INMETRO

Ministério do Desenvolvimento, Indústria e Comércio Exterior

GOVERNO FEDERAL BRASIL PAÍS RICO É PAÍS SEM POBREZA

4th International Conference on Biofuels Standards: Current Issues, Future Trends

November 13 - 15, 2012 ♦

Gaithersburg, MD

Development of CRM for Biofuels

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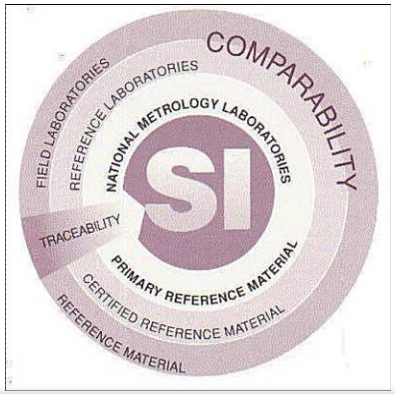
    - SENGE
  - DICEP
  - DITER
  - DIOPT
  - DIMEC
  - DIELE
  - DIAVI
  - DQUIM
    - LABEL
    - LABOR
    - LABAG
    - LABIN
    - LAMOC
  - DIMAT
  - DITEL
  - DINAM
  - DIBIO

Overview

- Introduction – why CRM?
- The cooperation NIST - Inmetro
- Inmetro Biofuel Strategy

Why Chemistry needs Metrology??



Yes! The chemical measurement are important.

Measurements: 80 billions Euros or 1% of European GDP

The Assessment of the Economic Role of Measurements and Testing in Modern Society. Survey directed by Geoffrey Williams, Pembroke College, Oxford, **2002**

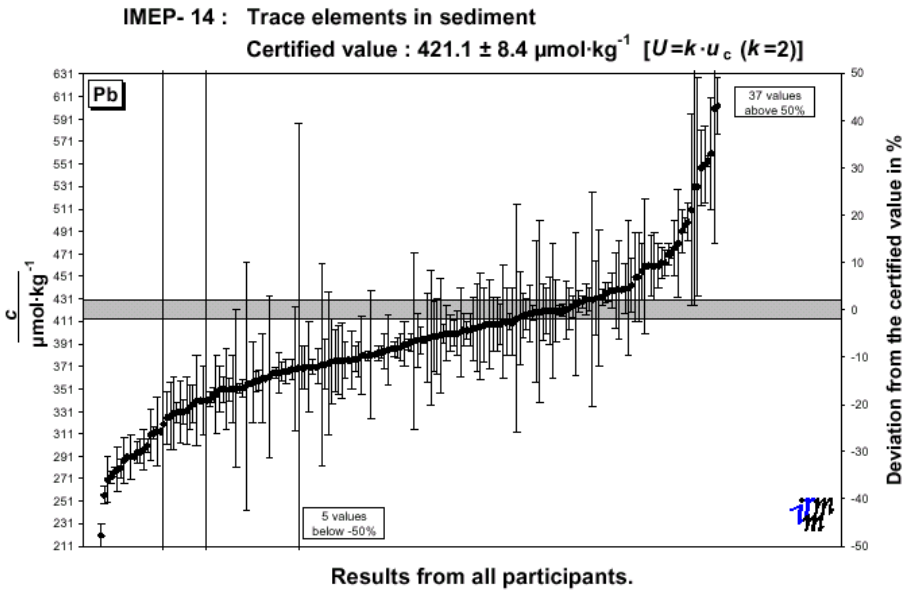
Chemical analysis: a big business...

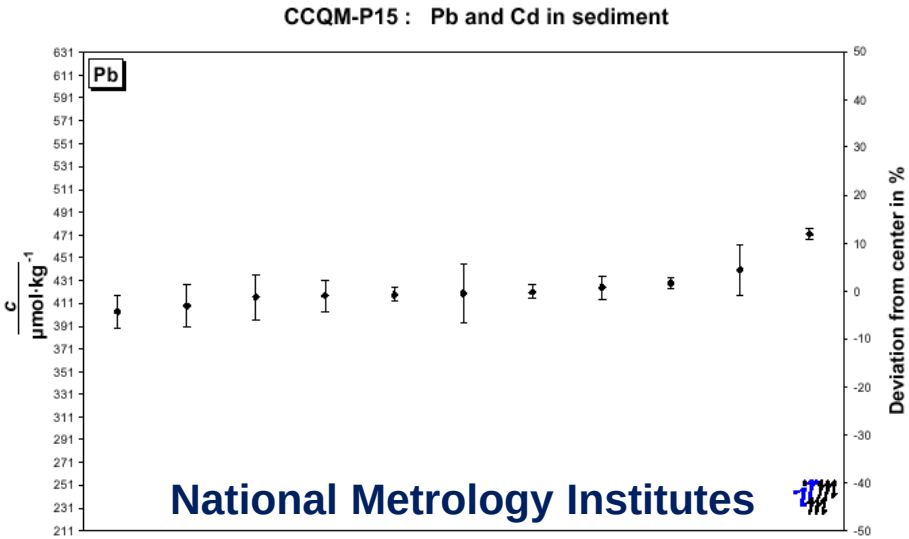
Are the laboratories prepared for this task??

