



0047626

**Sourcebook of Methods of Analysis  
for Biomass and Biomass Conversion  
Processes**

T.A. Milne

A.H. Brennan

B.H. Glenn

Produced by the Solar Technical Information Program,  
US Department of Energy, with joint funding from the  
International Energy Agency; Energy, Mines and  
Resources Canada; and the Solar Energy Research  
Institute



**ELSEVIER APPLIED SCIENCE**  
LONDON and NEW YORK

ELSEVIER SCIENCE PUBLISHERS LTD  
Crown House, Linton Road, Barking, Essex IG11 8JU, England

*Sole Distributor in the USA and Canada*  
ELSEVIER SCIENCE PUBLISHING CO., INC.  
655 Avenue of the Americas, New York, NY 10010, USA

© 1990 ELSEVIER SCIENCE PUBLISHERS LTD

**British Library Cataloguing in Publication Data**

Milne, T. A. (Thomas A.)  
Sourcebook of methods of analysis for biomass and biomass  
conversion processes.  
1. Energy sources. Biomass  
I. Title II. Brennan, A. H. III. Glenn, B. H.  
662.6

ISBN 1-85166-527-7

**Library of Congress CIP data applied for**

Published in collaboration with the  
Development and Communications Office



Solar Energy Research Institute  
A Division of Midwest Research Institute  
1617 Cole Boulevard  
Golden, Colorado 80401, USA

Operated for US Department of Energy

SERI/SP-220-3548  
DE89009494

No responsibility is assumed by the Publisher for any injury and/or damage to persons or property as a matter of products liability, negligence or otherwise, or from any use or operation of any methods, products, instructions or ideas contained in the material herein.

**Special regulations for readers in the USA**

This publication has been registered with the Copyright Clearance Center Inc. (CCC), Salem, Massachusetts. Information can be obtained from the CCC about conditions under which photocopies of parts of this publication may be made in the USA. All other copyright questions, including photocopying outside the USA, should be referred to the publisher.

All rights reserved. No part of this publication may be reproduced, stored in a retrieval system, or transmitted in any form or by any means, electronic, mechanical, photocopying, recording, or otherwise, without the prior written permission of the publisher.

**Sourcebook of Methods of Analysis  
for Biomass and Biomass Conversion Processes**

## Preface

Since the oil embargo of the 1970s, researchers around the world have made tremendous progress in developing and improving methods for converting biomass—trees, plants, and organic wastes—to useful fuels and chemicals. However, the lack of relevant standards and analytical methods has made comparison of results between laboratories and nations difficult. This *Sourcebook of Methods of Analysis for Biomass and Biomass Conversion Processes* is the result of an international effort to begin to fill this gap.

In 1986, the International Energy Agency began sponsorship of a ‘Voluntary Standards Activity’, designed to provide comparability of research results, increase research efficiency, and provide quality assurance to both researchers and industry. Canada, Finland, New Zealand, and the United States supported the activity initially; Italy joined in 1988. Major support also came from Energy, Mines and Resources Canada (E, M & R); the US Department of Energy’s Solar Technical Information Program (STIP); and the Solar Energy Research Institute (SERI).

The sourcebook presents titles and abstracts (when available) of methods relevant to all aspects of biomass conversion—from analyzing feedstocks to evaluating performance of biofuels. The authors assembled the sourcebook at SERI by searching the literature, reviewing industrial standards, and soliciting suggestions from scientists in the field. In addition, Finland prepared a special report (Appendix III) on measuring the efficiencies of small boilers and biomass stoves and furnaces.

