IEA VOLUNTARY STANDARDS ACTIVITY

METHODS OF ANALYSIS OF BIOMASS FOR FUELS AND CHEMICALS

1987 ANNUAL REPORT TO IEA JANUARY 31, 1988

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PREFACE

This report covers the highlights of 1987 activities under the three-year IEA Bioenergy Agreement, Biomass Conversion Activity on Voluntary Standards. Additional details are available in quarterly progress reports submitted to the Renewable Energy Branch of Energy, Mines and Resources Canada, one of the major in-kind supporters. In-kind support is also received from New Zealand, Finland and the United States.

Thomas A. Milne Activity Leader

Approved for Solar Energy Research Institute

Helena L. Chum, Manager

Mchum

Chemical Conversion Research Branch

A. Introduction

There is a growing concensus regarding the need for a whole range of standards activities in support of research, development, and commercialization of biomass for energy and chemicals. Key among these is an informed collection of analytical methods for the whole conversion cycle of biomass to fuels and energy-intensive products. These methods would be especially useful in the context of relating desirable feedstock characteristics (both chemical and physical) to the range of conversion options that are the subject of current research.

The objective of this work is to collect, select, test, and disseminate key analytical methods relevant to the thermochemical and biological conversion of biomass to fuels and chemicals, and to characterizing feedstocks. Work jointly supported by Canada, Finland, New Zealand, and the United States is described.

B. Solicitation of on Needs and Methods

Starting in the summer of 1986, response forms were distributed at several major gatherings of bioconversion scientists and technologists. These were:

- 1. Graz, Austria. A major IEA international symposium on the pretreatment of lignocellulosics was held in June 1986. One of the session topics was on analytical methods. This meeting was co-sponsored by the C-5 sugars group, also of the IEA.
- 2. Vancouver, Canada. The Sixth Canadian Bioenergy R&D seminar, held February 16-19, 1987, was attended by T. Milne and H. Chum.
 - A poster on the IEA Standards Project was presented. In two closed sessions after the R&D seminar, Milne presented an outline of the project and Chum led discussions from the floor on methods of interest. There was much interest and lively discussion of analytical needs resulting in suggestions for priority methods.
- 3. Denver, Colorado. In connection with the National American Chemical Society Meeting, April 5-10, 1987, a major international symposium on the "Production, Characterization, and Upgrading of Pyrolysis Oils from Biomass" was held which attracted some 60 participants. Included in the symposium was a session on standards, at which the IEA program was presented and discussed in panel format.
- 4. Boulder, Colorado. A poster paper was given on the IEA program at the Ninth Symposium on Biotechnology for Fuels and Chemicals, May 5-8, 1987. Response forms were distributed at this meeting, and many new contacts made.
- 5. Grenoble, France. The seminar on Analytical Studies of Wood and Wood Components May 4-5, 1987, organized by D. Robert (Centre d'Etudes Nucleaires de Grenoble) attracted approximately 50

participants from Japan, New Zealand, Australia, France, Finland, Sweden, Canada and the USA. Discussions on methods of analyses of woods and their components were held. Chum had the opportunity to introduce the IEA program.

As a result of these contacts and the response from mailings to some 350 researchers, we are now distributing quarterly reports to 204 correspondents who have expressed an interest in active participation in this activity. The quarterly reports will be used to make specific inquiries about methods or sources of methods.

A special stationery is being used for project correspondence to emphasize the international and IEA focus. (See example in Attachment I.)

C. Working Group

A crucial element in the success of this program is to receive constant and detailed advice from an international group of experts having broad knowledge of the analytical methods in biomass for fuels and chemicals. They will help focus on most needed methods, will provide references to methods in use, and will suggest the most needed round-robin tests. The group members are listed in Attachment I.

Twenty-two of the twenty-nine members met in Denver on November 18-20, 1987, to discuss and guide the program. (Three of the absent members sent representatives.) The program for the meeting is given in Attachment II. Three panels met concurrently and then reported their deliberations to the entire group. The panel leaders and recorders were as follows:

Feedstocks panel

Leader - W. E. Hillis
Recorder - R. Sutcliffe
Leader - K. Mackie
Recorder - M. Paice

Thermochemical panel Leader - R. Overend Recorder - D. Elliott

Each panel has now submitted a report on their deliberations and recommendations and the results are being synthesized to guide the round-robin tests and the form and content of the "Methods Manual". The panels expressed enthusiasm about continuing to interact, and identified volunteers for round-robin tests and opportunities for future face-to-face interactions.

