nmisa
National Metrology Institute of South Africa

Cost-efficient Biofuels Testing - International Best Practice

Sara Prins
Research Manager

Your measure of excellence

Overview

- What is a measurement?
- The international and national measurement environment
- The NMISA
  - Mandate and Structure
  - Biofuel activities
  - FAME analysis
  - Viscosity and Density
- The road forward

© NMISA 2010

nmisa
National Metrology Institute of South Africa

What is a measurement?

- When we test for something, we perform a measurement
- Measurement is the tool that allows us to compare our goods in terms of specific and (hopefully) objective terms
- Measurements have quantity

  (i.e. the property of a phenomenon, body, or substance, where the property has a magnitude that can be expressed as a number and a reference)
- Measurements have units
- Measurements have an uncertainty
- **To be accepted everywhere measurements need to be traceable to the SI**

2 Measurement

2.1 (2.1)
measurement
process of experimentally obtaining one or more **quantity values** that can reasonably be attributed to a **quantity**

NOTE 1 Measurement does not apply to **nominal properties.**

NOTE 2 Measurement implies comparison of quantities and includes counting of entities.

NOTE 3 Measurement presupposes a description of the quantity commensurate with the intended use of a **measurement result,** a **measurement procedure,** and a calibrated **measuring system** operating according to the specified measurement procedure, including the measurement conditions.

As per JCGM 2008 (International Vocabulary of Metrology)

© NMISA 2010

Traceability to the SI: Cu in Bioethanol

References

rement Procedure

We live on different continents. We all have different instruments, laboratory conditions, we buy chemicals from different suppliers, our analysts do things slightly different – how we then still ensure that our measurements compare?