Among 5 and 30% of the chemical measurement results are unsatisfactory!

Metrology in Chemistry. Current Activities and Future Requirements in Europe.

The idea of plug and play!

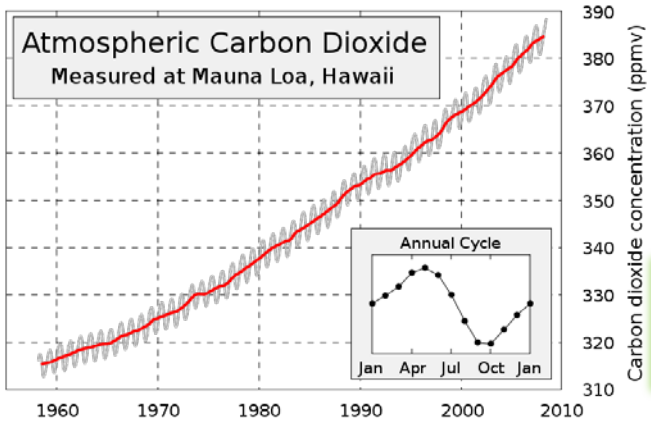




Chemical Analysis is trivial!

Metrology tools are crucial!

National Metrology Institutes



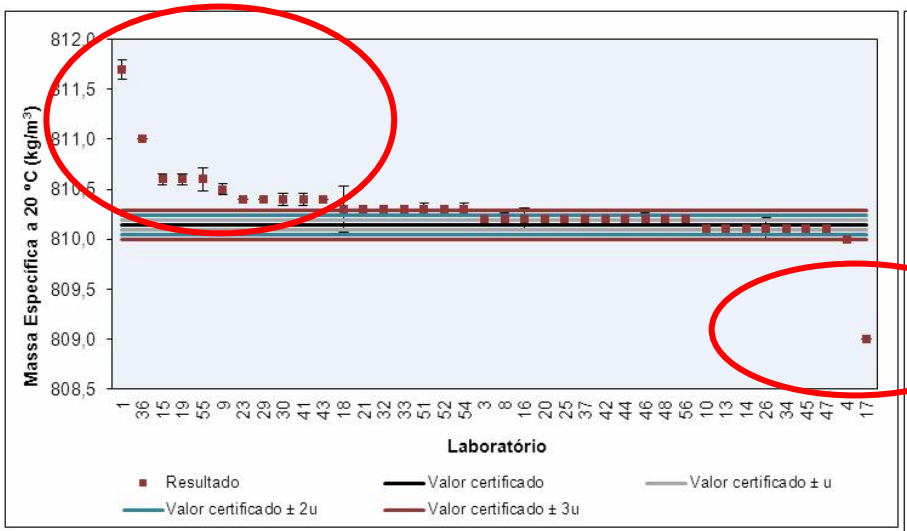
Fossil Fuels

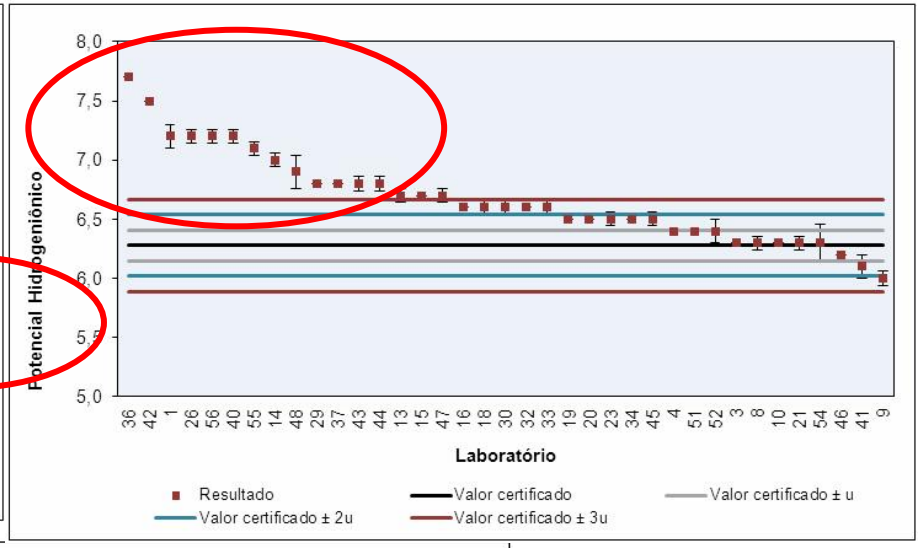
billions of oil barrels/year
And more: Coal, natural gas…

