IMEKO XIX World Congress
Lisbon, September 9th, 2009.

Ministério do Desenvolvimento, Indústria e Comércio Exterior

BRASIL UM PAÍS DE TODOS GOVERNO FEDERAL

Metrology Challenges for Biofuels

João Jornada
President of Inmetro- Brazil

Major world concerns today:

Environment

Energy

Social&Political issues

G8+5 Academies' joint statement: Climate change and the transformation of energy technologies for a low carbon future

Climate change and sustainable energy supply are crucial challenges for the future of humanity. It is essential that world leaders agree on the emission reductions needed to combat negative consequences of anthropogenic climate change at the UNFCCC negotiations in Copenhagen in December 2009. At the same time, agreement is needed on actions to ensure basic energy services are available to all of the world's people.

These global challenges require solutions flexible and varied enough to meet the needs of a wide variety of specific energy resources and energy security circumstances.

Reducing the human forcing of

Critical research areas include: increasing the resilience of urban and rural infrastructure and of natural areas (including watersheds and coastal areas); enhancing food and crop production; and water conservation technologies and methods.

The energy agenda

Fossil fuel sources remain the predominant energy source for the near future in reducing energy poverty and satisfying growing energy demand, and their exploitation must be consistent with the objective of reducing anthropogenic impact on climate change. Continuous improvement in efficiency and emission standards are needed in the production and use of fossil fuels.

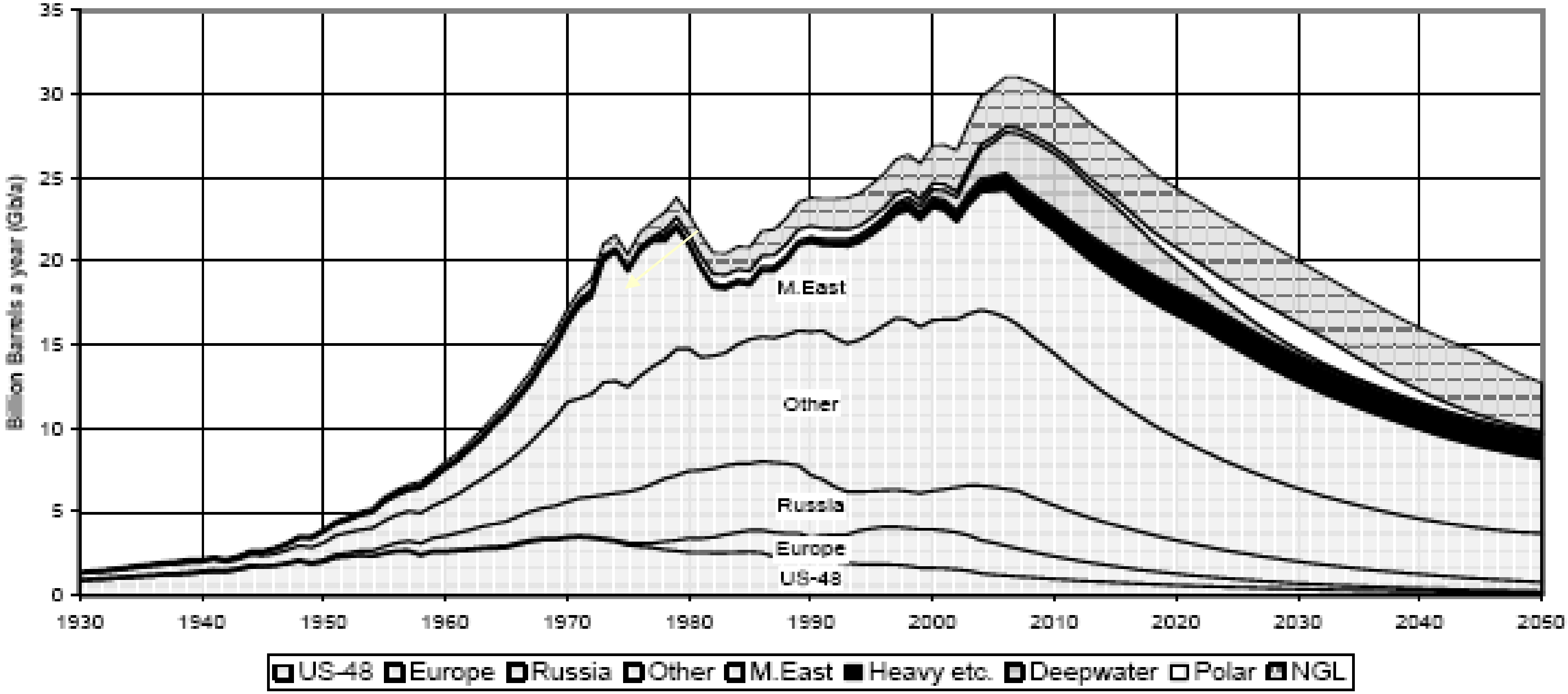
Economically viable low carbon energy technologies may

- rapidly increased adoption of, and investment in, renewable energy technologies such as wind, geothermal, solar energy, biofuels and wave power. The development of standards and certification for the environmentally sustainable implementation of these technologies is essential;

- rapid and wide-spread energy conservation measures particularly for industry, transport, and building design, construction and operation. This will require the development and implementation of existing and new technologies, policy tools, monitoring and certification processes, and public education. Energy saving and energy efficiency should be a critical priority in the short term;

- collaborate in the implementation of low carbon and climate-resilient infrastructure and technologies, and in the implementation of innovative incentives, through the use of economic and regulatory instruments, to accelerate adoption of clean "green" technologies;

Scarcity and Fossil Fuel Cost



Graph of Oil Production
Source: Colin Campbell of the Association for the Study of Peak Oil and Gas (ASPO) Newsletter as in Wikapedia http://en.wikipedia.org/wiki/Peak_oil

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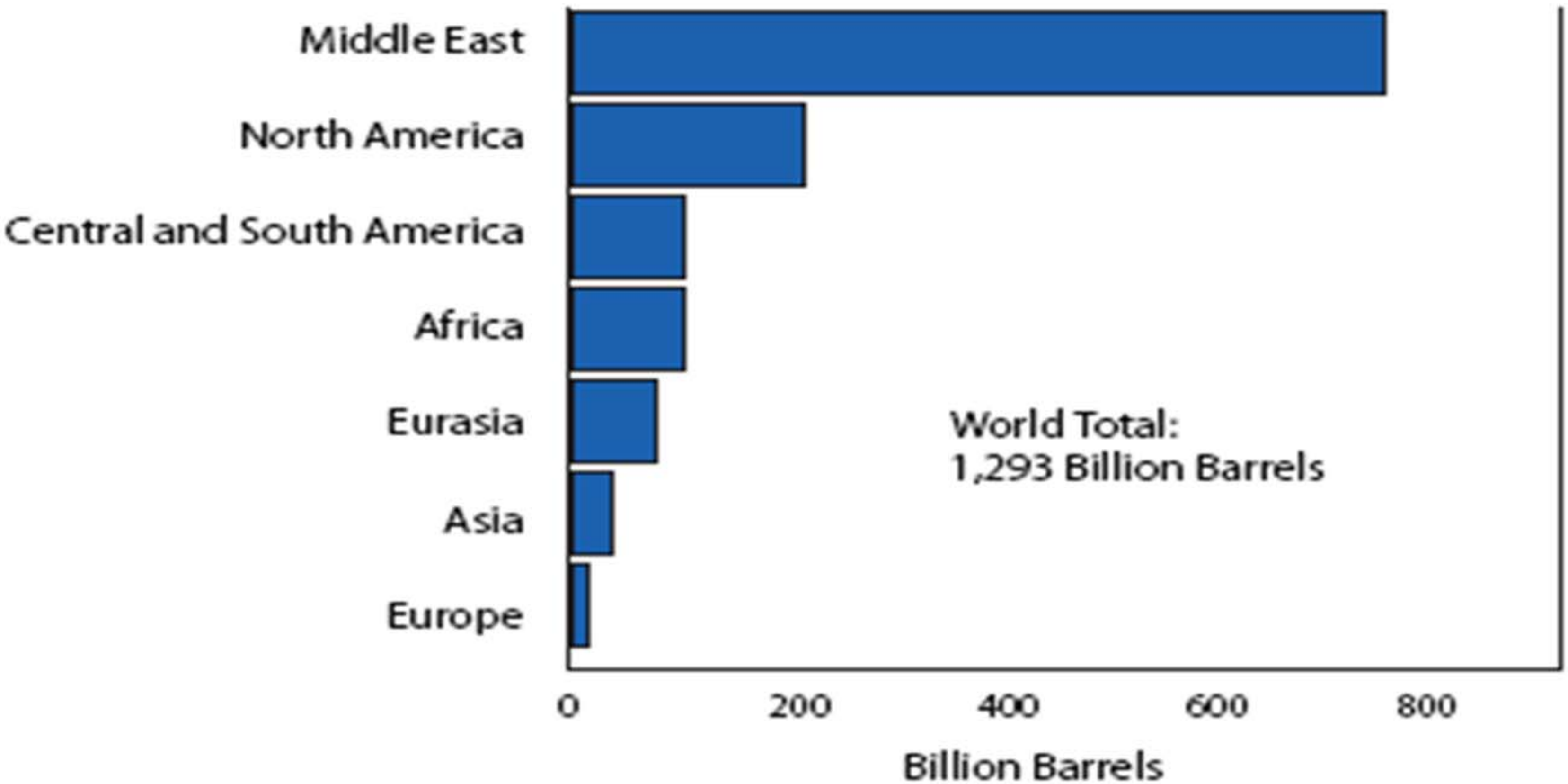
Chart 1.1g: World Proved Oil Reserves



Source: "Worldwide Look at Reserves and Production," *Oil & Gas Journal*, Vol. 103, No. 47 (December 19, 2005), pp. 24-25.

