Chemical Metrology

and its Impact on Industry and Quality of Life

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Secretary CIPM, President CCQM

SIM Senior Policy Makers Dialogue Forum

November 6, 2009 at INMETRO

Rio de Janeiro/Xerém, Brazil

Sustainable competitiveness and innovation

Growing metrological demand

- Global trade in commodities >12 trillon US$ (2005)
  - 80% affected by standards and regulation
  - compliance costs ~10% of production costs
- Global growth and spread of industrial and service activities
- Global increase of industrial and societal developments
- Requires innovative products and services
- Requires increasingly accurate measurements
- Requires comparable, traceable measurements

Sustainable competitiveness and innovation

Growing metrological demand

- Men travelling all around the globe
- Food products from everywhere
- Global spread of diseases
- Global environmental and climate issues
- Requires metrology in new areas, like
    - chemistry, bio technology
    - advanced materials, nano technology
    - information technology

Quality of life

Growing metrological demands

- food safety and nutrional content
- health care and protection
- environment, pollution control and climate change
- security and forensics
- anti doping
- “soft/perceptive” metrology (smell, taste, blends, color, glance, form,etc.)

Your dinner

- Cachaca
- Shrimp cocktail
- Smoked salmon
- Chicken tandoori
- Rice curry
- New York Steak
- Cheese platter
- Fruit cocktail
- Great Wall red wine
- Non-alcoholic drinks
- Tea or coffee
- Chocolate

Bon Appetit !

Are you sure ?

Why to bother?

Food scandals

- Growth hormones in beef
- BSE in beef
- Dioxine and melamine in milk
- Salmonella in eggs
- Heavy metals in rice and wine
- Glycol in wine, diesel oil in olive oil
- Toxic residues in fish, oyster, shrimp (from all waste water)
- Pesticides in fruits and honey
- Nitrates and nitrites

We have to analyse/measure !

Are we really doing the right job ?

- Do we know what we really like/intend to measure
- Did we clearly define the measurand
- Are we really measuring what we intend to measure
- Are our measurement results comparable, traceable
- Do we use validated methods and procedures
- Do we use the right reference measurement standards; Certified Reference Materials
- Do we know the accuracy/measurement uncertainty
- Does a reliable (accredited) measurement and testing infrastructure exist

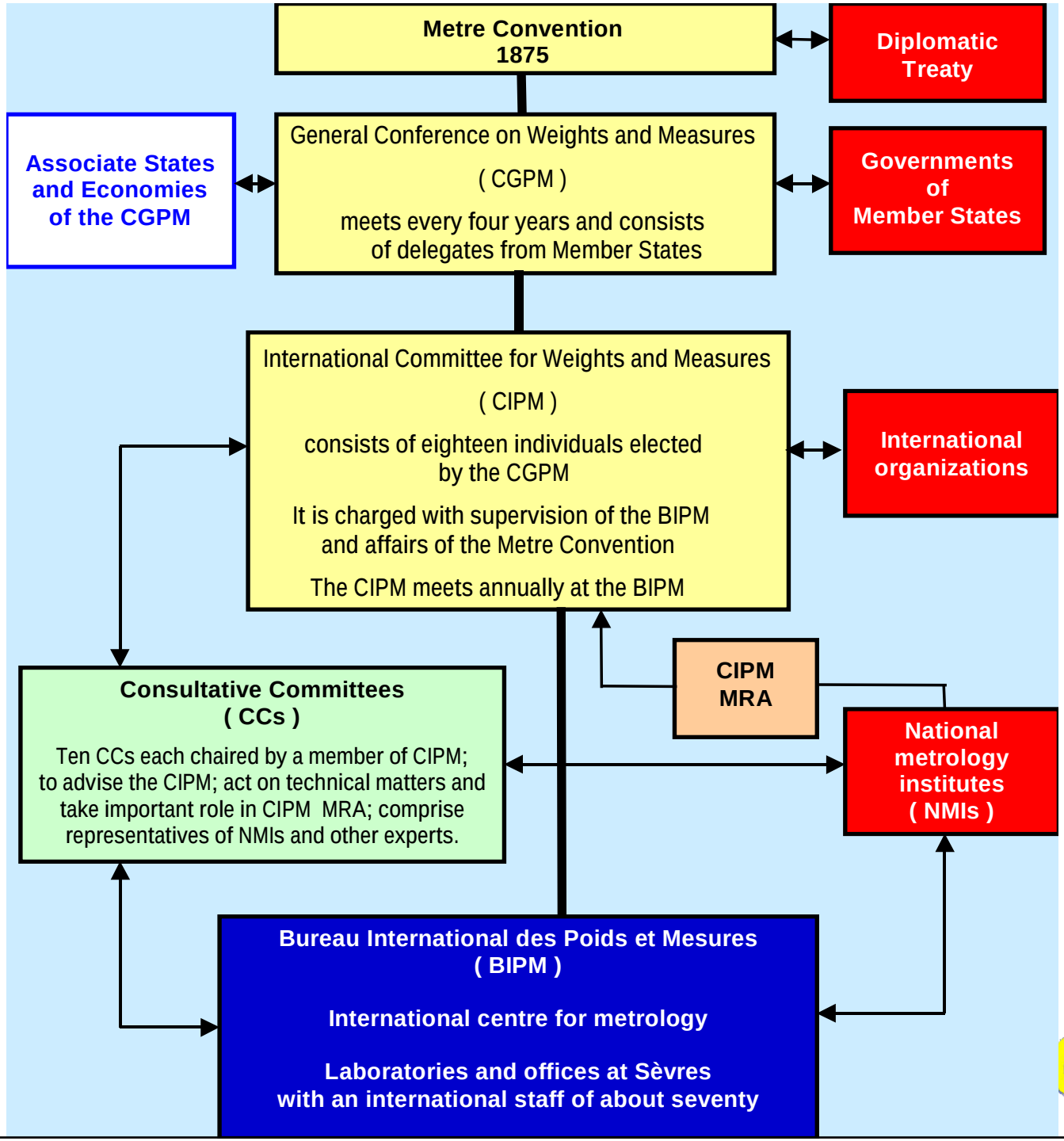
If not, we have a problem !