D. Round-Robin Tests

Some 76 laboratories have indicated potential interest in participating in round-robin tests of particularly important analytical methods. The Working Group gave major emphasis to selecting initial candidates for these tests. The Feedstocks

Panel recommended a round-robin on both woody and herbaceous species based on a refinement of a procedure recently published by O. Theander. It is hoped that the Oak Ridge National Laboratory/DOE production program can help supply samples through the help of J. Ranney and J. Cushman. This round-robin will be coordinated by H. Chum and T. Milne at SERI.

The Thermochemical Panel will initiate the first of several needed round-robin tests of properties of pyrolysis/liquefaction oils by distributing one or more oils to laboratories for measurement of water content. This round-robin will be led by R. Overend of NRCC. The Biochemical Panel has planned a test of a variety of commercial cellulases using a standard cellulase assay prepared by H. Esterbauer. These tests will be coordinated by K. Grohmann of SERI and H. Esterbauer of the University of Graz.

E. Handbook of Analytical Methods

A major physical product of this IEA activity will be a collection of useful analytical methods. These will not be standard methods in the sense used by ASTM, TAPPI, CSA, etc., nor will they bear the endorsement of the participating countries or agencies. We hope they will be a valuable reference collection for researchers producing or utilizing biomass for fuels and chemicals. The collection will be issued by SERI's Solar Technical Information Program in 1989. (Contact is P. Notari at SERI)

Using an arbitrary classification, the many methods collected for this activity, and supplied by correspondents, were compiled into 17 loose-leaf notebooks for reference use by the Working Group at the Denver meeting. A draft compilation will be started, following the guidance of the Working Group and sponsors.

The Working Group favored an easily updatable, loose-leaf notebook structured by method rather than substrate. We should concentrate on woody and herbaceous materials, with a major interest in agricultural residues and peat. MSW and other lignocellulosics will be emphasized later. The handbook, or methods manual, should contain titles and abstracts of published standards, world-wide, and an annotated bibliography of literature methods and collections of methods. A glossary should also be added.

F. <u>Special Project by Finland--Methods for Testing Efficiency of</u> Heating Systems Based on Direct Combustion or Gasification

This project is proceeding under the direction of H. Oravainen, with emphasis on collecting methods for measuring efficiencies of biomass combustion systems (fireplaces, single-house boilers, boilers for large houses, district heating plants, and fuel gas producers). Emissions are being de-emphasized in view of the related IEA activity on combustion emissions (Christel Benestad, Norway).

A library search has been carried out on efficiency testing methods, with several useful German (DIN) standards identified for large and small boilers and for fireplaces.

Heikki Oravainen, Senior Research Scientist with the Technical Research Centre of Finland, would welcome sources of information on relevant methods. Attachment III reproduces the questionnaire the Domestic Fuel Laboratory is sending out to 39 laboratories worldwide.

G. Publications and Presentations - 1986-87

Additional details can be found in the quarterly reports listed in Attachment IV. Past and planned presentations related to this activity are also listed.

H. Plans for 1988

In 1988 the emphasis will be on coordination of round-robin tests and the compilation and layout of the first edition of the methods manual or handbook.

A camera-ready manuscript of the methods is due in early 1989.

Interactions with the working group, sponsors and researchers will resume at the May IEA Thermochemical meeting in Phoenix, at the May Biotechnology Meeting in Gatlinburg, and at the June IEA Bioconversion Meeting in Ottawa.

Methods of Analysis of Biomass for Fuels and Chemicals

Advisory Board

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Keith Mackie Forest Research Institute Private Bag Rotorua, New Zealand (64) 73 475899 IEA Bioenergy Agreement — Biomass Conversion Annex IV — Voluntary Standards Activity Member Countries: Canada, Finland, New Zealand, United States

ATTACHMENT I

WORKING GROUP MEMBERS — 1987 FEEDSTOCKS PANEL

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Dr. Kai Sipila Tech. Research Centre of Finland Laboratory of Fuel Processing SF-02150 ESPOO FINLAND