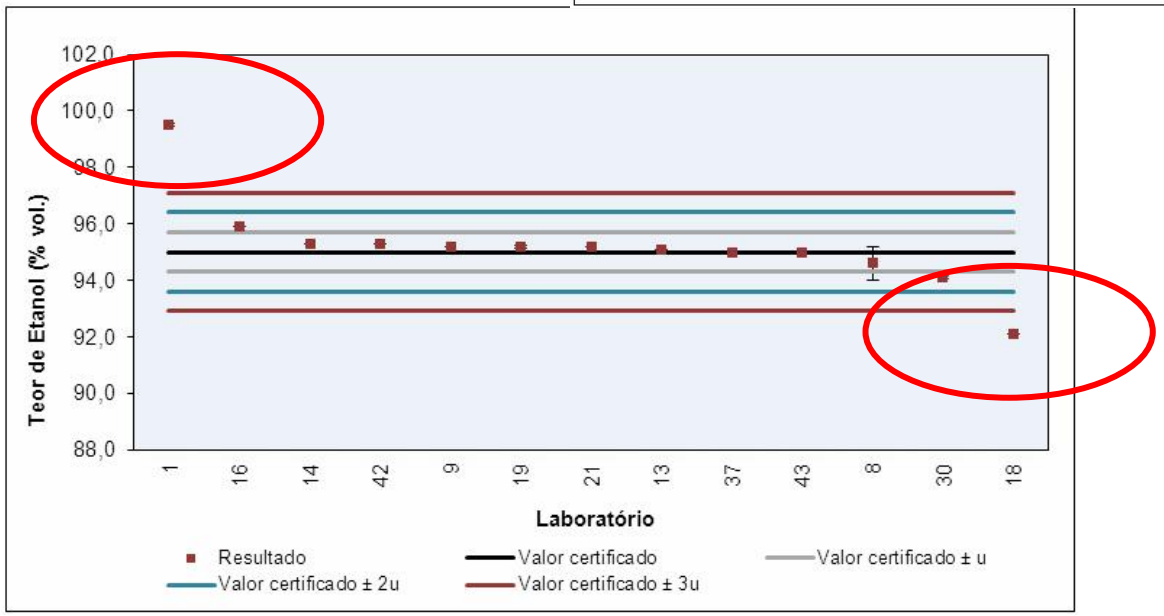
The CO_2 concentration in the atmosphere increased 35% since the Industrial Revolution

An alternative: the use of biofuels

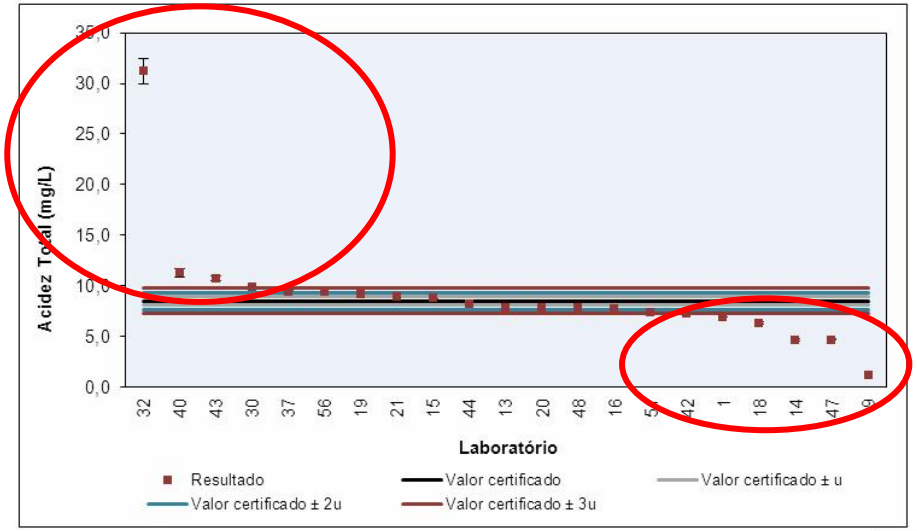
PTScheme in Brazil (Inmetro-ANP) - bioethanol