Cu

ration

nt of

od for Cu

Biofu

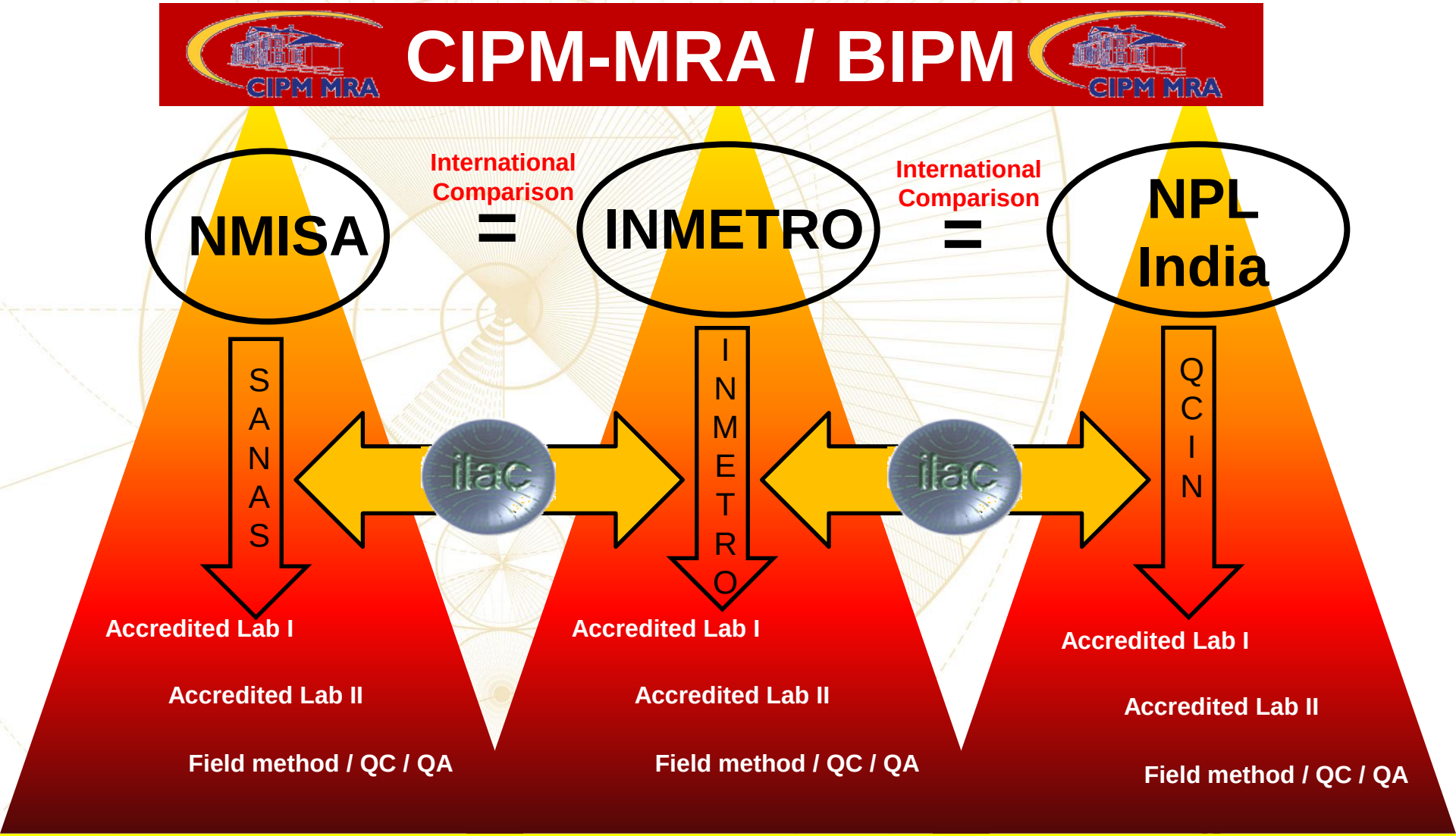
utine analysis

Traceable result

[<0.1 mg Cu/kg]

A traceable measurement allows you to compare results in time and space

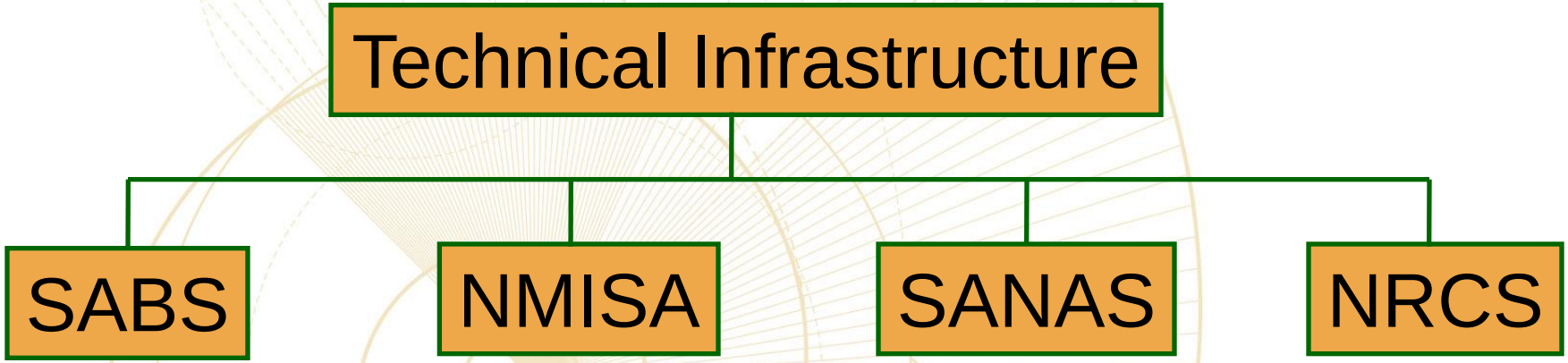
International Metrology: Measurement Equivalence



Measure once, accepted everywhere

Overcome technical barriers to trade

The South African measurement infrastructure



The Department of Trade and Industry ‘revamped’ the Technical Infrastructure:

- The Standards Act, Act 8 of 2008 (SABS – documentary standards)
- The National Measurement Unit and Measurement Standards Act, Act 18 of 2006 (NMISA – metrology, measurement traceability, measurement units, reference materials/methods)
- The Accreditation for Conformity Assessment, Calibration and Good Laboratory Practice Act, 2006 Act No. 19 of 2006 (SANAS – accreditation)
- And the National Regulator for Compulsory Specifications Act, Act 5 of 2008 (NRCS – legal (trade) metrology, conformance)

© NMISA 2010

History of the NMISA

The CSIR was founded in 1945, and the National Physical Laboratory (NPL) was established in 1947 as a division of the CSIR. The NPL was later known as the CSIR-National Metrology Laboratory

In August 1964 SA became the 40th signatory to the Metre Convention

Signed the CIPM MRA in October 1999 on behalf of the Department of Trade and Industry

Measurement Units and Measurement Standards Act (Act No 18 of 2006), makes provision for the establishment of the National Metrology institute of SA