Sustainable development:

the development that meets the needs of the present without compromising the ability of future generations to meet their own needs

("Our Common Future" –Gro Harlem Brundtland - Brundtland Report 1987)

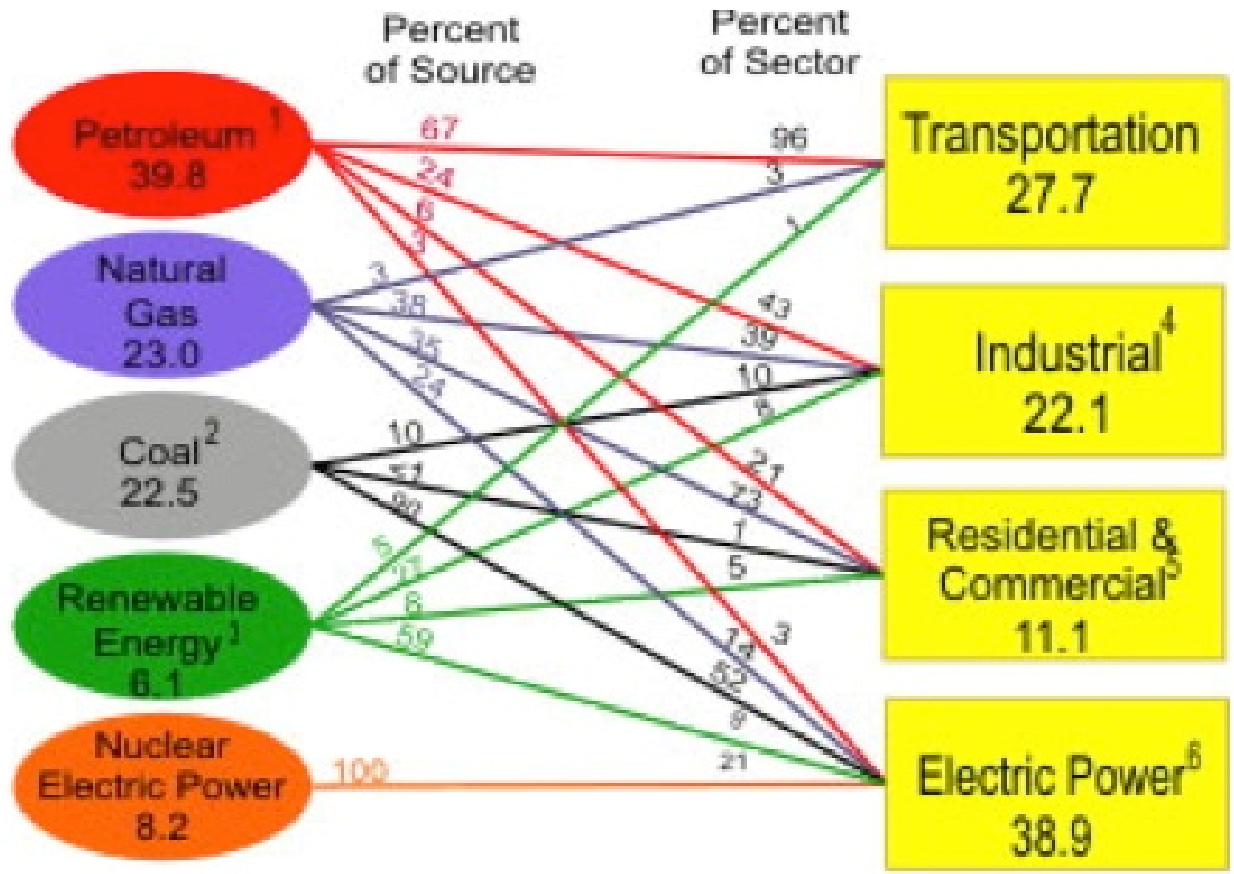
Biofuels

Biofuel is any fuel obtained from a biomass material(material of biological origin that is non fossil), as ethanol, biodiesel, butanol, methanol, biogas etc..

The importance of Biofuels in helping to overcome those concerns:

- *Environment- reducing the threat of climate change*
- *Energy- reducing dependence on oil.*
- *Social&Political issues– supply is not concentrated in few places; reducing poverty in some countries*

Consumo Primário de Energia nos EUA



FONTE: US ENERGY INFORMATION ADMINISTRATION

In response to this challenge, several countries and international organisations launched profound and far reaching programs in renewable energy and biofuels

EU Policy (Thomsen Report of September 2007- binding, 2008):

Targets for 2020:

- *20-30% GHG reduction*
- *20% energy efficiency improvement*
- *20% renewable energy including 10% biofuels*

Energy Independence & Security Act 2007

Type of Fuel	BGY
Total Renewable Fuels by 2022	**36 BGY**
Corn Ethanol (Capped at 15 BGY)	**15 BGY**
Advanced Biofuels – Includes imported biofuels and biodiesel. Includes 1 billion gpy biodiesel starting in 2009 All must achieve $\geq$ 50% reduction of GHG emissions from baseline*	**21**
Cellulosic Fuels – Includes cellulosic ethanol, biobutanol, green diesel, green gasoline All must achieve $\geq$60% reduction of GHG emissions from baseline*	**16**

*Baseline = average lifecycle GHG emissions as determined by EPA Administrator for gasoline or diesel (whichever is being replaced by the renewable fuel) sold or distributed as transportation fuel in 2005

Source- USA EPA- 2008- Hecht

Brazil- First Results for a 100% Ethanol-Powered Vehicle: 1979

First Results for a 100% Ethanol Vehicle: 1979

1979: Manufacturers begin to sell 100% Ethanol Vehicle

TE FIAT A ÁLCOOL

e atinge a velocidade máxima de 142 km/hora

Como anda um carro movico ape-
a álcool? Para responder a essa
gunta, **Quatro Rodas** testou com
usividade um Fiat a álcool produ-
na fábrica de Betim, Minas Ge-
, e já na sua versão praticamente
nitiva.
:, entre os resultados alcançados
teste, destacam-se o desempenho
to bom, superior ao dos modelos
série (apenas o esportivo Rallye
, em alguns pontos, marcas um
co superiores, mas, no conjunto,
de para o modelo a álcool), o con-
no bem mais elevado e a ótima di-
bilidade.

Com a instalação da quinta roda, medições exatas.

April, 1979: Publication in a technical magazine of the first test-drive of a 100% ethanol vehicle

Source: Brazilian Ministry of Mines and Energy

Some distinguished features of the biofuel issue:

1- Very large economic, social and environmental impact.

2- Rapid paradigm shift: need fast implementation to a whole new set of commodities, for worldwide use; change in production and commerce networks.

3- Imply new international and national frameworks strongly tied by formal constrains and regulations.

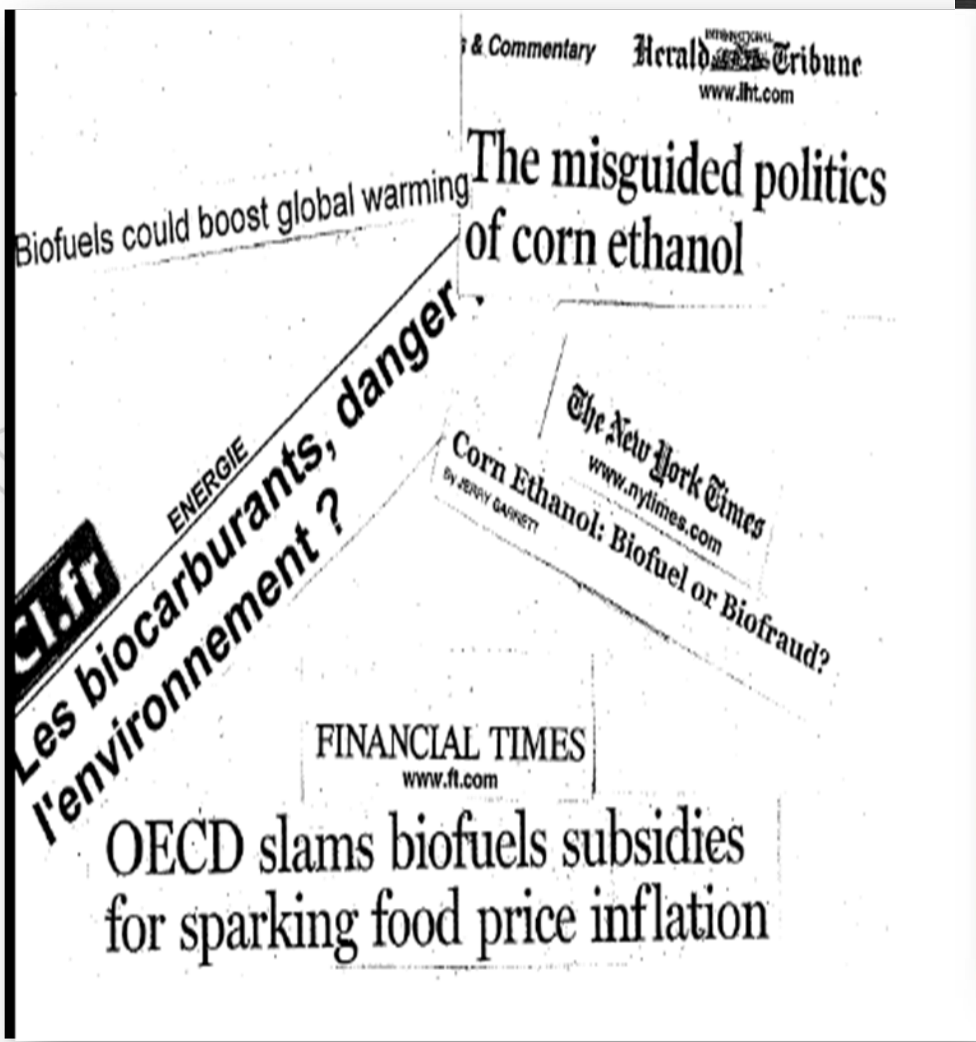
4- Politically sensitive subject- conflicting interests and disputed "truths"

5- Demand for uniformity and quality for measurements in a whole new set of situations, to underpin this paradigm shift. Confidence in measurements must arise from formal and well structured systems.