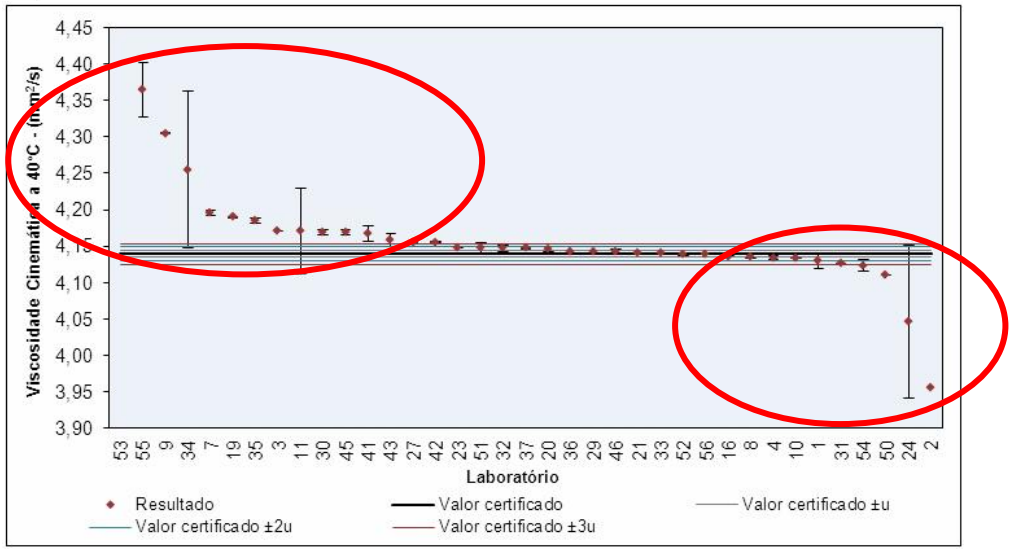






PTScheme in Brazil (Inmetro-ANP) - biodiesel





Main Purpose of Metrology

To provide confidence in measurements

↓

Traceability

Use of CRM

Ensuring Quality to the end Consumers involves definitions to the following questions:

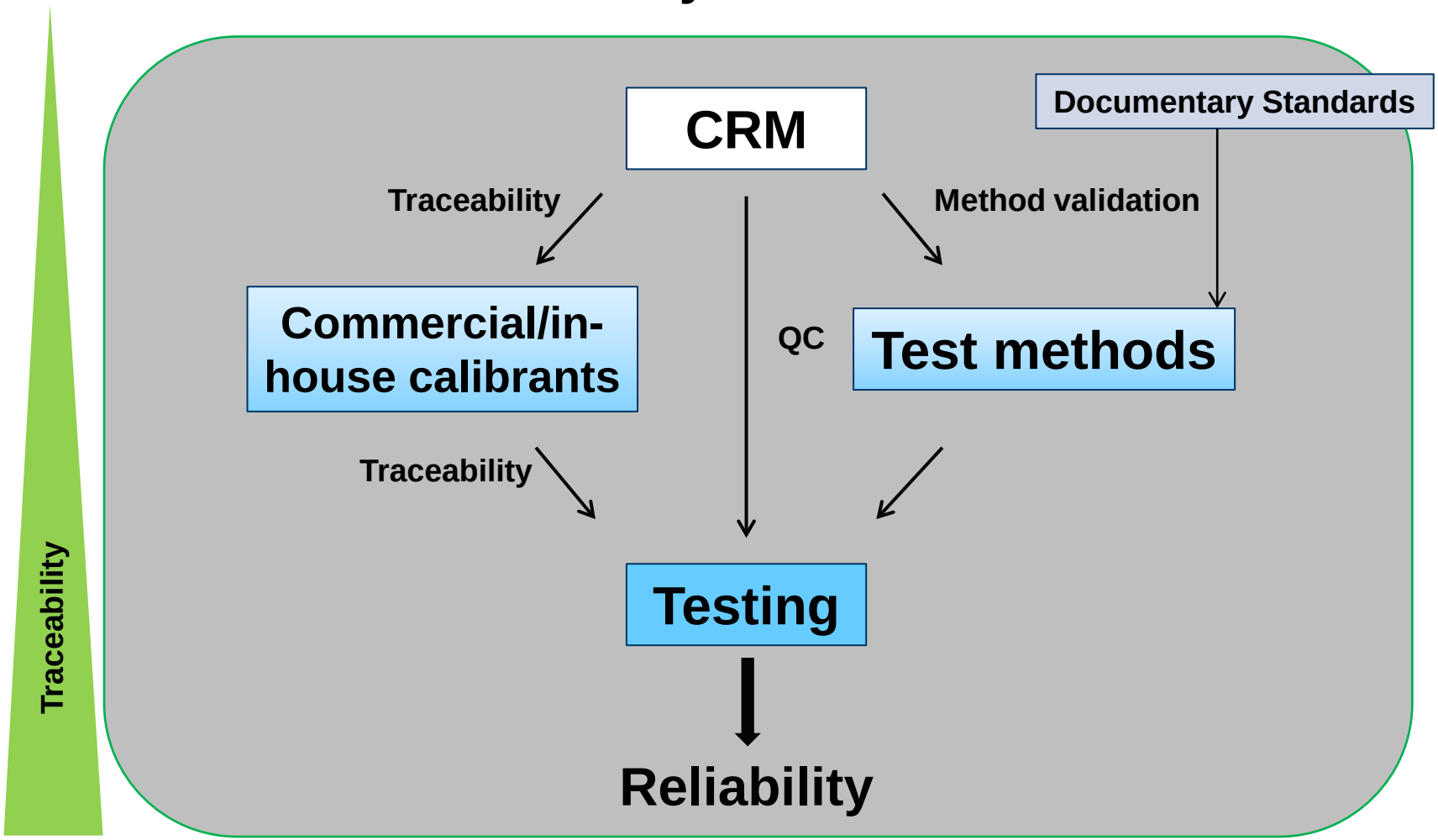
What to measure?	How much to measure?	How to measure?	How to ensure the measured value?
Set parameters	Define limits	Define methods	Prepare CRMs

Organisms involved in the definitions:

What and how much to measure	How to ensure the measured value
↓	↓
Normalization bodies	**NMIs**

SI
Mol

Why CRM?



