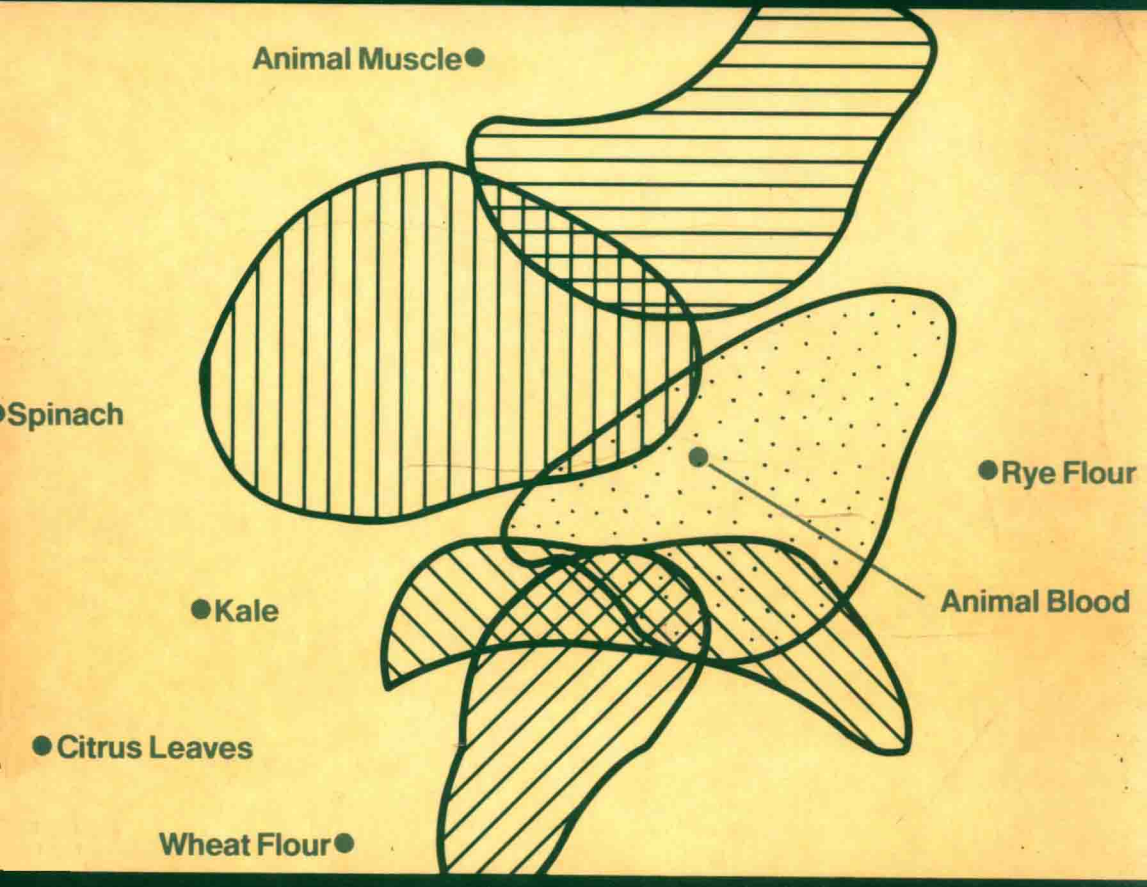
BIOLOGICAL REFERENCE MATERIALS:

Availability, Uses, and Need for Validation of Nutrient Measurement



Edited by Wayne R. Wolf

BIOLOGICAL REFERENCE MATERIALS

Availability, Uses, and Need for Validation of Nutrient Measurement

Edited by
WAYNE R. WOLF
Nutrient Composition Laboratory
Human Nutrition Center
United States Department of Agriculture
Beltsville, Maryland

A Wiley-Interscience Publication
JOHN WILEY & SONS
New York • Chichester • Brisbane • Toronto • Singapore

Library of Congress Cataloging in Publication Data:
Main entry under title:

Biological reference materials.