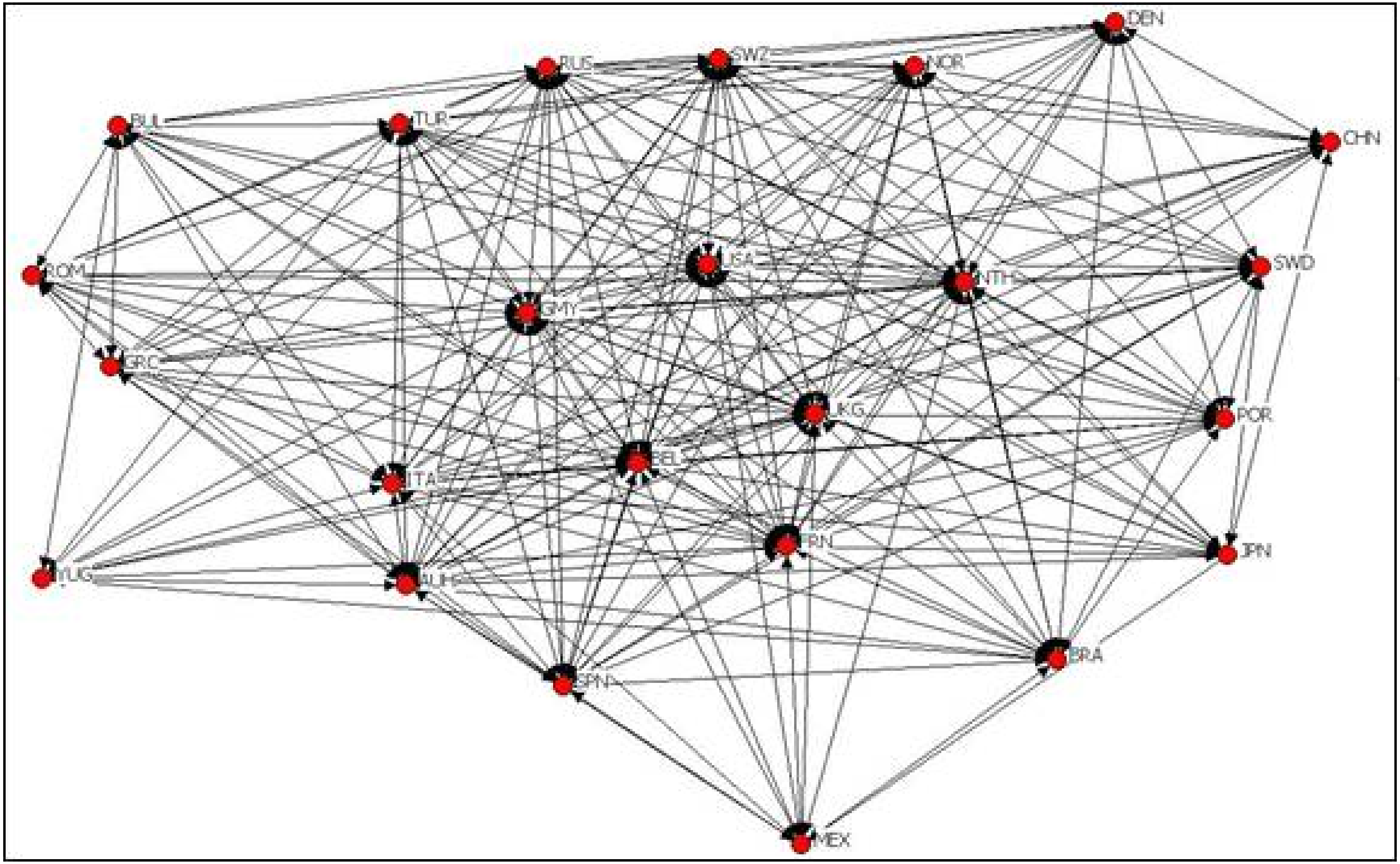
6- Deep knowledge of measurement science to support regulations and standardization needs, as well as new production processes.

7- Disputed opinions: importance, how quick, in what extent, for how long

Biofuels: is the cure worse than the disease?



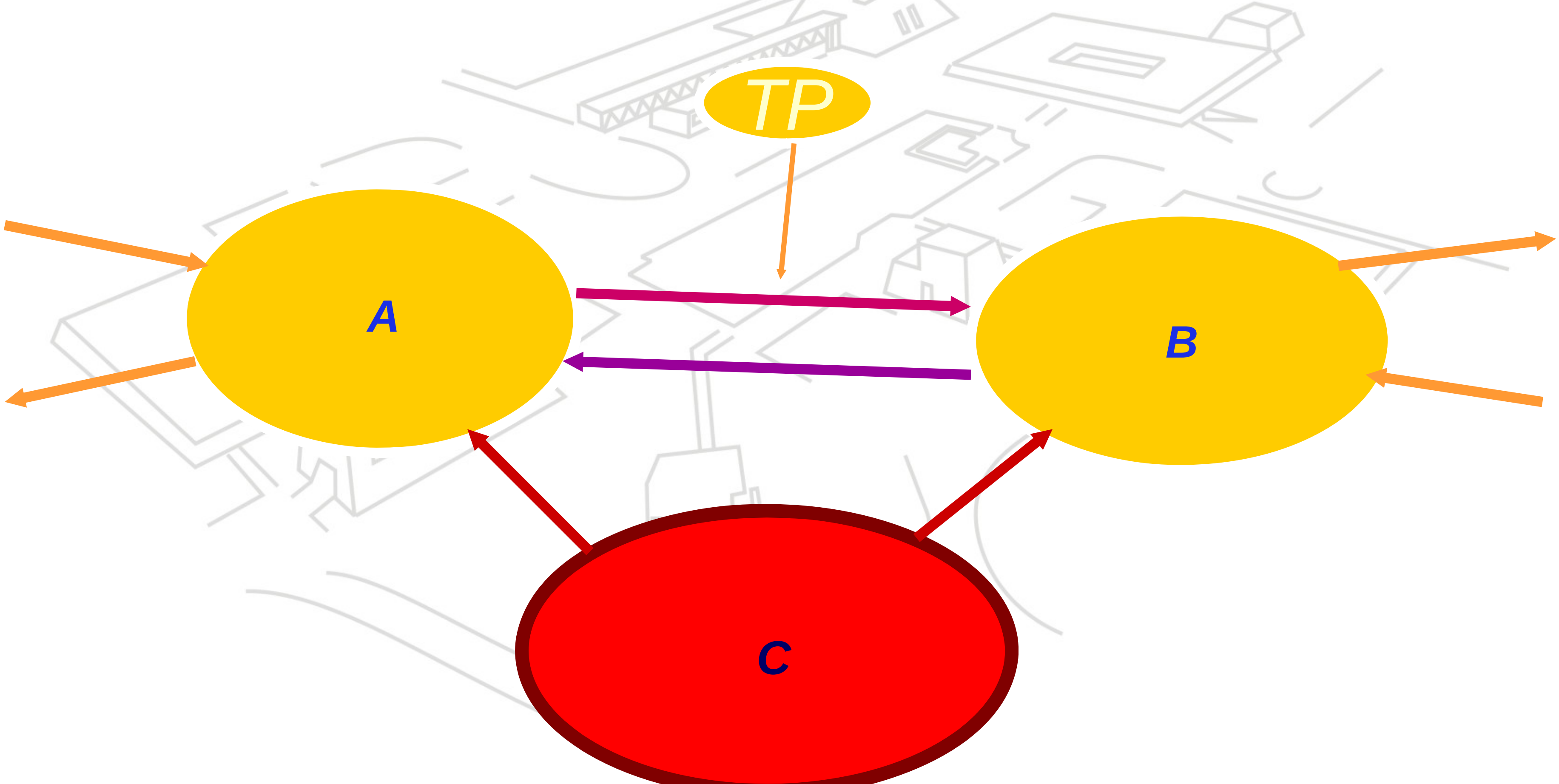
A difficult problem: rapid change in a very complex network of interdependent economical and technical players working coherently



Source: Institute for Research in Worlsd-Systems

KEY ENABLERS- INFRASTRUCTURE TECHNOLOGIES

METROLOGY, CONFORMITY ASSESSMENT, STANDARDIZATION AND TECHNICAL REGULATION



Biofuels and Metrology

- *Structure- Excellent international networking; infrastructure; institutional recognition;*
- *Focus- Formal demonstration of confidence in measurements;*
- *Knowledge- Deep scientific knowledge about measurements; set of proven practices- calibration, traceability demonstration, PT, CRM,...*