1 May 2007: CSIR NML becomes NMISA
The NMISA is a dti Technical Infrastructure Institute

Since 2007, participated in the USA/EU/Brazil Biofuel Tripartie workshops

© NMISA 2010

nmisa
National Metrology Institute of South Africa

Mandate

to maintain the SI units and to maintain and develop primary scientific standards of physical quantities for SA and compare those standards with other national standards to ensure global measurement equivalence. It must also provide reference analysis in the case of a measurement dispute and maintain and develop primary methods for chemical analysis to certify reference materials for SA and the region

Vision

to ensure that South Africa can prove measurement comparability and equivalence with the international community, in support of trade, in all national priority areas

© NMISA 2010

nmisa
National Metrology Institute of South Africa

NMISA Structure and Activities

Technical Areas

- Metrology in Chemistry
- Mechanical Metrology
- Electromagnetic Metrology
- Research

22 laboratories: 21 accredited to ISO/IEC 17025 and two to ISO 17034

Dissemination

- International comparisons
- Calibration services and method development
- Certified reference materials
- Consultancy
- Contract research
- Technical advisory forums

© NMISA 2010

nmisa
National Metrology Institute of South Africa

NMISA laboratories supporting biofuels

Metrology in Chemistry

- Ethanol content
- Moisture analysis
- Chemical composition

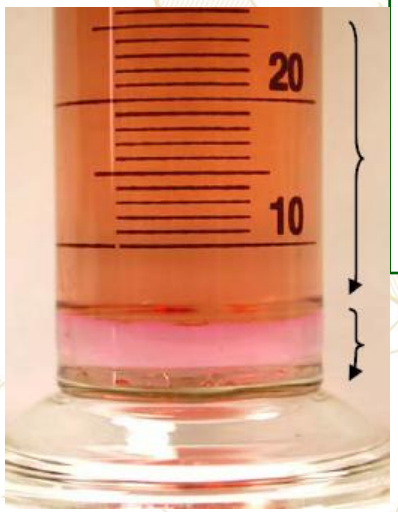
Physical metrology

- Viscosity
- Density
- Temperature
- Flashpoint
- Colour

nmisa
National Metrology Institute of South Africa

© NMISA 2010

Bioethanol specification: SANS 465



Ethanol, volume %, min	92.1
Methanol, volume %, max	0.5
Solvent-washed gum, mg/100 mL, max	5.0
Water content, volume %, max	1 (Note 1)
Denaturant content, volume %, min	1.96
volume %, max	4.76
Inorganic Chloride content, mass ppm (mg/L), max	40 (32)
Copper content, mg/kg, max	0.1
Acidity (as acetic acid CH_3COOH), mass % (mg/L), max	0.007 (56) (Note 3)
pHe	6.5 to 9.0
Sulfur, mass ppm, max	30
Appearance	Visibly free of suspended or precipitated contaminants (clear and bright)

*based on ASTM D 4806

Tests the NMISA is not set up for currently

© NMISA 2010

Biodiesel specifications: SANS 1935

Property	Measurement Unit	SA	NMISA
Ester concentration	% mass fraction, (min)	96.5	•
Density @ 15C	Kg/dm3	860-900	•*
Viscosity @ 40C	Mm2/s	3.5-5.0	•*
Carbon residue (on 10% distillation residue)	% mass fraction, (max)	0.3	○
Iodine	g Iodine / g FAME	140	•
Flashpoint	C, min	120	○
Cetane #	min	51	x
Suphur	mg/kg (max)	10	•
Oxidation stability at 110C	Hours min	6	○
water	% mass fraction, (max)	0.05	•
Acid value	Mg KOH/g	0.5	•
Copper strip corrosion 3 h at 50C	Rating ISO 2160	Class 1	•

• No capability

•*developing accredited methods

• NMISA can analyse

© NMISA 2010

Biodiesel specifications: SANS 1935

Property	Unit	SA	NMISA
Linolenic acid methyl ester	% mass fraction, (max)	12	•
Polyunsaturated (> = 4 double bonds) methyl esters	% mass fraction, (max)	1	•
Methanol content,	% mass fraction, (max)	0.2	•
Monoglyceride content	% mass fraction, (max)	0.8	•
Diglyceride content	% mass fraction, (max)	0.2	•
Triglyceride content	% mass fraction, (max)	0.2	•
Free glycerol	% mass fraction, (max)	0.02	•
Ester content	% mass fraction, (min)	95.5	•
Group I / II metals	mg/kg	5 each	•
CFPP	C	-4 (winter) 3 (summer)	x