"A Wiley-Interscience publication."
Includes index.
1. Food—Analysis. 2. Natural products—Analysis. 3. Pollutants—Analysis. 4. Food—Standards. 5. Natural products—Standards. 6. Pollutants—Standards. I. Wolf, Wayne R., 1943–
TX535.B56 1984 664'.07 84-7568
ISBN 0-471-80636-6

BIOLOGICAL REFERENCE MATERIALS

Contributors

Sebastian T. Agnese
Centers for Disease Control
Center for Environmental Health
Clinical Chemistry Division
Atlanta, Georgia

Robert Alvarez
Office of Standard Reference Materials
National Bureau of Standards
U.S. Department of Commerce
Washington, DC

David Bayse
Centers for Disease Control
Center for Environmental Health
Clinical Chemistry Division
Atlanta, Georgia

Carol J. Bell
Centers for Disease Control
Center for Environmental Health
Clinical Chemistry Division
Atlanta, Georgia

Shier S. Berman
Division of Chemistry
Analytical Chemistry Section
National Research Council of Canada
Ottawa, Ontario, Canada

H. J. M. Bowen
Chemistry Department
Reading University
Whiteknights
Reading, Berkshire
United Kingdom

Edgar R. Elkins
National Food Processors Association
Chemistry Division
Washington, DC

W. Harry Hannon
Centers for Disease Control
Center for Environmental Health
Clinical Chemistry Division
Atlanta, Georgia

Harry S. Hertz
Director
Center for Analytical Chemistry
National Bureau of Standards
U.S. Department of Commerce
Washington, DC

Milan Ihnat
Chemistry and Biology Research Institute
Research Branch
Agriculture Canada
Ottawa, Ontario, Canada

Sam A. Margolis
Center for Analytical Chemistry
National Bureau of Standards
U.S. Department of Commerce
Washington, DC

Herbert Muntau
Commission of the European Communities
Chemistry Division
Joint Research Centre, Ispra
Ispra, Italy

James E. Myrick
Centers for Disease Control
Center for Environmental Health
Clinical Chemistry Division
Atlanta, Georgia

Robert M. Parr
Medical Applications Section
International Atomic Energy Agency
Vienna, Austria

Kristine Y. Patterson
Vitamin and Mineral Nutrition Laboratory
Beltsville Human Nutrition Research Center
U.S. Department of Agriculture
Beltsville, Maryland

Don C. Reamer
Elemental Analysis Research Center
U.S. Food and Drug Administration
Cincinnati, Ohio

Hal T. Slover
Nutrient Composition Laboratory
Beltsville Human Nutrition Research Center
U.S. Department of Agriculture
Beltsville, Maryland

Markus Stoeppler
Institute of Applied Physical Chemistry
Nuclear Research Center (KFA)
Juelich, Federal Republic of Germany

James T. Tanner
Division of Nutrition
Bureau of Foods
U.S. Food and Drug Administration
Washington, DC

Raymond H. Thompson, Jr.
Nutrient Composition Laboratory
Beltsville Human Nutrition Research Center
U.S. Department of Agriculture
Beltsville, Maryland

George Uriano
Chief, Measurement Services
National Bureau of Standards
U.S. Department of Commerce
Washington, DC

Claude Veillon
Vitamin & Mineral Nutrition Laboratory
Beltsville Human Nutrition Research Center
U.S. Department of Agriculture
Beltsville, Maryland

Peter J. Wagstaffe
Community Bureau of Reference (BCR)
Commission of the European Communities
Brussels, Belgium

Stephen A. Wise
Center for Analytical Chemistry
National Bureau of Standards
U.S. Department of Commerce
Washington, DC

Wayne R. Wolf
Nutrient Composition Laboratory
Beltsville Human Nutrition Research Center
U.S. Department of Agriculture
Beltsville, Maryland

Rolf Zeisler, Head
Activation Analysis Section
Center for Analytical Chemistry
National Bureau of Standards
U.S. Department of Commerce
Washington, DC

Preface

The greatly increased awareness and concern of public health interests regarding optimal human intake of nutrients and/or exposure to harmful substances in the environment has led to a much increased demand for more detailed analytical data on the contents of these substances in a variety of biological materials. Many new analytical methodologies and techniques are being developed to obtain more specific and more precise information at lower levels of analyte in these materials. The increasing demand for data is leading to a demand for more "routine" methods that are fast, economic, and capable of higher throughput of analyses. These methods are part of overall measurement systems reflecting not only the techniques and procedures of the methodology, but also the conditions under which they are used and the persons using them. To validate these measurement systems and to link these new methods to more definitive, albeit more complex and expensive, methods requires validation, quality control, and accuracy transfer by use of appropriate certified biological reference materials (RM) capable of testing the whole measurement sytem.

To evaluate the present status of availability, uses, and needs for these biological RM's, a symposium on this topic was held at the 10th Annual Meeting of the Federation of Analytical Chemistry and Spectroscopic Societies (FACSS), Philadelphia,

PA, September 1983. The purpose of this symposium was to bring together international efforts on development and distribution of certified biological RM's and other homogeneous and well-characterized biological materials useful for improvement of analytical methods, with special emphasis on the measurement system of nutrient analysis. The papers presented at the symposium are being published in this book with the hope of documenting the rather widespread activity in this area and focusing these activities toward the goal of needed improvements in accuracy and interlaboratory comparability in this measurement system.