Basic needs for Metrology:

1- Production

2- Storage, transportatation and distribution

3- End-use- effects on engines environment an health

4- Regulation and Standardization

5- Quality and Conformity Assessment

6- Accreditation

7- Sincronization; harmonization of myriad of players

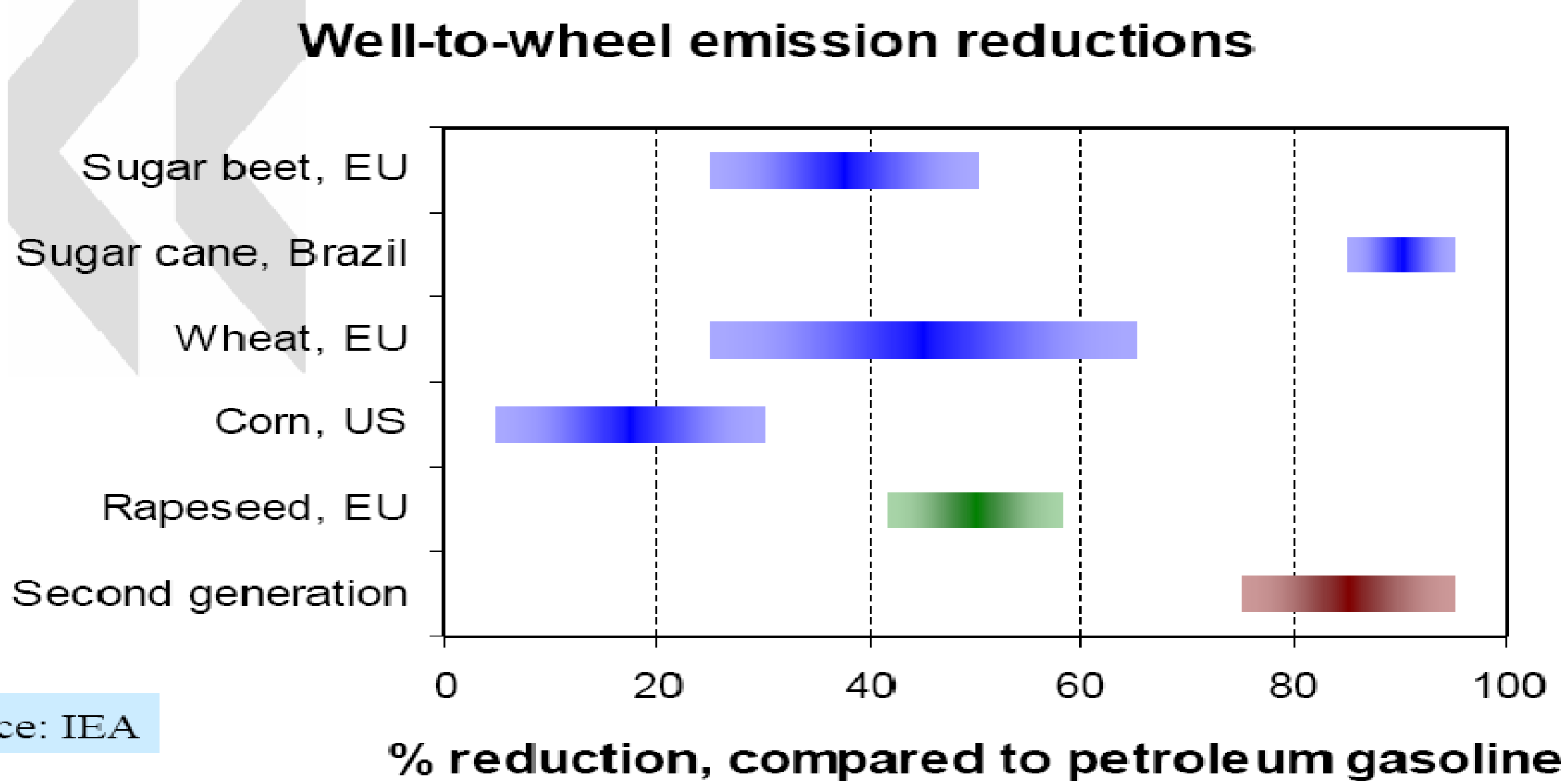
8- Commerce: International Articulation

9- Innovation, R&D

10- Biofuels as starting raw material for green chemistry

The concern with environment and the need for reliable Life Cycle Analysis-LCA

Climate Change mitigation potential



ource: IEA

Some specific needs:

1- Information about the origin, both geographical and related to raw materials; fundamental do comply with certain regulations.

2- CRM development and validation. Stability is a big issue, especially for biodiesel.

3- P.T. and training.

4- New instrumentation development aiming at reliability, simplicity, portability,

5- Deeper scientific and technological understanding to support: R&D, new instruments, standards, regulations and CA.

6- Measurements related to side effect of biofuels use: corrosion of engine parts, environmental and health effects.

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INMETRO 35 ANOS

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Measurement and Standards for Biofuels: Enabling a Transition from Petroleum as a Vehicular Energy Source

INMETRO

Time	Speak
8h30min	João Jornada
8h45min	Willie May
9h15min	João Jornada
9h45min	Curtis Williams
10h30min	José Felix Silva Jú
11h	Coffee Break
11h30min	Discussion
12h30min	Lunch
14h	Robert Goldberg
14h30min	Expedito José de Sá Pa
15h	Expedito José de Sá Pa
15h30min	Paulo Anselmo Vian
15h30min	Coffee Break
16h – 17h	Discussions

Time	Speak
9h	Luciano Almeida
9h30min	Panel
10h15min	Coffee Break
10h30min	Panel
12h	Lunch
14h	

Symposium on Biofuels

Measurements and Standards to Facilitate the Transition to a Global Commodity

Hosted by:
the US National Institute of Standards and Technology (NIST)
and
Brazil's National Institute of Metrology, Standardization and Industrial Quality (INMETRO)

in conjunction with the
11th Annual Green Chemistry & Engineering Conference:
From Small Steps to Giant Leaps – Breakthrough Innovations for Sustainability
Capital Hilton in Washington, DC
June 26-29, 2007

GREEN CHEMISTRY

WHITE PAPER ON INTERNATIONALLY COMPATIBLE BIOFUEL STANDARDS

TRIPARTITE TASK FORCE

BRAZIL, EUROPEAN UNION & UNITED STATES OF AMERICA

DECEMBER 31 2007

(Criação conjunta do GT Brasil-EUA-CE para harmonização de especificações para biocombustíveis)

Classification of Bioethanol Specifications

Category A ***similar***	**Category B** ***significant differences***	**Category C** ***fundamental differences***
color	ethanol content	water content*
appearance	acidity	
density	phosphorus content	
sulfate content	pHe	
sulfur content	gum / evaporation residue	
copper content	chloride content	
iron content		
sodium content		
electrolytic conductivity		

*** This topic is under discussion. While the differences in water level allowed does not prevent ethanol trade, there are costs associated with additional drying**

Classification of Biodiesel Specifications

Category A *similar*	Category B *significant differences*	Category C *fundamental differences*
sulfated ash	total glycerol content	sulfur content
alkali and alkaline earth metal content	phosphorus content	cold climate operability
free glycerol content	carbon residue	cetane number
copper strip corrosion	ester content	oxidation stability
methanol and ethanol content	distillation temperature	mono, di-, tri-acylglycerides
acid number	flash point	density
	total contamination	kinematic viscosity
	water content and sediment	iodine number
		linolenic acid content
		polyunsaturated methyl ester

INMETRO BIOFUELS PROGRAM- AIMING AT PRODUCTS, PRODUCTION, USES AND IMPACTS:

- *Conformity Assessment activities, including Certification schemes and Accreditation of labs and certification bodies*
- *LCA for GHG savings and related issues*
- *Proficiency Testings and Training*
- *Analytical methods and Procedures*
- *Support to Industry and Government*
- *Support to regulation*
- *S&T knowledge; R&D*
- *International cooperation*
- *Measurements Standards- CRM*

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Parameters for Bioethanol to be included in MRC

As presented in the

White Paper

Density
Sulfate content
Sulfur content
Copper content
Iron content
Sodium content
Ethanol content
Acidity
Phosphorus content
pHe
Chloride content
Water content

Production of high-quality measurement standards

Inmetro and NIST joint program for biofuels CRMs- ethanol and soy and animal fat biodiesel

Parameters and certified values of CRM of Anhydrous Bioethanol Fuel.

Parameters	Certified Value*	Unit
Total acidity	(20.5 ± 3.0)	mg/L
Electrolytic Conductivity (electrical) at 25.0 ° C	(1.71 ± 0.33)	µS/cm
Density at 20.0 ° C	(0.79045 ± 0.00012)	g/cm^3
pH at 25.0 ° C	(7.07 ± 0.33)	---
Sulphate	(0.364 ± 0.068)	mg/kg
Water content	(0.3648 ± 0.0059)	% (mass fraction)
Ethanol content	(99.57 ± 0.07)	% (mass fraction)

Expanded uncertainty (k=2, confidence level of approximately 95%).

Parameters and certified values of CRM of Soy-based biodiesel.