T.A. MILNE  
A.H. BRENNAN  
B.H. GLENN

## Acknowledgments

This sourcebook was produced by the Solar Technical Information Program (STIP), US Department of Energy. The contents are the result of contributions by many nations and individuals. Financial support came from IEA member countries Canada, Finland, Italy, New Zealand, and the United States; from the Solar Energy Research Institute's Director's Development Fund; from Energy, Mines and Resources Canada; and from STIP. Special thanks are due the Technical Advisory Board members, Dan Asplund, the late Doug Hayes, and Keith Mackie, and the IEA Biomass Conversion Annex Leader, Don Stevens. The following were most helpful in the working group panels and in the round-robin interlaboratory comparisons: Peter Dare, Doug Elliott, Hermann Esterbauer, Karel Grohmann, Barbel Hahn-Hagerdahl, Ted Hillis, James Linden, Jim McKinley, Keith Mackie, Ralph Overend, Michael Paice, Vic Phillips, Roger Sutcliffe, Olof Theander, and K. K. Wu, among others. Assistance in planning for standard reference materials was generously provided by Jerry Cherney, Janet Cushman, Gene Domalski, Keith Mackie, Jack Ranney, Stan Rasberry, Bill Reed, and Olof Theander. The staff at the National Center for Standards and Certification at the National Institute for Standards and Technology (NIST, formerly National Bureau of Standards) were most helpful in locating relevant standards. Heikki Oravainen contributed the special report on combustor efficiencies. Thanks also go to the members of the working group who met in Denver, to Diane Christodaro who arranged the meeting, and to the many correspondents who responded to questionnaires, sent in methods, and joined the round-robin tests. Finally, we are especially indebted to Helena Chum for her technical guidance and contributions throughout the project.

### NOTICE

This report was prepared as an account of work sponsored by an agency of the United States government. Neither the United States government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States government or any agency thereof.

## Contents

<i>Preface</i> . . . . .	v
<i>Acknowledgments</i> . . . . .	vi
Introduction . . . . .	1
Key to Standards-Setting Bodies Cited . . . . .	3
Standards and Analytical Methods . . . . .	5
Feedstock Sampling and Preparation . . . . .	7
Physical and Thermal Properties . . . . .	19
Density and Specific Gravity . . . . .	21
Particle Size Determination and Sieving . . . . .	27
Pore Size and Capacity . . . . .	37
Heats of Combustion . . . . .	39
Thermal Properties . . . . .	45
Elemental Analyses . . . . .	51
Ultimate Analysis . . . . .	53
Oxygen Analysis . . . . .	57
Nitrogen Analysis . . . . .	61
Sulfur Analysis . . . . .	67
Halogen Analysis . . . . .	71
Metals and Other Inorganics . . . . .	77
Chemical Component and Group Analyses . . . . .	87
Proximate Analysis . . . . .	89
Moisture . . . . .	93
Ash, Mineral Matter and Dirt . . . . .	105
Cellulose and Hemicellulose . . . . .	117
Hemicellulose . . . . .	123
Lignins . . . . .	127
Extractives . . . . .	139
Functional Groups and Compound Classes . . . . .	151
Sugars and Other Carbohydrates . . . . .	155

Proteins . . . . .	163
Special Categories of Biomass Materials . . . . .	167
Forage Analysis . . . . .	169
Microalgae . . . . .	173
Oilseeds and Crops . . . . .	175
Peat . . . . .	179
Miscellaneous Analyses of Biomass Materials . . . . .	183
Fuel Product Analyses . . . . .	191
Solid Fuels . . . . .	193
Petroleum-Derived Liquid Fuels . . . . .	207
Lignocellulosic and Oxygenated Liquid Fuels . . . . .	227
Gaseous Fuels . . . . .	239
Non-Fuel Products . . . . .	243
Waste Product Analyses . . . . .	245
Solid Waste . . . . .	247
Liquid Waste . . . . .	251
Atmospheric Analysis . . . . .	259
Conversion System Performance and Specifications . . . . .	269
Combustion . . . . .	271
Gasification . . . . .	279
Bioconversion . . . . .	283
Enzymatic Assays . . . . .	287
Fire and Flammability . . . . .	293
Measurements, Units, Quality Assurance, and Round-Robin Tests	303
Temperature, Pressure, and Flow . . . . .	315
Chromatography . . . . .	323
Spectrometry . . . . .	331
Appendix I: Working Group Members for the IEA Voluntary Standards Activity . . . . .	I-1
Appendix II: Standards Organizations . . . . .	II-1
Appendix III: Methods for Testing Combustion Equipment Energy Efficiency: Finland Report . . . . .	III-1
Appendix IV: Round-Robin Tests . . . . .	IV-1
Appendix V: Reference Biomass Materials . . . . .	V-1
Appendix VI: Other Sources of Information . . . . .	VI-1
Appendix VII: Glossary of Useful Terms . . . . .	VII-1
Index of Standards by Organization . . . . .	Index I-1
Subject Index . . . . .	Index II-1



## Introduction

Earth has a huge diversity of biomass feedstocks, including trees, crops, liquid and solid municipal wastes, oil-producing plants, waste wood, and agricultural residues. Today's options for converting these feedstocks range from direct burning for heat and electricity to chemical, thermochemical, and biochemical processes for making liquid fuels and chemicals. These processes, combined with sustainable management of the biomass resource, can be part of the solution to the world's energy supply and environmental problems.