The first half-day session of the symposium included talks detailing "certified" biological RM's available or in preparation. This included discussion of one of the earliest, kale, prepared by Dr. H. J. M. Bowen (Reading, U.K.). Also discussed were the programs of the U.S. National Bureau of Standards; the International Atomic Energy Agency, Vienna, Austria; the Community Bureau of Reference, Commission of the European Communities, Brussels, Belgium; and the National Research Council of Canada, Ottawa; followed by a discussion evaluating the presently available certified biological RM's for inorganic nutrient analysis.

The second session began with several papers describing efforts to prepare large pools of units of homogeneous and well-characterized materials that could potentially be used for the improvement of analytical methodology. These pools of materials have been put together either as candidate RM's or as quality control materials for large-scale studies. Not all of these materials would have a potential for formal certification process, mainly because of the extreme cost of this process for each individual material or because of the somewhat limited number of units of each material (greater than 1000 units of each certified biological RM are required to ensure supply for a long enough time to justify the cost of certification). However, these are well-characterized materials for specific uses and would be very valuable for specific method development and validation.

The papers describing available biological RM's almost exclusively described materials for which the analytes of interest were the inorganic elements. This reflects the present extreme lack of available

materials with certified or well-characterized content of organic nutrient constituents. This is an area of recognized need. The third session of the symposium addressed this need in the form of papers describing efforts of several groups to prepare and use appropriate quality control materials for organic nutrient analysis. A previous workshop on this topic had been held at the National Bureau of Standards, Gaithersburg, MD, in October 1980. A number of recommendations for biological RM's for organic nutrient analysis were presented in the report of that workshop. That report is included in this publication (Appendix) in order to significantly round-out coverage for all classes of nutrients and to make more widely available the ideas and recommendations formulated in that workshop.

The formal presentations of the symposium concluded with discussion of the programs on environmental specimen banking being carried out in Germany and the United States. The existence in these large-scale monitoring programs of a wide variety of biological materials that are extremely well-characterized for a number of nutrients and other constituents and that are appropriately documented and stored over long periods of time can become a stable analytical base for measurement systems involving biological materials. These programs can be very important future focal points regarding analytical methodology and quality assurance. The symposium ended with an open discussion by the participants.

All manuscripts for the proceedings were submitted to the editor before the meeting or shortly thereafter. The manuscripts were distributed to qualified reviewers and all authors were given a chance to respond to or comment on the reviews. All papers were retyped in camera-ready copy by the word processing unit at the Beltsville Agricultural Research Center, whose thoroughly professional efforts and extreme cooperation are very greatly appreciated by the editor. Acknowledgment should also go to the Nutrient Composition Laboratory, USDA, Beltsville, for finanical support of the symposium and the publication, and to the organizers and support staff of the FACSS meeting for arrangements to hold this symposium in a scientific atmosphere very conducive

to the excellent interactions and discussions that took place.

Thanks go to the participants of the symposium for their excellent cooperation, enthusiasm, and valuable contributions. Special thanks to Dr. Herbert Muntau, Ispra, Italy, who first suggested the need for a meeting of biological RM producers during a visit to his laboratory in October 1982.

While this volume was in preparation, additional information was forwarded to me concerning the reference material program of the National Institute of Environmental Science (JAPAN). Unfortunately this program was not represented at the workshop. This is a major program which has available several certified reference materials including Pepperbush (NIES CRM No. 1), Pond Sediment (NIES CRM No. 2), Chlorella (NIES CRM No. 3), and Mussel (NIES CRM No. 6). In preparation are Human Serum (CRM No. 4) and Hair (CRM No. 5) to be used for trace element analysis. Values for the certified materials are listed at the end of Chapter 17. Additional information and details of this program can be obtained from Dr. K. Okamoto, Division of Chemistry and Physics, National Institute of Environmental Sciences, (NIES), Yatabe, Isukuba, Ibaraki, 305 JAPAN.

Wayne R. Wolf

Beltsville, Maryland
October 1984

BIOLOGICAL REFERENCE MATERIALS

Contents

PART 2

HOMOGENEOUS AND WELL-CHARACTERIZED MATERIALS

PART 3

QUALITY CONTROL MATERIALS FOR NUTRIENT ANALYSIS

PART 1

Certified Materials Available or in Preparation