SYSTEMS APPROACH TO FOOD ANALYSIS</b>	 1
<i>Harry L. Pardue</i>	
Rationale for Problem-Oriented Role of Analysis	1
Rationale for Systems Approach to Analysis	3
Illustrative Example	5
Functional Processes	5
Operational Choices	11
Discussion	25
Epilogue	27
Bibliography	28
 <b>2 DETERMINATION OF NUTRIENTS IN FOODS: FACTORS THAT MUST BE CONSIDERED</b>	 29
<i>G.R. Becher and J.T. Vanderslice</i>	
Introduction	29
Selection of Nutrients for Analysis	30
Selection of Foods for Nutrient Analysis	50
Summary	53
Bibliography	54
 <b>3 COMPUTERS IN THE FOOD ANALYSIS LABORATORY</b>	 57
<i>Raymond E. Dessy</i>	
Hardware	58
Network Architecture	59
Operating Systems	63
Languages	64

# vi CONTENTS

	Laboratory Information Management Systems	66
	The Company Environment	67
	Robotics—Computerization vs. Automation	68
	Implementation and Installation	68
	Bibliography	69
<b>4</b>	<b>SAMPLE PREPARATION AND ITS ROLE IN NUTRITIONAL ANALYSIS</b>	<b>71</b>
	<i>Harry G. Lento</i>	
	Introduction	71
	Reducing Variation by Unit Compositing	72
	Problems in Unit Compositing and Sample Preparation	73
	Variability and Label Value	77
	Preparation of Sample	78
	Summary	79
	Bibliography	79
<b>5</b>	<b>NBS STANDARD REFERENCE MATERIALS FOR FOOD ANALYSIS</b>	<b>81</b>
	<i>Robert Alvarez</i>	
	Introduction	81
	Reliability of Food Analyses	83
	Validation of Methods and Analytical Data	85
	NBS Standard Reference Materials for Food Analysis	86
	SRMS under Development	92
	Applications of NBS Standard Reference Materials	93
	Future SRMS for Food Analysis	93
	Bibliography	94
	Appendix	95
<b>6</b>	<b>QUALITY ASSURANCE OF ANALYSIS OF INORGANIC NUTRIENTS IN FOODS</b>	<b>101</b>
	<i>James M. Harnly, Wayne R. Wolf, and Nancy J. Miller-Ihli</i>	
	Introduction	101
	General Aspects of Quality Assurance for Atomic Spectrometry	102
	Case Study—Determination of Inorganic Nutrients in U.S. Fruit and Vegetable Juices	112
	Bibliography	126
<b>7</b>	<b>ATOMIC ABSORPTION AND PLASMA ATOMIC EMISSION SPECTROMETRY</b>	<b>129</b>
	<i>M. Ilnat</i>	
	Introduction and History of Analytical Atomic Absorption and Emission Spectrometry	129
	Basic Principles of Theory and Instrumentation	133
	Scope of Applications to Food Analysis	139

Fundamental Considerations of the Absorption and Emission Techniques	147
Analytical Procedures for Food Analysis	155
Newer Developments and Future Directions in Atomic Spectrometry	159
Bibliography	161
<b>8 REFLECTANCE SPECTROSCOPY</b>	<b>167</b>
<i>Karl H. Norris</i>	
Introduction	167
Optical Geometry for Diffuse Reflectance	167
Reflectance Standards	170
Factors Affecting Reflectance Data	171
Data Treatments	175
Regression Techniques for Calibration	177
Applications	179
Conclusion	185
Bibliography	185
<b>9 BIOLOGICAL AND BIOCHEMICAL ASSAYS IN FOOD ANALYSIS</b>	<b>187</b>
<i>John R. Whitaker</i>	
The Whole Animal in Analysis	188
Whole Cells as Analytical Tools	192
Immunoassay Techniques	196
Enzyme-Based Analytical Assays	210
Summary	219
Bibliography	221
<b>10 MICROBIOLOGICAL ASSAYS</b>	<b>227</b>
<i>J.M. Jay</i>	
Amino Acids	228
B Vitamins	233
Improved Microbiological Assay Techniques	241
Evaluation of Protein Quality	245
Antibiotics	253
Mutagens	256
Miscellaneous Substances	257
Bibliography	258
<b>11 SENSORY ANALYSIS AS AN ANALYTICAL LABORATORY TOOL IN FOOD RESEARCH</b>	<b>265</b>
<i>R.M. Pangborn</i>	
Introduction	265
Ad Libitum Mixing	268
Time-Intensity Procedures	272
Deviation-from-Reference Descriptive Analysis	277

viii CONTENTS

	Conclusions	287
	Bibliography	288
<b>12</b>	<b>DETERMINATION OF FLAVOR COMPONENTS</b>	<b>293</b>
	<i>Gary A. Reineccius</i>	
	Introduction	293
	Sample Preparation	294
	Isolation of Food Flavors	295
	Flavor Isolation	295
	Analysis of Flavor Isolates	309
	Bibliography	316
<b>13</b>	<b>GAS CHROMATOGRAPHY</b>	<b>319</b>
	<i>Walter Jennings</i>	
	Introduction	319
	Basic Principles of Gas Chromatography	319
	Problem Areas in Food Analyses	322
	Developments in GC Hardware/Methodology	322
	Recent Advancements	334
	Bibliography	337
<b>14</b>	<b>RECENT DEVELOPMENTS IN THE ANALYSIS OF PESTICIDES</b>	<b>339</b>
	<i>Gunter Zweig</i>	
	Introduction	339
	Methods of Pesticide Analysis	340
	The Modern Pesticide Analytical Laboratory	352
	Multiresidue Methods	353
	Residue Analysis of Carbaryl	361
	Pesticide Analysis, Then and Now	365
	Bibliography	366
<b>15</b>	<b>FLOW INJECTION ANALYSIS: A NEW TOOL FOR THE AUTOMATION OF THE DETERMINATION OF FOOD COMPONENTS</b>	<b>369</b>
	<i>Kent K. Stewart</i>	
	Introduction	369
	Flow Injection Analysis	370
	FIA Stopped Flow Systems	373
	FIA Dilution Systems	373
	Pseudotitrations and Other Exponential Dilution Systems	373
	Theory of FIA Systems	376
	Theory of Pseudotitrations and Exponential Dilution Chambers	377
	Some General Comments and Observations	377
	Bibliography	378



<b>16 MODERN LIQUID CHROMATOGRAPHY: EVOLUTION AND BENEFITS</b>	<b>381</b>
<i>James R. Kirk</i>	
Introduction	381
Chromatographic Concepts	382
Band Broadening	383
Separation Mechanisms	383
Extracolumn Band Broadening	385
Resolution and Separation	385
Modern Liquid Chromatography Systems	386
Pumping Systems	386
Gradient Elution	387
Sample Injectors	387
Detectors	388
Columns	389
Ion-Pair Chromatography	393
Properties and Characteristics of Solvents	394
Selected Chromatographic Techniques for Quantitation of Micronutrients	395
Computers and HPLC	404
Bibliography	405
<b>Index</b>	<b>407</b>