Scientists and engineers have made great progress from the basic conversion technology available 15 to 20 years ago. Similar advances have taken place in analytical techniques and equipment. There is a growing consensus that establishing standard methods for analyzing biomass and its conversion products would speed research and improve its quality.

Several groups and nations have shown interest in establishing biomass standards. A 1984 workshop organized by the Solar Energy Research Institute (SERI) and supported by the U.S. National Bureau of Standards (NBS, now National Institute of Standards and Technology, NIST), the Pacific Northwest Laboratory (PNL), Oak Ridge National Laboratory (ORNL), the National Research Council of Canada (NRCC), the International Energy Agency (IEA), and the National Science Foundation (NSF) was a major step in discussing the need for standards. The American Society for Testing and Materials (ASTM) has expressed strong interest in continuing to issue biomass standards. Energy, Mines, and Resources (E,M&R) of Canada is pursuing the issue as well by establishing standard materials or feedstocks for conversion research and by coordinating common analyses of pyrolysis oils and enzymatic assays.

In 1986, the International Energy Agency (IEA) began a program to start to address the need for voluntary standards. Sponsored initially by Canada, Finland, New Zealand, and the United States, the program has three tasks: (1) to carry out selected analysis comparisons between laboratories, (2) to establish standard reference materials for both woody and herbaceous plants, and (3) to assemble a sourcebook of relevant analytical methods and standards now being used by laboratories and industry.

The IEA standards activity has benefited from the contributions of many scientists from around the world. An International Advisory Board has provided broad guidance to the program. A Working Group of about 30 scientists was established in 1987 to help plan and conduct specific activities. Advisory Board and Working Group members are listed in Appendix I. Another 250 scientists (correspondents) have received regular information on the project, and many have contributed methods for the sourcebook.

Preparation of the sourcebook has been the major task within the IEA activity. Methods selected were primarily those adopted as voluntary standards by private associations. A number of literature citations are included as well. These represent newer instrumental methods and those relevant to conversion processes such as fermentation and pyrolysis.

Standards specific to biomass energy exist in only a few cases, for example, municipal solid waste and wood fuels. Most of the standard methods come from related industries such as pulp and paper, agriculture, and fossil fuels. In fact, many biomass researchers have adopted these methods as interim standards. However, the applicability to biomass of some of these is limited, as is noted in the literature.

Each section of the sourcebook includes citations of both standard methods and literature methods. The sourcebook lists only citations and abstracts (where available) to save space and abide by copyright limitations. Citations include information on availability. Complete addresses of sources of standards are provided in Appendix II.

Reports on other tasks within the IEA Standards Activity are included in the appendices. The Finland report on small combustor efficiency is Appendix III. Reports on the round-robin tests are included in Appendix IV. Recommendations on selection and pretreatment of standard reference materials are presented in Appendix V.

The last two appendices may also be helpful. Appendix VI lists additional sources of information. Appendix VII presents a glossary of terms related to biomass.

We recognize that the collection of citations in the sourcebook is far from complete and covers predominantly U.S. standards. We hope that those who use the sourcebook will suggest additional methods, comment on limitations of those included, and provide feedback on the usefulness of the publication. The IEA Voluntary Standards Activity will continue through 1991 to allow for conducting more round-robin tests, establishing reference biomass materials, and issuing additional methods for the sourcebook. Please send comments and suggestions for the sourcebook to:

Thomas A. Milne  
Chemical Conversion Research Branch  
Solar Energy Research Institute  
1617 Cole Boulevard  
Golden, CO 80401-3393