Parameters	Certified Value*	Unit	Observation
Hexadecanoic Acid, Methyl Ester (C16:0) (Palmitic Acid, Methyl Ester)	(1.71 ± 0.33)	g/kg	Certified Values
(Z)-9-Hexadecenoic Acid, Methyl Ester (C16:1 n-7) (Palmitoleic Acid, Methyl Ester)	(1.32 ± 0.18)		
(Z)-9-Octadecenoic Acid, Methyl Ester (C18:1 n-9) (Oleic Acid, Methyl Ester)	(233 ± 6)		
(Z,Z)-9,12-Octadecadienoic Acid, Methyl Ester (C18:2 n-6) Linoleic Acid, Methyl Ester)	(770 ± 17)		
(Z,Z,Z)-9,12,15-Octadecatrienoic, Methyl Ester (C18:3 n-3) (Linolenic Acid, Methyl Ester)	(204 ± 3)		
Water	(0.018 ± 0.002)	% (mass fraction)	
Density at 20 ° C	(0.88132 ± 0.00006)	g/cm^3	
Kinematic Viscosity at 40 ° C	(4.0843 ± 0057)	mm^2/s	

Expanded uncertainty (*k*=2, confidence level of approximately 95%).

Parameters and certified values of CRM of Soy-based biodiesel.

Parameters	Certified Value*	Unit	Observation
Pentadecanoic Acid, Methyl Ester (C15:0)	(0.104 ± 0.008)	g/kg	Reference values
Heptadecanoic Acid, Methyl Ester (C17:0)	(1.03 ± 0.02)		
Docosanoic Acid, Methyl Ester (C22:0) (Behenic Acid, Methyl Ester)	(3.7 ± 1.1)		
Acid number	(0.180 ± 0.007)	mg/g KOH	
Oxidation Stability of Fatty Acid Methylesters at 110 ° C	(4.41 ± 0.27)	g/cm³	
Gross Heating Value	(9465 ± 3)	cal/g	
Methanol	(587 ± 44)	mg/kg	
Free Glycerin	(164 ± 16)		
Monopalmitin	(29.7 ± 2.3)		
Monolein, Monolinolein, and Monolinolenin	(1994 ± 98)		
Diolein and Diolinolein	(707 ± 31)		
Triolein	(241 ± 17)		
Density at 25 ° C	(0.8776 ±0.0001)	g/cm³	
Kinematic Viscosity at 25 ° C	(5.683 ± 0.011)	mm²/s	
Speed of Sound at 25 ° C	(1467.7 ± 0.1)	m/s	

*Expanded uncertainty (k=2, confidence level of approximately 95%).

Parameters and certified values of CRM of Soy-based biodiesel.

Parameters	Certified Value*	Unit	Observation
Sulfur	<0.5	mg/kg	Information Values
Sodium	0.07		
Potassium	<0.1		
Calcium	0.5		
Magnesium	<0.2		
Phosphorous	<0.4		
Iron	<0.2		
Copper	<0.008		
Monoglycerides	3620		
Diglycerides	1960		
Triglycerides	1230		
Total Glycerin	1520		
Ethanol	<0,2		

Certificate of Analysis

Standard Reference Material® 2772

B100 Biodiesel (Soy-Based)

Standard Reference Material (SRM) 2772 is a commercial 100% biodiesel produced from soy. SRM 2772 is intended for use in evaluating analytical methods for the determination of selected chemical and physical properties in pure biodiesel (B100). A unit of SRM 2772 consists of five 10-mL ampoules, each containing approximately 10 mL of biodiesel.

The development of SRM 2772 was a collaboration between the National Institute of Standards and Technology (NIST), USA and the National Institute of Metrology, Standardization, and Industrial Quality (INMETRO), Brazil.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for nine fatty acid methyl esters and water are provided in Tables 1 and 2, respectively. Certified values for density at 20 °C and kinematic

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Certificado de Material de Referência

Item Identification

Item: Anhydrous Bioethanol Fuel

Certificatory Institutes: National Institute of Metrology, Standardization and Industrial Quality – Inmetro
National Institute of Standards and Technology – NIST

Identification Code: CRM 06.3/09-0001 **Service Code:**

Administrative Information concerning Reference Material Certification

Inmetro Register: No applied **Certification Date:** May, 12th 2009

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Reference Materials for Biofuel Specifications

VSL

NIST

irmm Institute for Reference Materials and Measurements

INMETRO

NPL National Physical Laboratory

LGC Setting standards in analytical science

BIOREMA

Within the framework of an EC funded project with the acronym BIOREMA, REference MAterials for BIOfuel specifications, possible reference materials for Bioethanol and Biodiesel are being investigated. The aim of this project is to demonstrate the feasibility of preparing and characterizing reference materials for biofuels. Thereby, the focus is on providing SI-traceable reference values. Further, the project will establish the current state of measurement quality by means of interlaboratory comparisons using these test materials.

Introduction

With the introduction of the European Directives on renewable energies (RED 2009/28/EC) and on fuel quality (FQD 2009/30/EC), and with the increased addition of biological products to gasoline and diesel, e.g. bio-ethanol and FAME, the quality of these products is becoming more important. There is however up to now no international consensus on the technical specifications of biofuel. Neither is it clear what measurement standards, reference materials and measurement techniques are needed.

Needs

Reference materials for biofuels with well-characterized reference values are essential for the development and validation of measurement methods. Also, these materials are an important tool in quality assurance of the day-to-day measurements, *i.e.*, in obtaining reliable, traceable, measurement results.

Objectives

The main objective of the project is to establish the feasibility of the preparation of biodiesel and bio-ethanol reference materials with traceable reference values. To that end, test materials will be prepared and characterized with high-level measurement methods. Additionally, the short-term as well as the long-term stability of the reference values will be assessed.

Another important objective is to establish the current quality of the measurement practice of field laboratories. Information on the quality (repeatability, reproducibility, bias of measurement) will be obtained from interlaboratory comparisons that are organized using the characterized test materials.

Partners Involved

- VSL (the Netherlands), project coordinator
- IRMM (European Commission)
- NPL (United Kingdom)
- INMETRO (Brazil)
- NIST (USA)
- LGC (United Kingdom)