• No capability

•*developing accredited methods

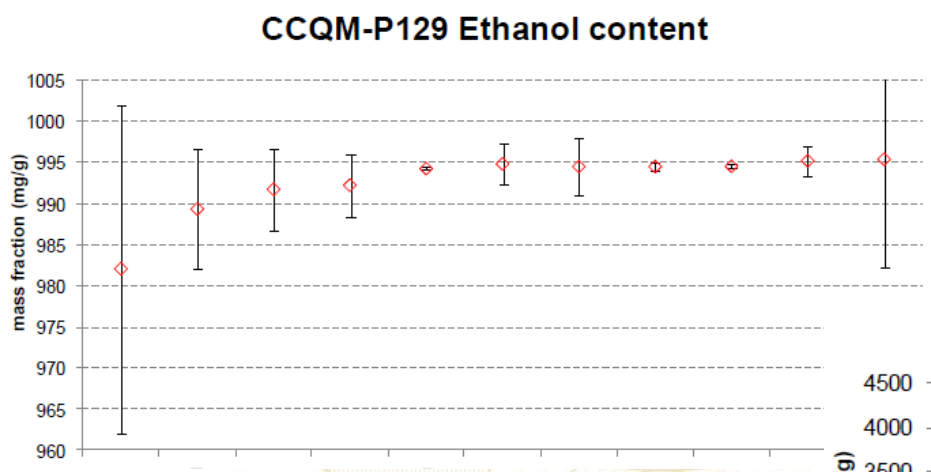
• NMISA can analyse

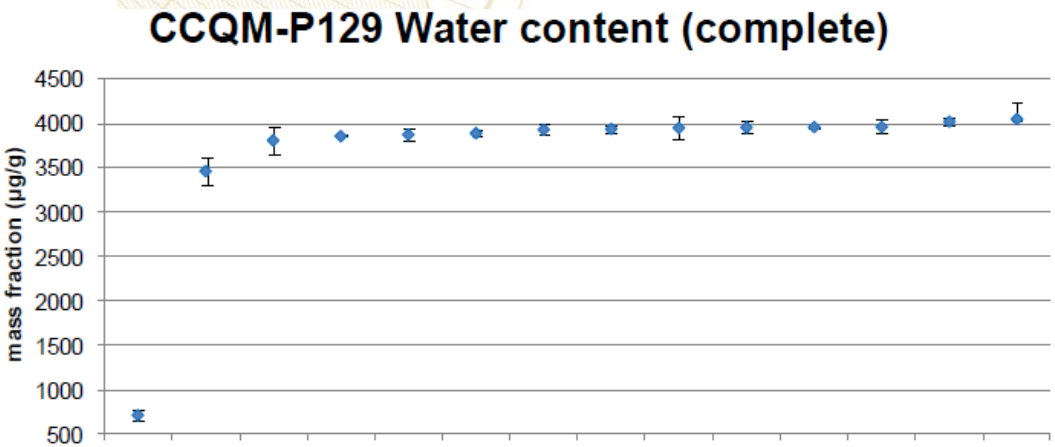
© NMISA 2010

NMISA Biofuel Comparisons

- The NMISA participated in two dedicated bio-ethanol international comparisons:

  CCQM-P129 and PEEARL bilateral





© NMISA 2010

The NMISA FAME project

- Fatty Acid Methyl Esters (FAMEs) analysis is important, both for the food industry and in the biofuels area, where these compounds form the major components
- In the biofuels market, the need to differentiate between *cis* and *trans* double bond isomers is not important as the naturally occurring FAMEs are almost exclusively *cis* double bonds
- Thus analysis of FAMEs in biodiesel produced from plant material can be undertaken using GC-FID or GC-MS
- This allows for the determination of fatty acids differing by carbon number and unsaturated FAME isomers differing by the number and position of the double bonds in the compound