Adp. Baldan, Annarita/Biorema Project

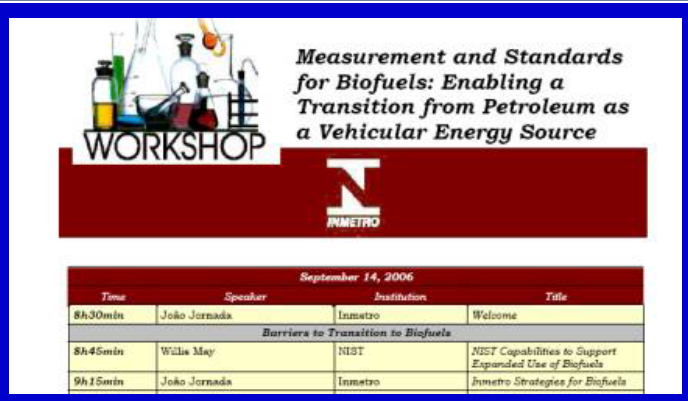
The road map of the development, preparation and certification of biofuels RM

Cooperation Inmetro - NIST

NIST

INMETRO

President Bush's first visit to Brazil. It was the launch of the biofuel seed.



September 14, 2006			
Time	Speaker	Institution	Title
8h30min	João Jornada	Inmetro	Welcome
Barriers to Transition to Biofuels			
8h45min	Willie May	NIST	NIST Capabilities to Support Expanded Use of Biofuels
9h15min	João Jornada	Inmetro	Inmetro Strategies for Biofuels

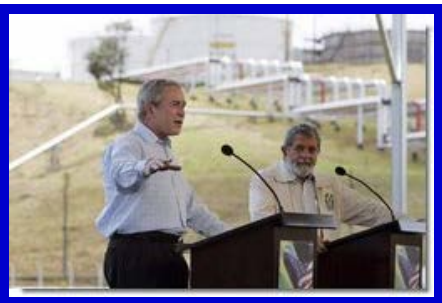
Nov. 2005 **Jun.2006** **Set. 2006**

Meeting "US-Brazil Commercial Dialogue" between the Secretary Gutierrez (USA) and Minister Furlan (Br).

”US-Brazil Ministerial Joint Comission Meeting”.

The U.S.-Brazil JCM acknowledged the long history of cooperation between the United States and Brazil in the area of the measurement sciences. **The JCM confirmed the importance of cooperation in the fields of metrology and standards and welcomed the report on the activitie agreed to by the National Institute for Standards and Technology (NIST) and the National Institute of Metrology, Standardization and Industrial Quality (INMETRO) under the Science and Technology Agreement between the Government of Brazil and the Government of the United States and under the U.S.-Brazil Commercial Dialogue in measurement standards**. It noted the mutually agreed desire for future collaboration in the areas of nanoscale science, postdoctoral exchanges, and measurement standards in high-priority areas such as biofuels and nano-materials.

The JCM recognized and encouraged expanded cooperation in standards and metrology activities that impact innovation and competitiveness and support economic growth and development, includin in nanotechnology and **biofuels**



Second President Bush's visit to Brazil. Signed the "Memorandum of Understanding" on cooperation in the area of biofuel.

In Brasilia, the second "Commercial Dialogue meeting" between the Secretary Gutierrez and the Minister Miguel Jorge, INMETRO and NIST defined the agenda of cooperation :

Mar. 2007 **Jul. 2007** **Jul. 2007** **Oct.2007**

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 the "Washington Symposium" was organized the "Tripartite Task Force" involving Brazil, USA and EU related to harmonization of quality specifications for biodiesel and bioethanol.

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1) Joint development and production of Certified Reference Materials – CRM´s with the main quality parameters of Regional ethanol and biodiesel specifications.

2) Development and validation of a method for the biodiesel source identification (eg. Soy, animal fat, palm oil, castor oil etc.) based on chemical fingerprint.

3) Development of a method based on isotope ratio analysis by mass spectrometry to identify the geographical origin of biodiesel as a tool for the certification process.