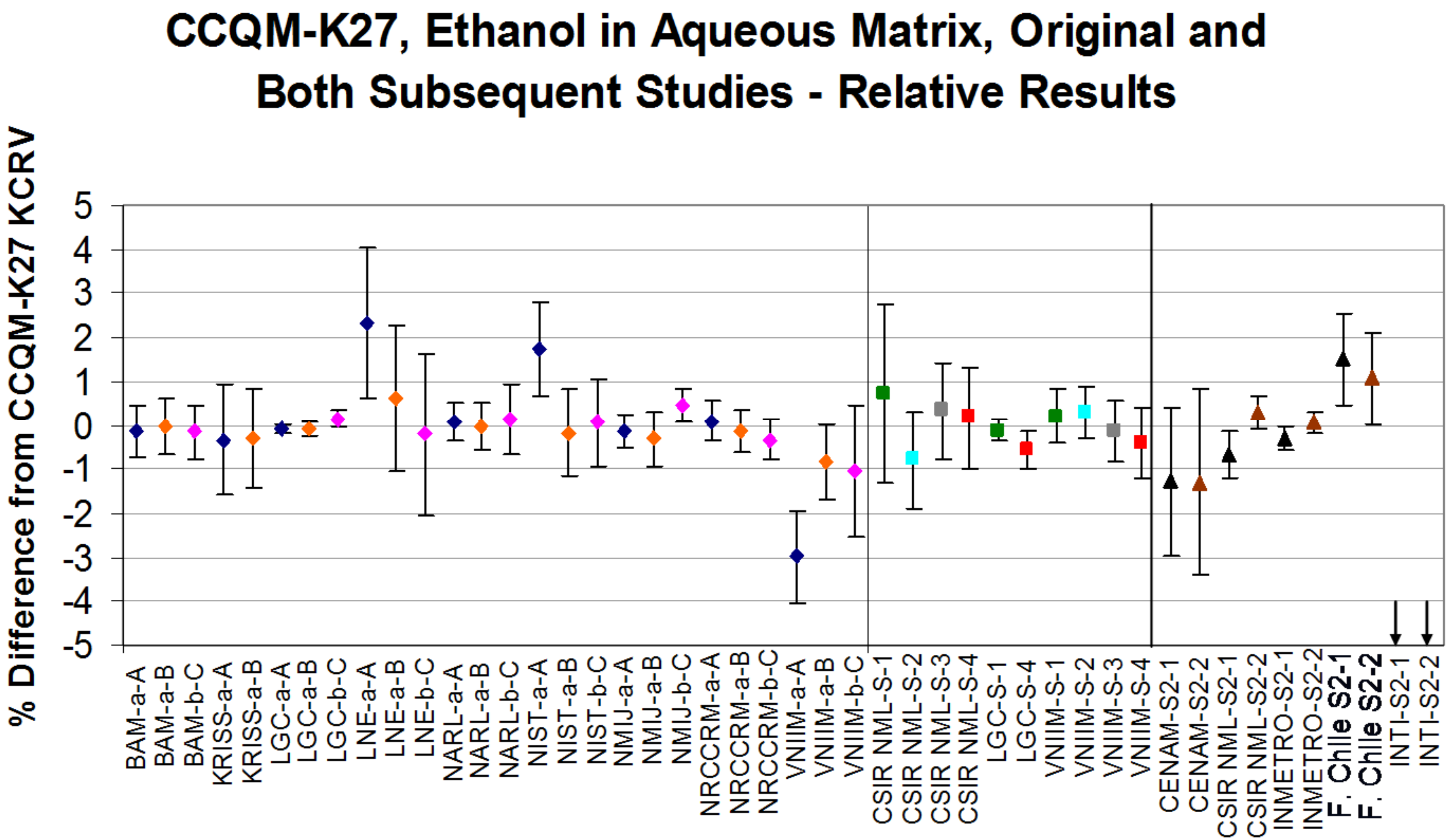
SEVENTH FRAMEWORK PROGRAMME

Project BIOREMA-CRMs with UE

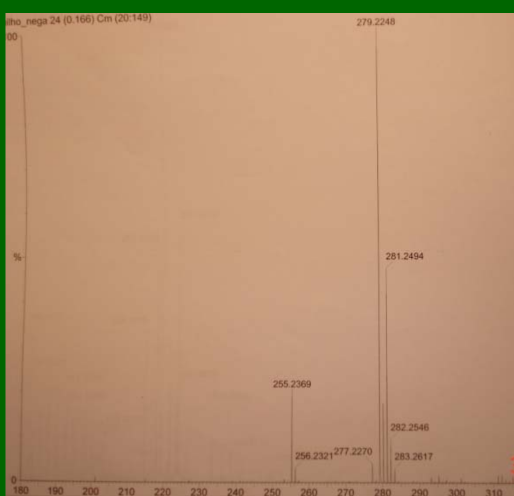
Involving:

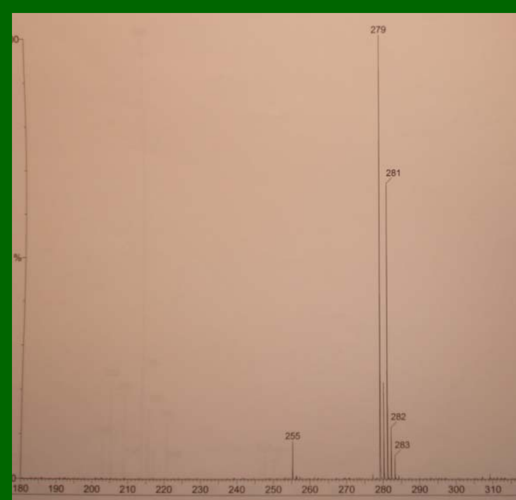
-Feasibility of the preparation of biodiesel and bioethanol reference materials.

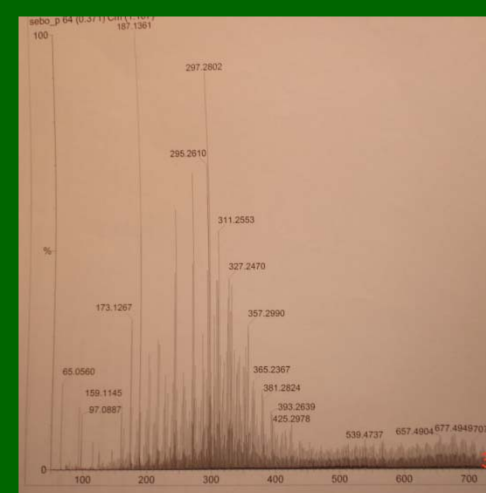
-To establish the current quality of the measurement practice in laboratories (about 35 laboratories around the world will participate).


CCQM-K27, Ethanol in Aqueous Matrix, Original and Both Subsequent Studies - Relative Results
% Difference from CCQM-K27 KCRV
5
4
3
2
1
0
-1
-2
-3
-4
-5
BAM-a-A
BAM-a-B
BAM-b-C
KRISS-a-A
KRISS-a-B
LGC-a-A
LGC-a-B
LGC-b-C
LNE-a-A
LNE-a-B
LNE-b-C
NARL-a-A
NARL-a-B
NARL-b-C
NIST-a-A
NIST-a-B
NIST-b-C
NMIJ-a-A
NMIJ-a-B
NMIJ-b-C
NRCCRM-a-A
NRCCRM-a-B
NRCCRM-b-C
VNIIM-a-A
VNIIM-a-B
VNIIM-b-C
CSIR NML-S-1
CSIR NML-S-2
CSIR NML-S-3
CSIR NML-S-4
LGC-S-1
LGC-S-4
VNIIM-S-1
VNIIM-S-2
VNIIM-S-3
VNIIM-S-4
CENAM-S2-1
CENAM-S2-2
CSIR NML-S2-1
CSIR NML-S2-2
INMETRO-S2-1
INMETRO-S2-2
F. Chile S2-1
F. Chile S2-2
INTI-S2-1
INTI-S2-2

Biodiesel with guaranteed origin

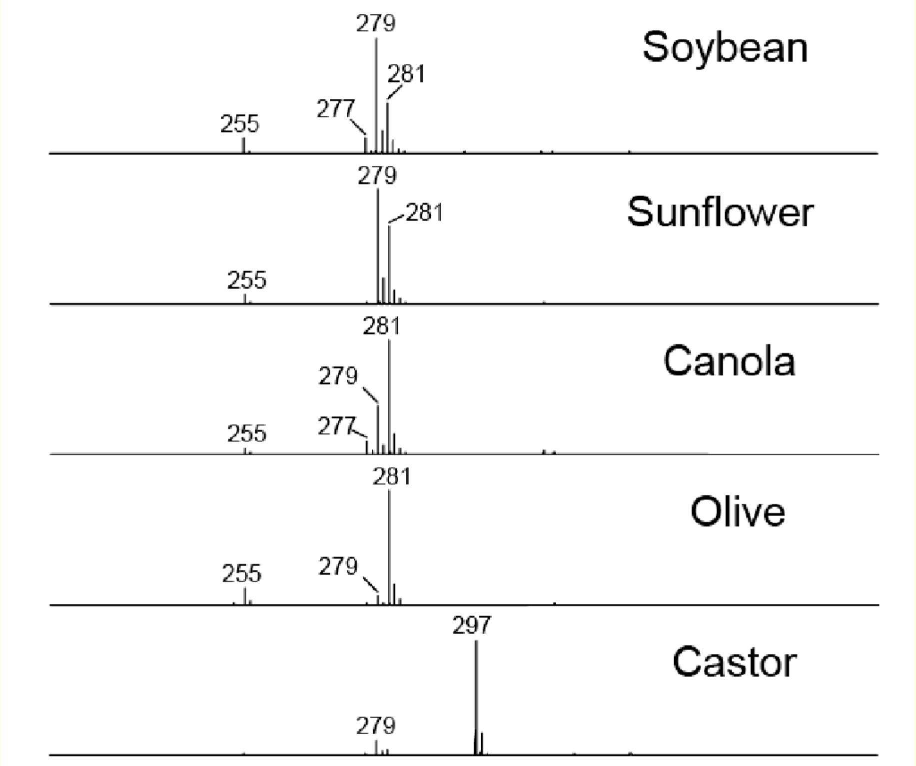






Program to identify the composition of Biodiesel and to produce samples with guaranteed origin.

An ESI-MS (electrospray ionization mass spectrometry) technique is used giving a fingerprint for each biodiesel.



Development of new analytical techniques to improve identification and characterization of biofuels

-Blends composition
-oil source origin
-contaminants identification (SVO addition)