### **Key to Standards-Setting Bodies Cited**

<b>AFNOR</b>	Association Francaise de Normalisation
<b>AGA</b>	American Gas Association
<b>AIChE</b>	American Institute of Chemical Engineers
<b>ANSI</b>	American National Standards Institute
<b>AOAC</b>	Association of Official Analytical Chemists
<b>APFI</b>	Association of Pellet Fuel Industries
<b>APHA</b>	American Public Health Association
<b>API</b>	American Petroleum Institute
<b>Appita</b>	Australian and New Zealand Pulp and Paper Industry Technical Association
<b>ASAE</b>	American Society of Agricultural Engineers
<b>ASHRAE</b>	American Society of Heating, Refrigerating and Air-Conditioning Engineers
<b>ASME</b>	American Society of Mechanical Engineers
<b>ASTM</b>	American Society for Testing and Materials
<b>AWWA</b>	American Water Works Association
<b>BSI</b>	British Standards Institution
<b>CBS</b>	Canadian Boiler Society
<b>CFR</b>	Code of Federal Regulations
<b>CGA</b>	Canadian Gas Association
<b>CPPA</b>	Canadian Pulp and Paper Association
<b>CSA</b>	Canadian Standards Association
<b>DIN</b>	Deutsches Institut fur Normung
<b>EN</b>	European Committee for Standardization
<b>EPA</b>	U.S. Environmental Protection Agency
<b>FPL</b>	U.S. Forest Products Laboratory
<b>GOST</b>	USSR State Committee for Standards
<b>ISO</b>	International Standards Organization

<b>IUPAC</b>	International Union of Pure and Applied Chemistry
<b>JIS</b>	Japanese Industrial Standards
<b>NFPA</b>	National Fire Protection Association
<b>SAA</b>	Standards Association of Australia
<b>SANZ</b>	Standards Association of New Zealand
<b>SCAN</b>	Scandinavian Pulp, Paper and Board
<b>SFS</b>	Suomen Standardisoimisliitto
<b>TAPPI</b>	Technical Association of the Pulp and Paper Industry
<b>UL</b>	Underwriters' Laboratories, Inc.
<b>ULC</b>	Underwriters' Laboratories of Canada











## FEEDSTOCK SAMPLING AND PREPARATION (STANDARDS)

**Title:** Wood: General Requirements for Tests; Physical and Mechanical Tests

**Citation:** NF B 51-003-85

**Content:**

**Availability:** AFNOR

---

**Title:** Sampling of Peat

**Citation:** AOAC 2.198

**Content:** For moss, humus, and reed-sedge types.

**Availability:** AOAC

---

**Title:** Preparation of Peat Sample

**Citation:** AOAC 2.199

**Content:** Place representative field sample on square rubber sheet, paper, or plastic. Reduce sample to amount required by quartering and place in moisture-proof container. Work rapidly to prevent moisture losses.

**Availability:** AOAC

---

**Title:** Sampling of Plants

**Citation:** AOAC 3.001

**Content:** When more than one plant is sampled, include enough plants in sample to ensure that it adequately represents average composition of entire lot of plants sampled. (This number depends upon variability in composition of the plants.) Determine details of sampling by purpose for which sample is taken.

**Availability:** AOAC

---

**Title:** Preparation of Plant Sample

**Citation:** AOAC 3.002

**Content:** For mineral constituents and carbohydrates.

**Availability:** AOAC

---

**Title:** Sampling of Animal Feed: Procedure

**Citation:** AOAC 7.001

**Content:**

**Availability:** AOAC

---

**Title:** Methods of Test for Pulp and Paper (metric units); Preparation of Wood Samples for Chemical Analysis

**Citation:** AS 1301, P2m:1973 and Appita P2m-73

**Content:** This standard prescribes the procedure for reducing wood samples to a suitable state of subdivision for chemical analysis.

**Availability:** Appita, SAA

---

**Title:** Methods of Test for Pulp and Paper (metric units); Sampling Paper and Board for Testing

**Citation:** AS 1301, P417m:1973 and Appita P417m-73

**Content:** Specifies a method of obtaining a representative sample of a lot of paper for test purposes.

**Availability:** Appita, SAA

---

**Title:** Standard Method of Collection and Preparation of Coke Samples for Laboratory Analysis<sup>1</sup>

**Citation:** ASTM D 346-78

**Content:** Coke, especially run of oven coke, or foundry coke, or both, is a difficult