© NMISA 2010

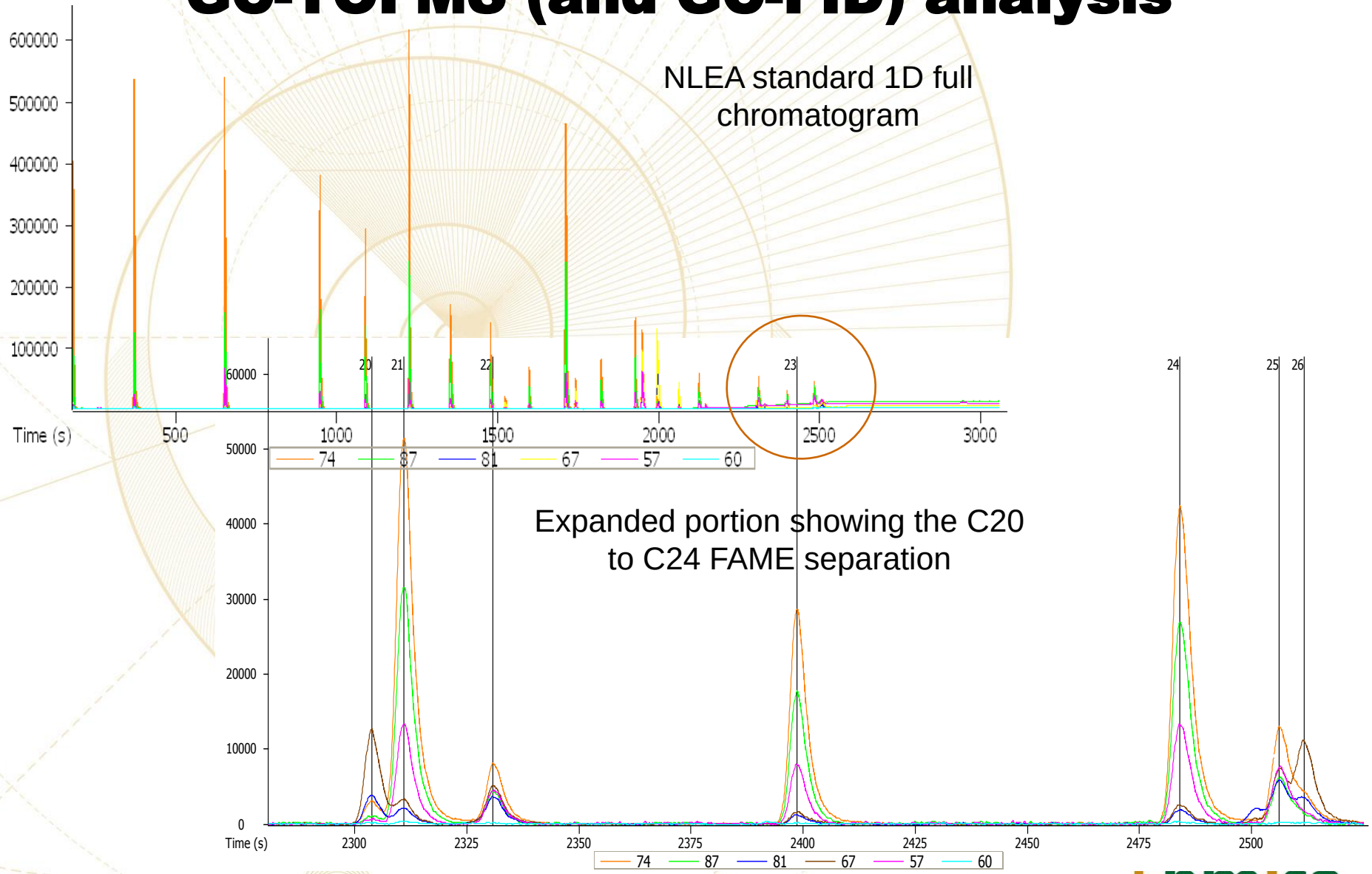
nmisa
National Metrology Institute of South Africa

Approach to FAME project

- The NMISA established a GCxGC-FID screening method for the analysis of a FAME standard
- Most laboratories do have access to GC-FID, if not GCxGC
- A GC-TOFMS method was also developed in order to identify the individual FAMEs in the specific standard
- Samples from Thailand (*Jatropha Curcas Linn* & Palm Oil) and SA were compared
- Results were quantified using the NLEA standard