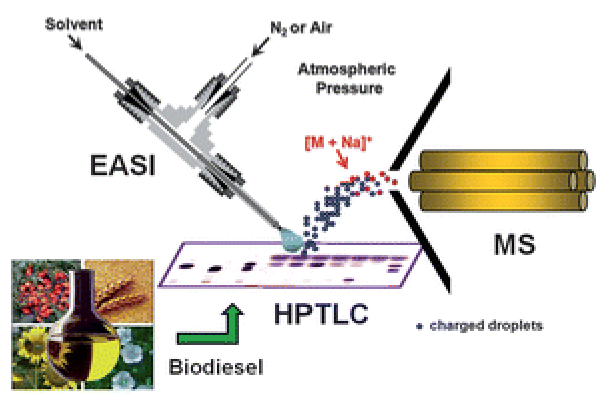
Paper

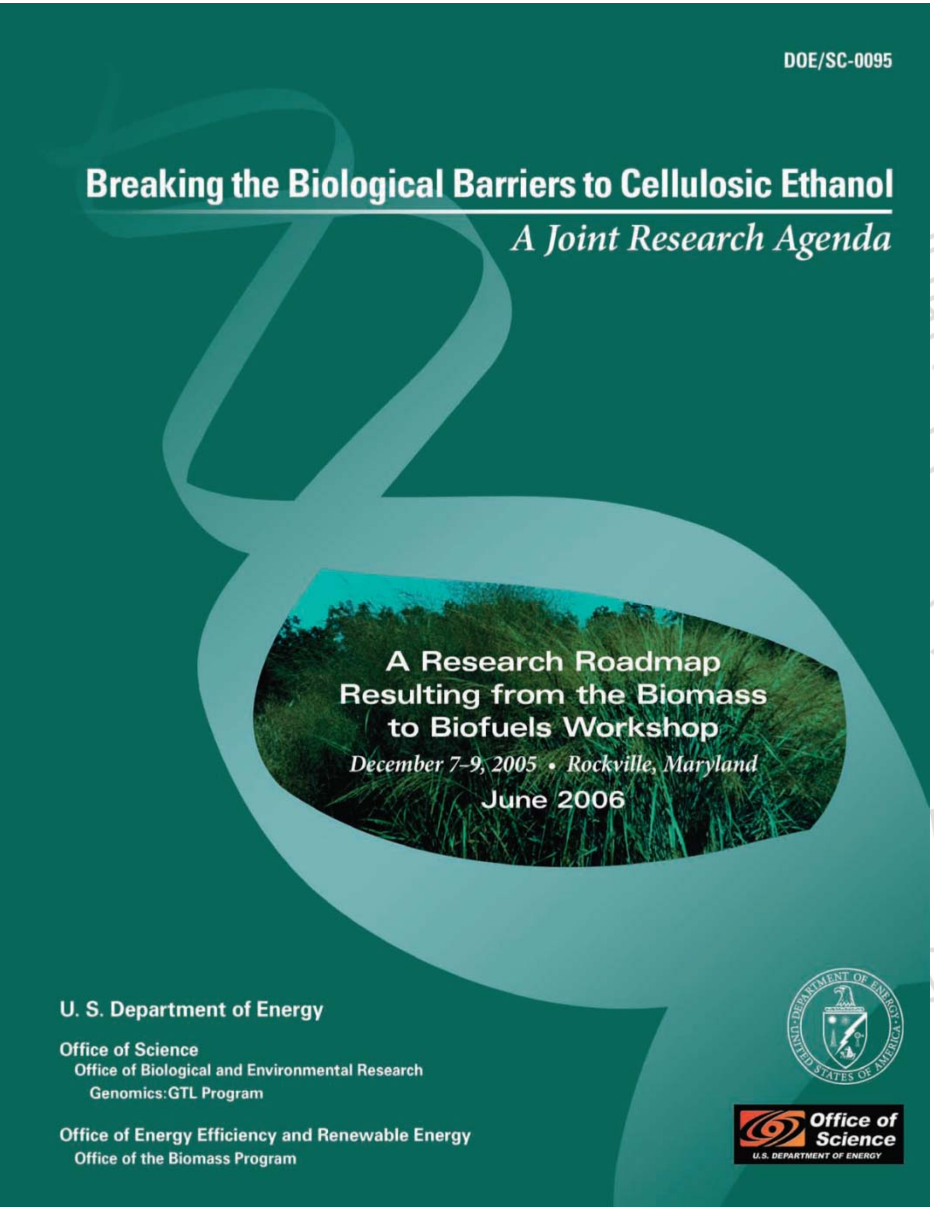
Analyst, 2009, **134**, 1652 - 1657, **DOI:** 10.1039/b817847j

Analysis of biodiesel and biodiesel–petrodiesel blends by high performance thin layer chromatography combined with easy ambient sonic-spray ionization mass spectrometry

Livia S. Eberlin, Patricia V. Abdelnur, Alan Passero, Gilberto F. de Sa, Romeu J. Daroda, Vanderlea de Souza and Marcos N. Eberlin

High performance thin layer chromatography (HPTLC) combined with on-spot detection and characterization *via* easy ambient sonic-spray ionization mass spectrometry (EASI-MS) is applied to the analysis of biodiesel (B100) and biodiesel–petrodiesel blends (BX). HPTLC provides chromatographic resolution of major components whereas EASI-MS allows on-spot characterization performed directly on the HPTLC surface at ambient conditions. Constituents (M) are detected by EASI-MS in a one component–one ion fashion as either $[M + Na]^+$ or $[M + H]^+$. For both B100 and BX samples, typical profiles of fatty acid methyl esters (FAME) detected as $[FAME + Na]^+$ ions allow biodiesel typification. The spectrum of the petrodiesel spot displays a homologous series of protonated alkyl pyridines which are characteristic for petrofuels (natural markers). The spectrum for residual or admixture oil spots is characterized by sodiated triglycerides $[TAG + Na]^+$. The application of HPTLC to analyze B100 and BX samples and its combination with EASI-MS for on-spot characterization and quality control is demonstrated.





SECOND GENERATION BIOFUELS

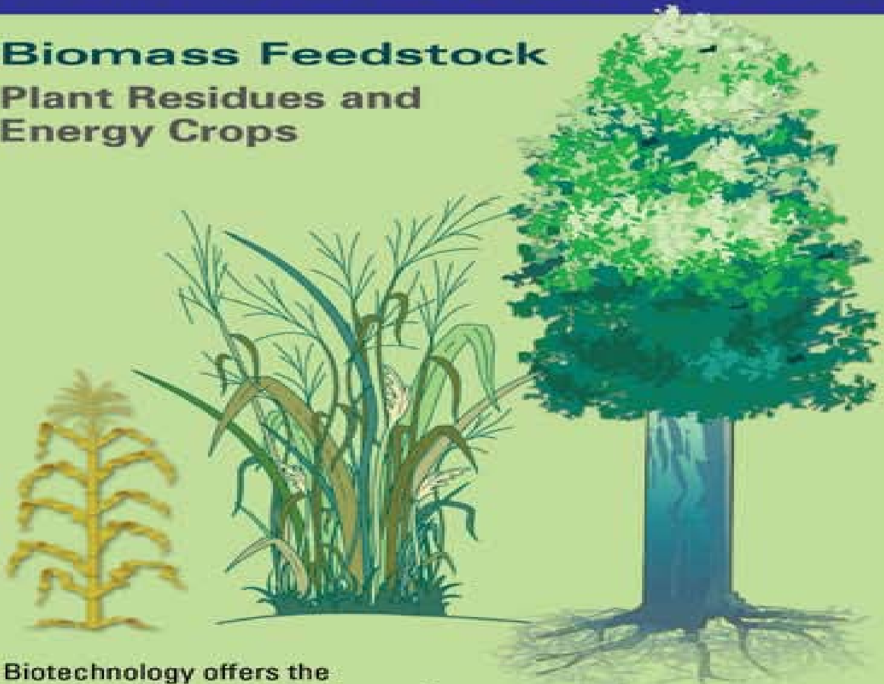
Fuels derived from cellulosic biomass—the fibrous, woody, and generally inedible portions of plant matter—offer one such alternative to conventional energy sources that can dramatically impact the need for clean fuel

Research is centered on enzymatic breakdown of cellulosic biomass to component 5- and 6-carbon sugars and lignin, using a combination of thermochemical and biological processes, followed by cofermentation of sugars to specified endproducts such as ethanol.

From BIOMASS to CELLULOSIC ETHANOL

Biomass Feedstock

Plant Residues and Energy Crops



Biotechnology offers the promise of dramatically increasing ethanol production using cellulose, the most abundant biological material on earth, and other polysaccharides (hemicellulose). Residue including postharvest corn plants (stover) and timber residues could be used, as well as such specialized high-biomass "energy" crops as domesticated poplar trees and switchgrass.

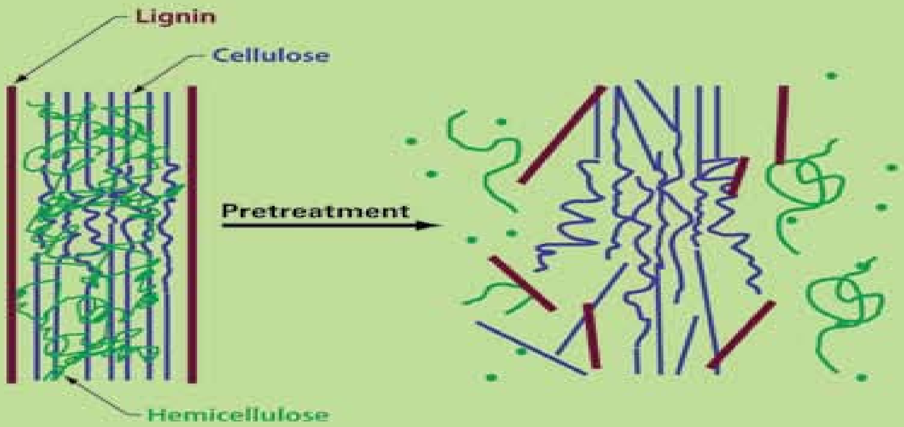
Biochemical conversion of cellulosic biomass to ethanol for transportation fuel currently involves three basic steps:

- Pretreatments to increase the accessibility of cellulose to enzymes and solubilize hemicellulose sugars
- Hydrolysis with special enzyme preparations to break down cellulose to sugars
- Fermentation to ethanol

Making cellulosic biomass conversion to ethanol more economical and practical will require a science base for molecular redesign of numerous enzymes, biochemical pathways, and full cellular systems.

Pretreatment

Goal: Make cellulose more accessible to enzymatic breakdown (hydrolysis) and solubilize hemicellulose sugars

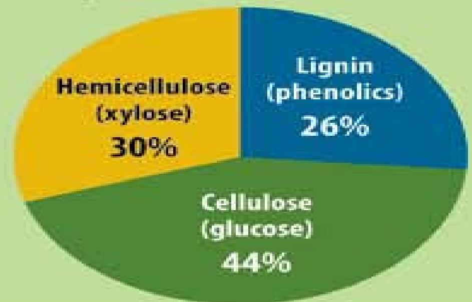


Cellulose exists within a matrix of other polymers, primarily hemicellulose and lignin. Pretreatment of biomass with heat, enzymes, or acids removes these polymers from the cellulose core before hydrolysis.

Pretreatment, one of the most expensive processing steps, has great potential for improvement through R&D.