Metrological traceability

property of a **measurement result** whereby the result can be related to a reference **through an unbroken chain** of calibrations, each contributing to the **measurement uncertainty** JCGM 200:2008 (VIM 3)

Traceability to the SI, or if not (yet) possible to another internationally agreed reference (hardness, pH, WHO International Units)

"Once measured, everywhere accepted" requires Comparability through Traceability

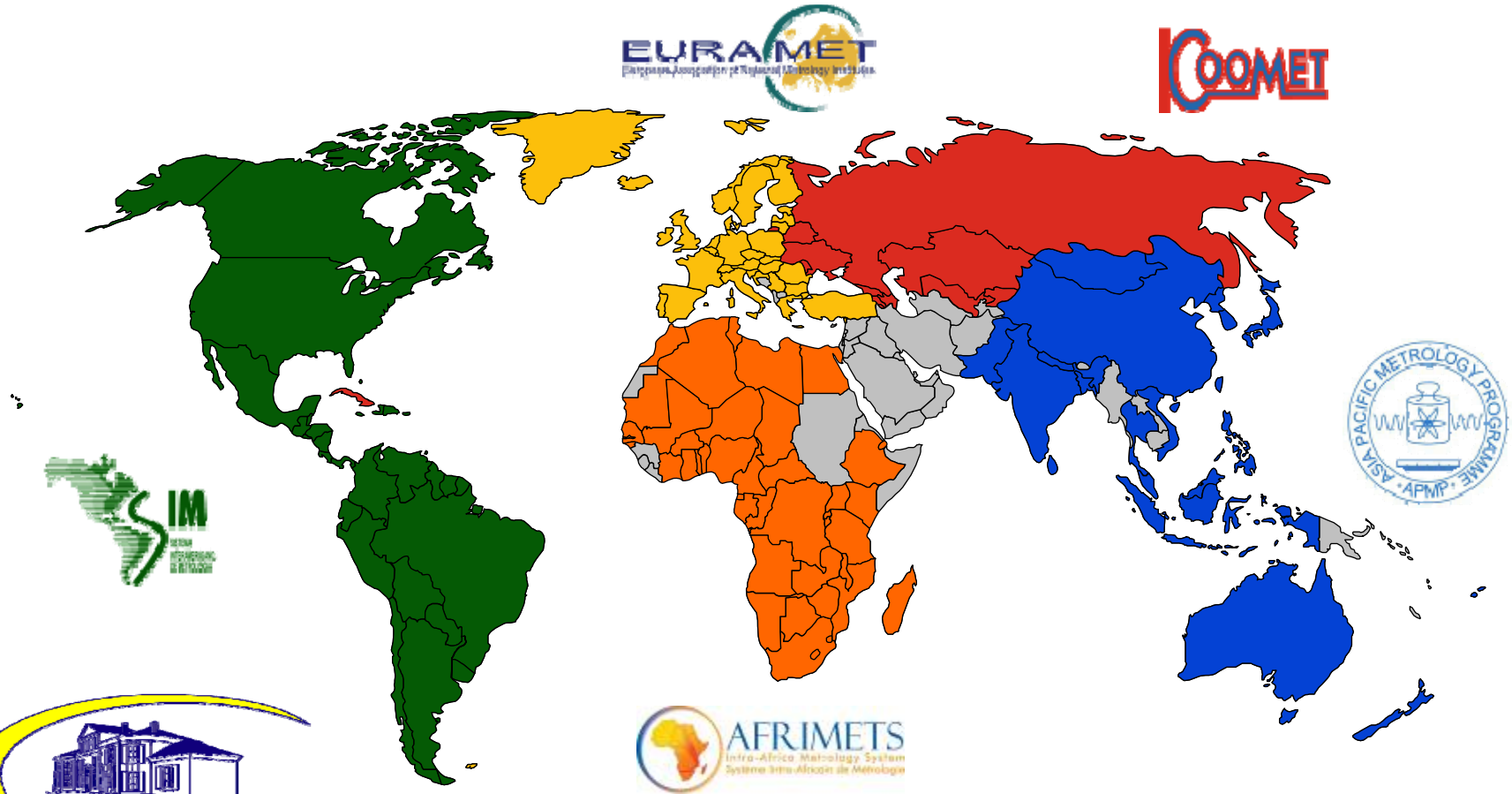

Metre Convention
1875
Diplomatic
Treaty
Associate States
and Economies
of the CGPM
General Conference on Weights and Measures
(CGPM)
meets every four years and consists
of delegates from Member States
Governments
of
Member States
International Committee for Weights and Measures
(CIPM)
consists of eighteen individuals elected
by the CGPM
It is charged with supervision of the BIPM
and affairs of the Metre Convention
The CIPM meets annually at the BIPM
International
organizations
CIPM
MRA
Consultative Committees
(CCs)
Ten CCs each chaired by a member of CIPM;
to advise the CIPM; act on technical matters and
take important role in CIPM MRA; comprise
representatives of NMIs and other experts.
National
metrology
institutes
(NMIs)
Bureau International des Poids et Mesures
(BIPM)
International centre for metrology
Laboratories and offices at Sèvres
with an international staff of about seventy
BIPM

Member States and Associate Members

Per 17 september 2009

- Members States
  - 53 States (including recent Members States Kazakhstan and Croatia; per Jan. 2010 Kenya)
- Associate Members
  - 28 Associates of the CGPM representing
    - 38 countries and economies (including recent Associates Paraguay and Ghana)

Regional Metrology Organizations



Planned GULFMET and AFRIMETS sub-region NEWMET

BIPM

Consultative Committee for Amount of Substance CCQM - Metrology in Chemistry