During the meeting of the CCQM in Paris. NIST and INMETRO finalized the agreement for the production of MRC with definitions for quantities, packaging and marketing

Oct. 2007 **Jan. 2008** **Jan. 2008** **Apr. 2008**

On January 23-24, 2008, representatives from NIST and INMETRO met to discuss the joint development of three biofuel CRMs in 2008. NIST was represented by Dr. Willie E. May, Director of the NIST Chemical Science and Technology Laboratory (CSTL) and Dr. Stephen A. Wise, Chief of the Analytical Chemistry Division within CSTL. INMETRO was represented by Prof. João Jornada, President INMETRO; Prof. Humberto S. Brandi, INMETRO Director of Scientific and Industrial Metrology; Prof. Romeu Jose Daroda, Biofuels Project Coordinator; and Dr. Vanderlea Souza, Head of the Chemical Metrology Division.

Based on discussions/agreements from the January meeting, NIST and INMETRO will work collaboratively to produce CRMs for both bioethanol and biodiesel. INMETRO will take the lead in producing a bioethanol CRM, and NIST will take the lead in producing the biodiesel CRMs. INMETRO is currently preparing two candidate CRMs for bioethanol, anhydrous ethanol and hydrated ethanol. NIST has prepared two candidate biodiesel CRMs, one material is soy-based and the other material is animal fat-based.

Starting at Inmetro the BioMRC Project, funded by FINEP.

The Project Includes R & D with broad spectrum in biofuels, including 2nd generation of ethanol production by identifying enzymes for cellulose degradation and development of methodology for identifying the geographical origin of biofuels.

NIST (U.S.), and **INMETRO** (Brazil) have established plans for cooperative development of:

Certified Reference Materials:

-Anhydrous and Hydrated bioethanol (Inmetro)
-Soy and Animal-based biodiesel (NIST)

-for **calibrating** measurement instruments to a known and internationally accepted reference
-for **validating** the accuracy of measurement results and measurement platforms, space and time.

Reference Measurement Methods:

-Chemical pattern recognition to identify feedstock source of biodiesel (e.g. soy, rapeseed, animal fat)
-Isotope metrology to distinguish between renewable/nonrenewable fuels

Start the Project BIOREMA involving the NMIs: Inmetro, NIST, VSL, NPL, IRMM and LGC. Realization of an intercomparison with ethanol and biodiesel, involving 30 lab around the world. MRC was used for bioethanol produced by Inmetro biodiesel produced by NIST and production of a CRM from rapeseed by IRMM.

Reference Materials for Biofuel Specifications

BIOREMA

Oct. 2008

Nov. 2008

During the International Conference on Biofuels: "Biofuels as a vector for sustainable development," Professor Jornada (President of Inmetro) delivery to President Lula sample of bioethanol Certified Reference Material produced by Inmetro in a partnership with NIST.

Biofuel Reference Material (BIOREMA) Project

Objective

- Establish the preparation of bioethanol (Inmetro) and biodiesel (NIST) reference material with traceability
- Organize a PT scheme about 30 laboratories of the world with biofuels (bioethanol and biodiesel) samples certified by:

Executive Summary from BIOREMA Reference Materials for Biofuel specifications http://www.inmetro.gov.br/painelsetorial/palestras/publishable-summary.pdf.

INMETRO Ministério do Desenvolvimento, Indústria e Comércio Exterior

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Availability of the CRM of biodiesel to users, developed by NIST in partnership with Inmetro

National Institute of Standards & Technology

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

March 2009

Inmetro, Nist and the European Commission organized in Brussels the "2nd International Conference on Biofuels Standards".
Objective: To discuss the harmonization of biofuel specifications and test methods used.

Certified Reference Material for Biodiesel

May, W. / NIST

SRM 2772 B100 Biodiesel (Soy-based)

-Received from a farmer-owned cooperative in Nebraska (US)

-Produced from refined, bleached, and deodorized soy oil

-10 mL amber ampoules evacuated with argon prior to filling with 10 mL of biodiesel and flame-sealing

- 9 FAME, water, density, viscosity at 40º C (Certified values)
- 3 FAME, acid number, viscosity 20 and 30º C, oxidation, gross heating value,, methanol, individual glycerides (Reference values)
- Trace elements, and summed glycerides (Informative values)

Availability of the CRM of anhydrous ethanol to users, developed by Inmetro in partnership with NIST.

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

Early 2010 **March 2010** **July 2010**

"3rd International Conference on Biofuels Standards" held in Amsterdam - Netherlands, along with the Congress World Biofuels Markets. The Conference was organized by INMETRO, NIST and the European Commission.
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Conducting a training in bioethanol with representatives of NMIs from Latin America, Central America, the Caribbean and Africa.
PEAAL - Standards of ethanol to Africa and Latin America. Conducting an Interlaboratory Comparison Program with the countries involved.