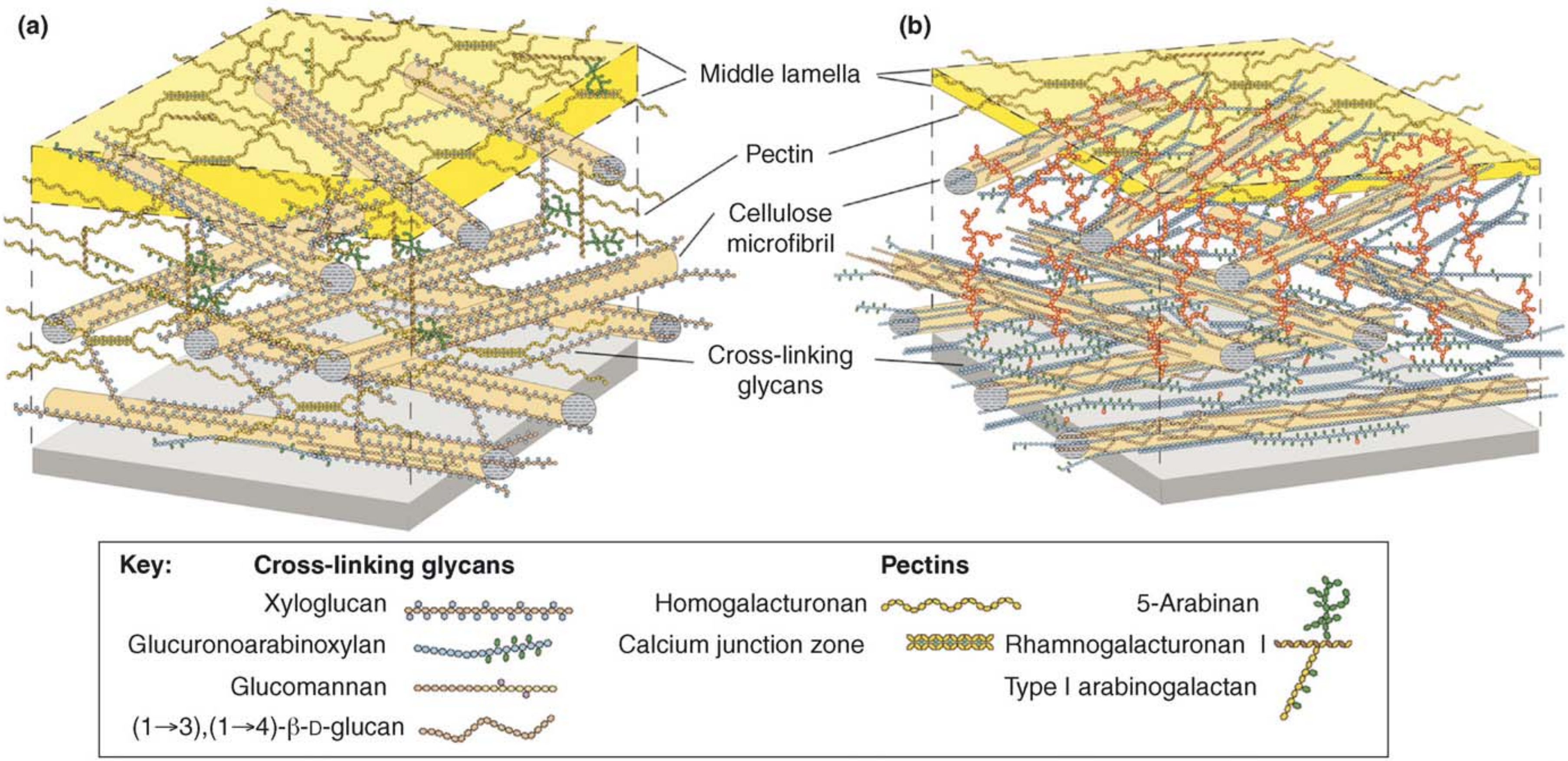
[Figure adapted from N. Mosier et al. 2005. "Features of Promising Technologies for Pretreatment of Lignocellulosic Biomass," Bioresource Technology **96***(6), 673–86. Reprinted with permission from Elsevier.]*

Composition of Biomass (lignocellulose)



SUGAR CANE BAGASSE BIOMASS

COMPLEX STRUCTURE



Production of CRM for Sugarcane Bagasse

Raw Bagasse → Maceration → Dry Milling

↓

Fractionation → Wet Milling → Autoclaving

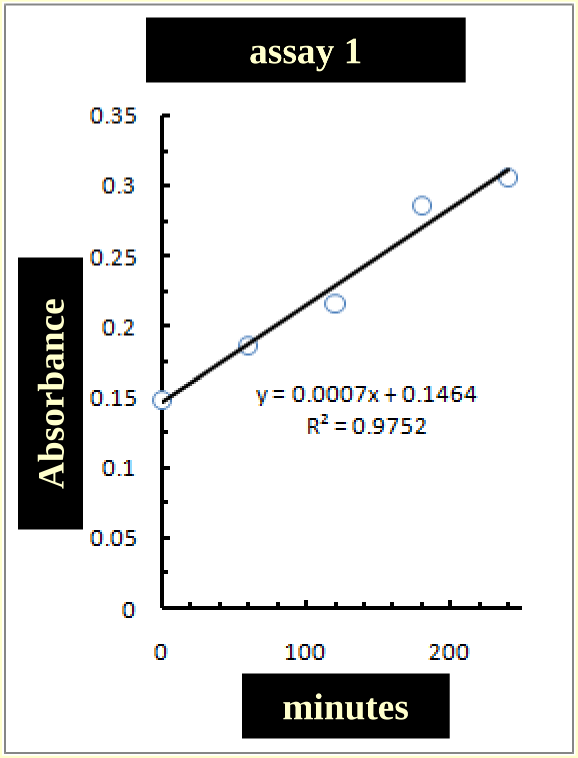
↓

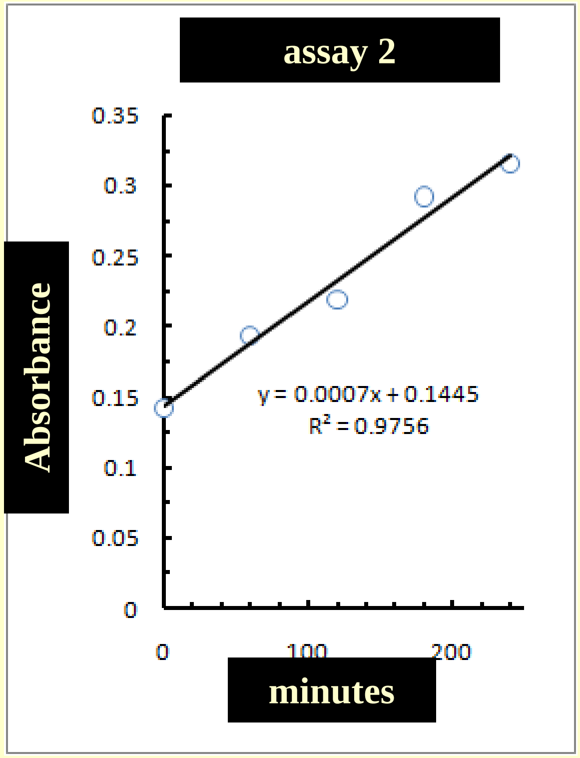
Quantification → Powdered Sugarcane Bagasse, colloidal

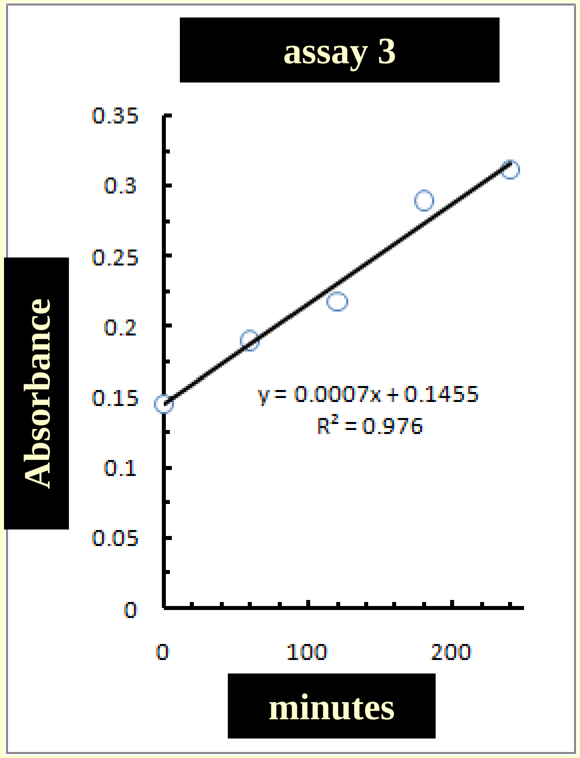
Suitable for enzymatic assays

- *Stability 4ºC – 6 months*
- *Freeze*
- *Centrifugation*
- *Liophylization*
- *Ultrasound Sensibility*

Reproducibility (Trichoderma)







Conclusions

1- Biofuels are really important, at least in the short term

2- Metrology is a key enabling technology for the worldwide use of biofuels.

3- Excellent opportunity for metrology and metrogists

4- Wide range of demands- from very technical details to cutting edge scientific problems.

5- Some relevant short term needs: CRM, PT, awareness, work with regulators and SDOs, biology metrology, origin determination, new methods (esp. in field),…

6- Exiting time!

Ministério do Desenvolvimento, Indústria e Comércio Exterior

BRASIL UM PAÍS DE TODOS GOVERNO FEDERAL