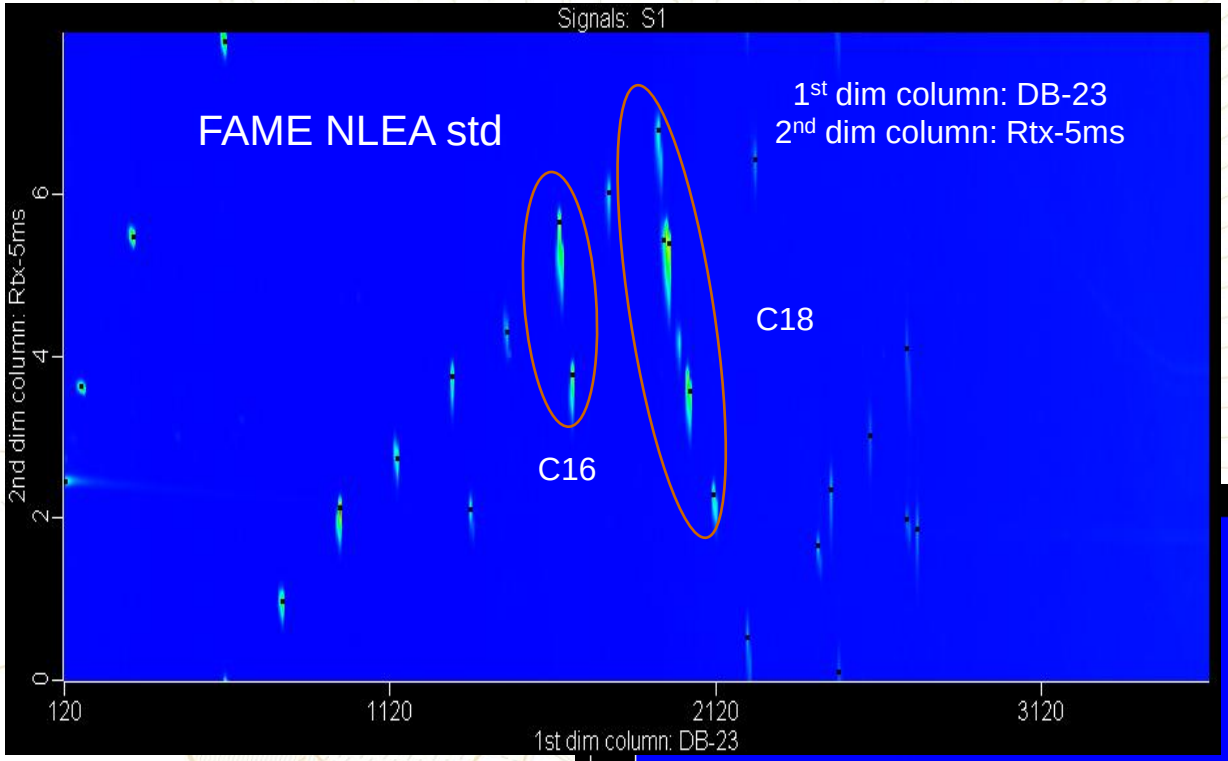
© NMISA 2010

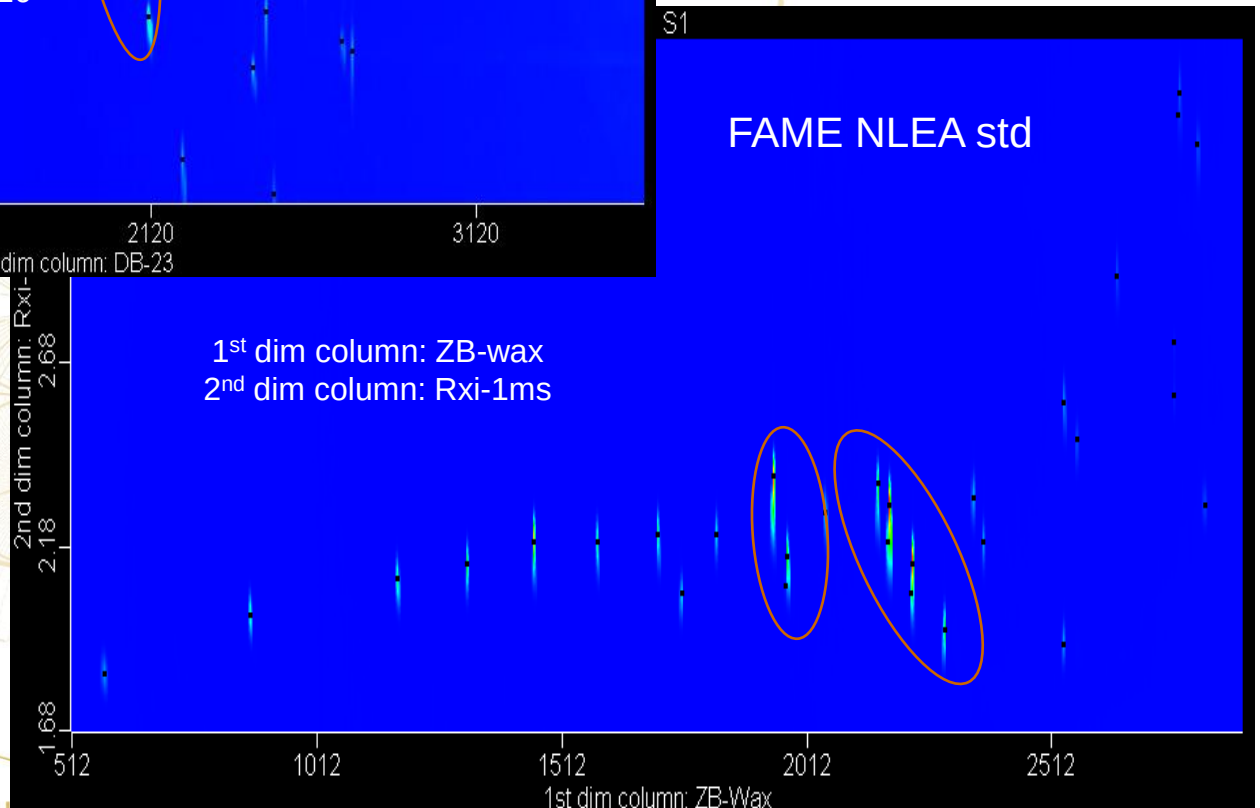