AGENDA METHODS OF ANALYSIS OF BIOMASS FOR FUELS AND CHEMICALS

IEA WORKING GROUP MEETING

Sheraton Hotel, Lakewood, Colorado November 18-20, 1987

WEDNESDAY, NOVEMBER 18

8:00 - 8:30 a.m.	Registration/Coffee and rolls Red Rocks	
INTRODUCTION		
8:30 - 8:35 a.m.	Meeting Opens - Mt. Vernon	T. Milne/SERI
8:35 - 8:45 a.m.	SERI Welcome	S. Bull/SERI
8:45 - 9:00 a.m.	IEA Welcome and Remarks	D. Stevens/Battelle
9:00 - 9:15 a.m.	E, M & R Welcome and Remarks	D. Hayes/Energy Mines & Resources
9:15 - 9:25 a.m.	SERI STIP Program	P. Notari/SERI
9:25 - 9:40 a.m.	Charge to the Working Group, Introductions	T. Milne/SERI
BACKGROUND 9:40 - 9:55 a.m.	Finland's Special Task	H. Oravainen/ Technical Research Centre of Finland
9:55 - 10:15 a.m.	BREAK - Red Rocks	
10:15 - 10:45 a.m.	CPD Experiences and Future Activities	K. Mackie/Forest Research Institute
10:45 - 11:05 a.m.	Round Robin Testing	E. Domalski/National Bureau of Standards
11:05 - 11:30 a.m.	Perils and Pitfalls in Synfuels Characterization	G. Gibbon/ Pittsburgh Energy Technology Center
11:30 a.m 12:00 p.m.	Status of Methods and their Availability, Logistics	T. Milne/SERI
12:00 - 1:30 p.m.	LUNCH ON YOUR OWN	

1:30 - 5:00 p.m. PANELS: Feedstocks Panel - Red Rocks Biochemical Panel - Mt. Vernon Thermochemical Panel - Boardroom (Panels will meet separately through Thursday - joint coffee breaks in the Mt. Vernon) BREAK - Mt. Vernon 3:30 p.m. 6:00 - 8:00 p.m. Cash Bar Reception Lookout Mtn. (Supper by your own arrangements) THURSDAY, NOVEMBER 19 8:00 - 8:30 a.m. Coffee and rolls - Mt. Vernon Panels meet on own schedule 10:30 a.m. Coffee Break - Mt. Vernon 3:00 p.m. Coffee Break - Mt. Vernon FRIDAY, NOVEMBER 20 Coffee and rolls 8:00 - 8:30 a.m Red Rocks

8:00 - 8:30 a.m Coffee and rolls
Red Rocks

8:30 - 10:00 a.m. Reports from Panels - Mt. Vernon

10:00 - 10:30 a.m. BREAK - Red Rocks

10:30 a.m. - Discussion of Panel Reports
12:00 p.m. Future Activities of Working
Group/Panels

12:00 p.m. Adjourn

12:00 - 1:30 p.m. Panel Chairpersons meet for debriefing with Project Leaders Mt. Vernon

Mt. VCI HOII

1:30 - 5:00 p.m. Tours of SERI and meetings with SERI Researchers by appointment

TECHNICAL RESEARCH CENTRE OF FINLAND Domestic Fuel Laboratory Heikki Oravainen

14,12,1987

Dear Sir

In the International Energy Agency(IEA) biomass conversion program, Annex IV, task 7(Methods for testing efficiency and emissions of biomass combustion systems) we are collecting and evaluating the available standard methods for testing efficiency of biomass combustion systems. The methods concern fireplaces, single house boilers, boilers for large houses, district heating plants and fuel-gas producers.

We are now asking if You please could describe the methods You are using concerning this subject. Also if You could comment on the useability of standard methods used for biofuels. We would like to have some calculation examples for comparison.

We would also be very pleased to have contact addresses of persons who You know are working with calculation of efficiencies.

Thanking You in advance.

Sincerely Yours,

Heikki Oravainen

senior research scientist
MSc(Tech)

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ATTACHMENT IV

Reports and Presentations in 1987

Reports

"First Quarterly Progress Report - Methods of Analysis of Biomass for Fuels and Chemicals", February 16-May 15, 1987. Prepared for Renewable Energy Branch, Energy, Mines and Resources of Canada, Ottawa, Canada, DSS Contract File No.: 23216-6-6183, May 31, 1987.

"Second Quarterly Progress Report", ibid. May 16-Aug. 15, 1987.

"Third Quarterly Progress Report", ibid. Aug. 16-Nov. 15, 1987.

November and December SERI Monthly Reports to DOE cover the last portion of 1987.

Presentations

- Sixth Canadian Bioenergy R & D Seminar, Feb. 16-1987, Vancouver, B.C., Poster paper and Group Discussion on Biochemical and Thermochemical Conversion analytical needs. (H. Chum & T. Milne)
- Denver National ACS Meeting, April 5-10, 1987, Denver, Colorado.
 Panel discussion as part of Symposium on Production
 Characterization and Upgrading of Pyrolysis Oils from Biomass.
 (T. Milne and Attendees)
- Ninth Symposium on Biotechnology for Fuels and Chemicals, May 5-8, 1987, Boulder, Colorado.

 Poster paper on Voluntary Standards activity. (T. Milne)
- Seminar on Analytical Study of Wood and Wood Components, May 4-5, 1987. Grenoble, France.

IEA program introduced by H. Chum, followed by discussion.