PEAAL
SEMINÁRIO PADRÕES PARA O ETANOL DA ÁFRICA E DA AMÉRICA LATINA

Programação
Programme

13 - 14 de julho
13 - 14 July

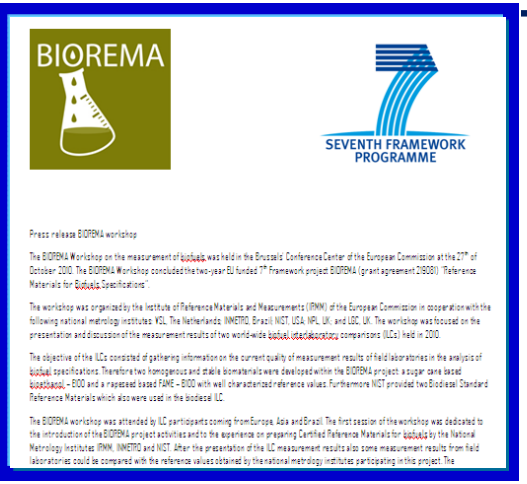
INMETRO

Certified Reference Material for Bioethanol

-**Anhydrous and Hydrated from sugar cane**
-Received from a producer of São Paulo (BR)
-20 mL amber ampoules evacuated with argon prior to filling with 20 mL of bioethanol and flame-sealing

- Acidity
- Chloride content
- Copper content
- Electrolytic conductivity
- Iron content
- Density
- pHe
- Sodium content
- Sulphate content
- Water content
- Ethanol content

Conclusion of the BIOREMA Project with a Workshop, presenting the results of the Interlaboratory Comparison Program, attended by representatives from 30 laboratories around the world who participated in the ILC



BIOREMA

SEVENTH FRAMEWORK PROGRAMME

Press release BIOREMA workshop

The BIOREMA Workshop on the measurement of biofuels was held in the Brussels Conference Center of the European Commission at the 27th of October 2010. The BIOREMA Workshop concluded the two-year EU funded 7th framework project BIOREMA (grant agreement 218081) "Reference Materials for Biofuels Specifications".

The workshop was organized by the Institute of Reference Materials and Measurements (IRMM) of the European Commission in cooperation with the following national metrology institutes: VSL, The Netherlands; INMETRO, Brazil; NCT, UCA; NPL, UK; and LGC, UK. The workshop was focused on the presentation and discussion of the measurement results of two world-wide biofuel interlaboratory comparisons (ILCs) held in 2010.

The objective of the ILCs consisted of gathering information on the current quality of measurement results of field laboratories in the analysis of biofuel specifications. Therefore two homogenous and stable biomaterials were developed within the BIOREMA project: a sugar cane based bioethanol – B100 and a rapeseed based FAME – B100 with well characterized reference values. Furthermore NCT provided two Biodiesel Standard Reference Materials which also were used in the biodiesel ILC.

The BIOREMA workshop was attended by ILC participants coming from Europe, Asia and Brazil. The first session of the workshop was dedicated to the introduction of the BIOREMA project activities and to the experience on preparing Certified Reference Materials for biofuels by the National Metrology Institutes (IRMM, INMETRO and NCT). After the presentation of the ILC measurement results also some measurement results from field laboratories could be compared with the reference values obtained by the national metrology institutes participating in this project. The

Out. 2010

Out. 2010

Inmetro through Dquim, participated in the CCQM, BIPM, as coordinator of a pilot comparison of bioethanol MRC for measuring ethanol content and water content.

CCQM-P129 Ethanol content

Direct method (GC-FID/MS)

Mass balance approach

QNMR

Adiabatic calorimetry

Visit of the President Obama to Brazil. In the MoU signed by the Presidents was mentioned the development of CRM for aviation biofuels in a joint cooperation Inmetro - NIST

TODAY
4th International Conference on Biofuels Standards: Current Issues, Future Trends

Marc 2011 **Aug. 2011** **Nov. 2012**

In a meeting at NIST (Inmetro-NIST) was defined to conduct an International Conference on biofuels with a special focus on aviation biofuels and to develop a CRM for.

Inmetro's Biofuel Strategy

Seeds

Lignin;
Cellulose
Alcohols

Extraction

Tart

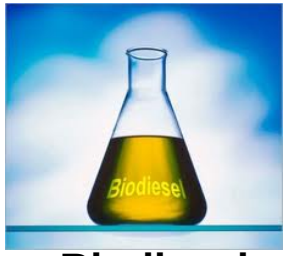
LTC

Vegetable Oil

Emulsificants
aditives
Biochart,
biooil,special
fluids;