GC-TOFMS (and GC-FID) analysis



© NMISA 2010

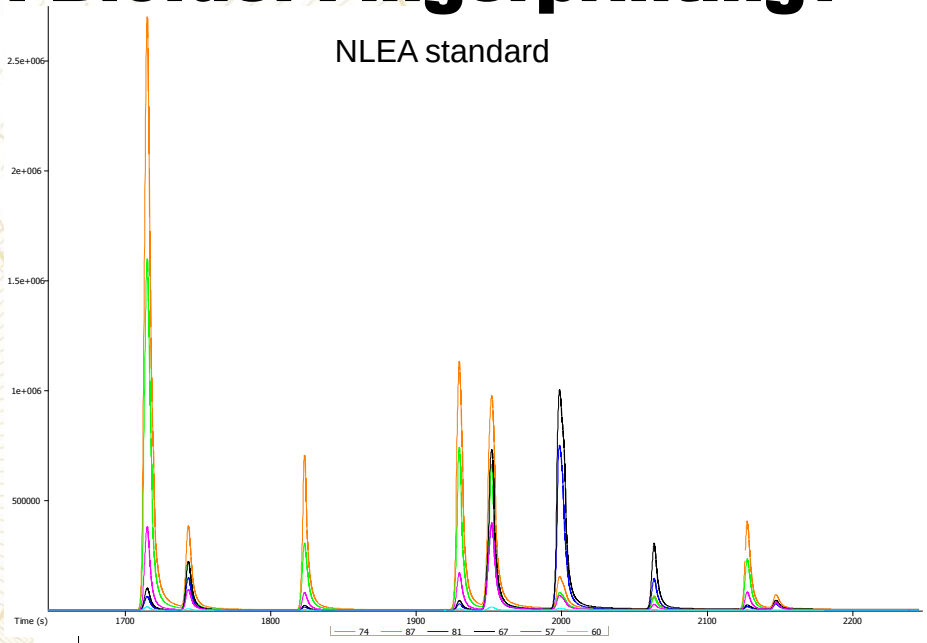
Biodiesel – separation science!

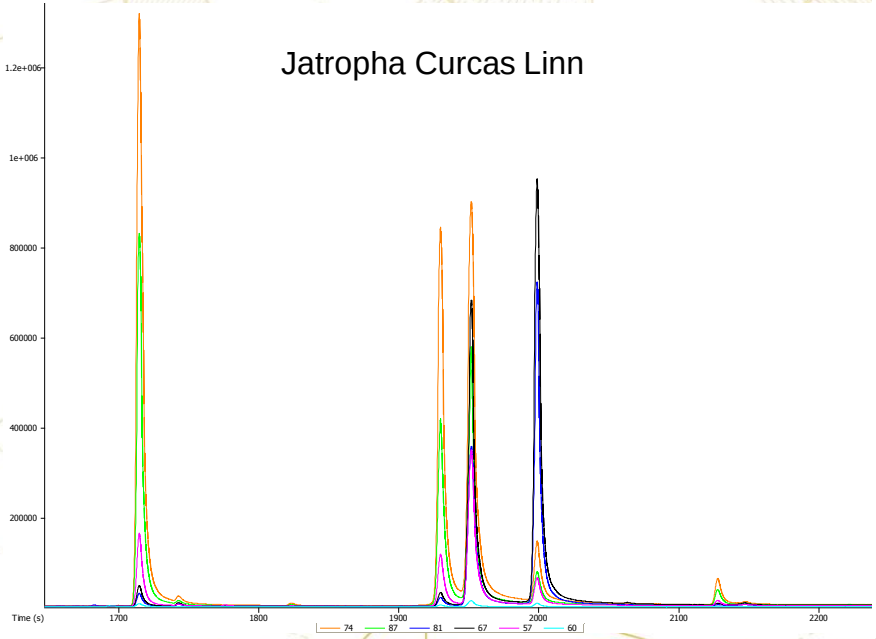


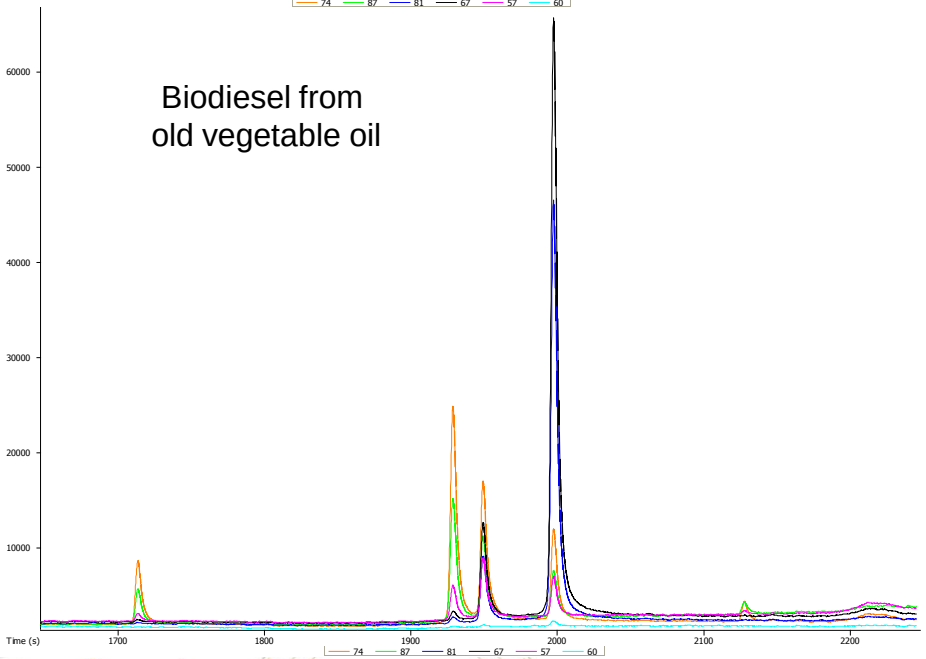


Tax incentives/rebates: Biofuel Fingerprinting?

- The NLEA standard was used to quantify the FAME compounds in the Thailand and SA biodiesel samples
- Below is an example of the C16 and C18 GC-TOFMS results obtained for two of the samples







Other new national standards in support of biofuels

Viscosity

- Established facility in 2009/10
- Viscometer constants vary from 0.002 mm^2/s^2 to 100 mm^2/s
- Kinematic viscosities from approximately 0,4 mm^2/s to 100 000 mm^2/s
- 20 °C, 60 °C and 100 °C
- International equivalence : CCM.V-K2.1 comparison
- Accreditation planned for end of 2011
- Dynamic viscosity in planning, or calculate with density

Density

- The NMISA calibrates hygrometers for fuel industry
- Solid and liquid density facility is being commissioned
- Will cover the densities specified in the SANS specifications

© NMISA 2010

nmisa
National Metrology Institute of South Africa

The Road Forward

- Ensure international alignment of SANS with other standards, while meeting national requirements
- Accessible, easy and affordable QC methods for especially small biofuel producers
- International comparison (all parameters) between NMIs to establish comparability and identify areas that require analytical support
- Range of CRMs – especially for biodiesel
- Improved communication between all stakeholders in SA

© NMISA 2010

nmisa
National Metrology Institute of South Africa

Cost-efficient biofuel testing

- Well defined and agreed parameters
- Traceable accurate measurement methods
- Fit-for-purpose methods
- Validated methods
- Certified reference materials

Measured once, accepted everywhere through metrological traceability and accredited testing facilities

© NMISA 2010

nmisa
National Metrology Institute of South Africa

NMISA is committed to the development of measurement methods and reference materials to ensure compliance of SA biofuels to international standards

sprins@nmisa.org bjdevos@nmisa.org

© NMISA 2010