Transesterification

Glycerin

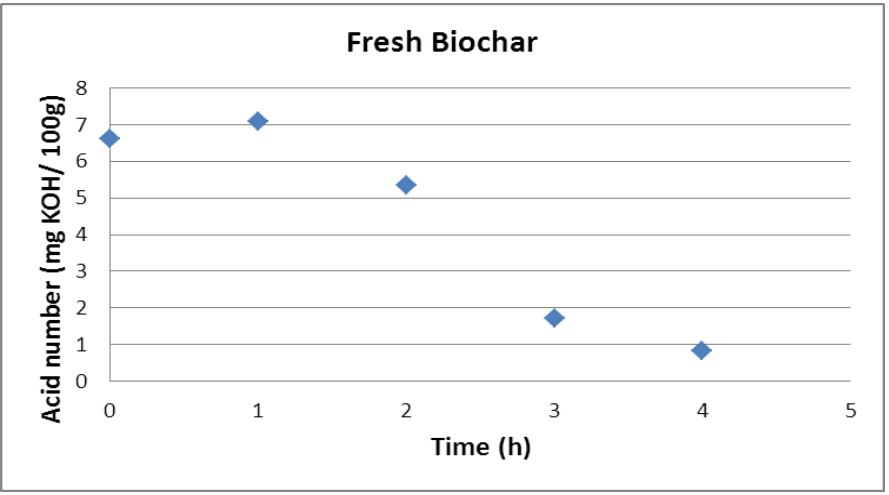


Biodiesel

Emulsificants
Special fluids
Aviation
biofuel

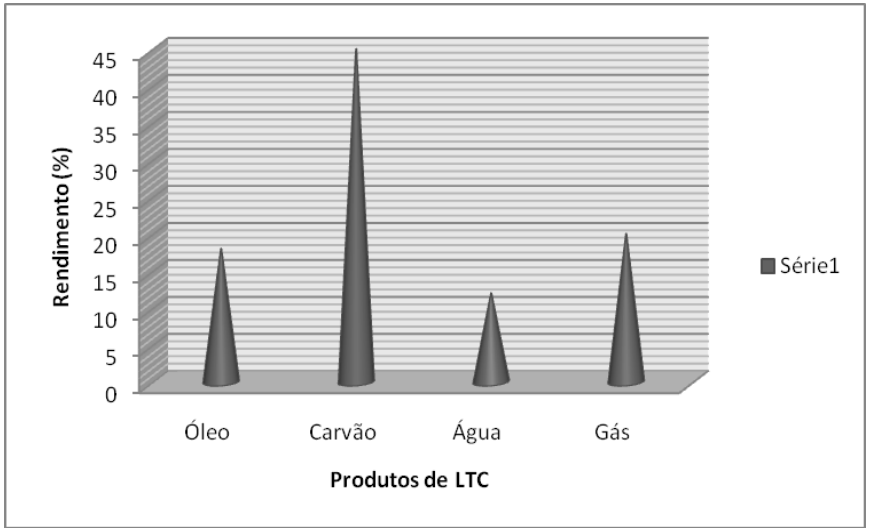
Total Use Strategy

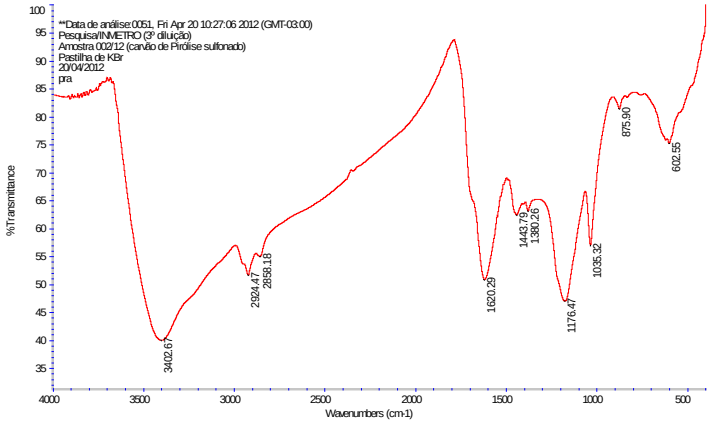
Biochar for acidity reduction in oil for biodiesel production

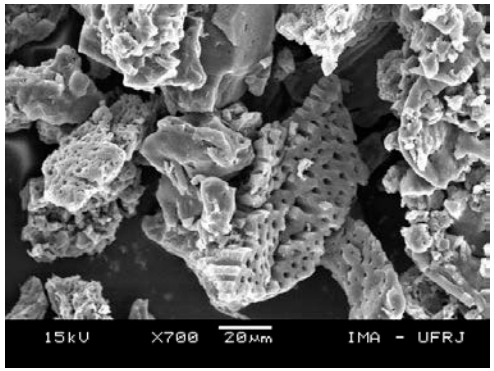


LTC – Low Temperature Conversion

LTC tart of Jatropha Curcas







Technical Cooperation Inmetro – FIAT (February/2010)

Objectives: R&D&I in biofuel and engines.

-Characterization of a B20 commercial vehicle
-Application of SVO as fuel to Diesel engines
-Nanotechnology: application in the friction reduction between engine components (new materials and coating)

Homepage databank of biodiesel parameters

Thanks for your attention!

INMETRO

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Tel: (21) 2679-9069

1925: First tests using ethanol blends with gasoline

Source: Ministry of Mines and Energy