Aim and tasks

- To establish **worldwide comparability**, through
- **Traceability to SI**, or if not (yet) possible to other internationally agreed references, by
- Development of **primary and other methods of higher order**, databases and
- **Primary (pure) reference materials** and
- **Validation** of traceable methods/measurement uncertainty
- To organize **Pilot study comparisons** and **Key Comparisons**
- **To liaise with all stakeholders**
- To contribute to the establishment of a globally recognized system of national measurement standards and facilities and the implementation of the **CIPM MRA**
- To advise the CIPM and the BIPM on metrology in chemistry

Inter-governmental organisations and other international bodies

Organisations with clear interest in metrology

- MoU with WHO, WMO, ILAC, JCTLM (IFCC and ILAC)
- IAEA, Codex Alimentarius Commission/IAM, CIE, IAU, ITU, ICRU, IUGG, IUPAC, IUPAP, WADA, Pharmacopeia, IAFSI/ENFSI, a.o.
- Approaches to WTO (Technical Barriers to Trade Committee) and World Customs Organisation (WCO)
- CIPM Consultative Committees open for observership from and cooperation with these inter-governmental and international organisations

CCQM – Metrology in Chemistry

CCQM Working Groups

• Key Comparisons and CMC Quality	NMIA	L. Mackay
• Organic Analysis	NIST	W. May
• Inorganic Analysis	LGC	M. Sargent
• Gas Analysis	NPL	M. Milton
• Electro-chemical Analysis	SMU	M. Mariassy
• Surface Analysis	BAM	W. Unger
• Bio-Analysis	LGC	H. Parkes

• 3 ad hoc WGs (KCRV, EET, redefinition SI)

Priority areas in the USA (NIST)

- Energy (biofuels, hydrogen fuel, solar, wind)
- Environment and climate change (WMO GAW)
- Healthcare
  - Diagnostics (EU IVD directive driven)
  - Therapeutic (WHO)
  - Pharmaceuticals (USP, a.o.)
- Food safety and nutritional value (FDA, EU, etc.)
- Homeland security

Priority areas in the EU (EURAMET)

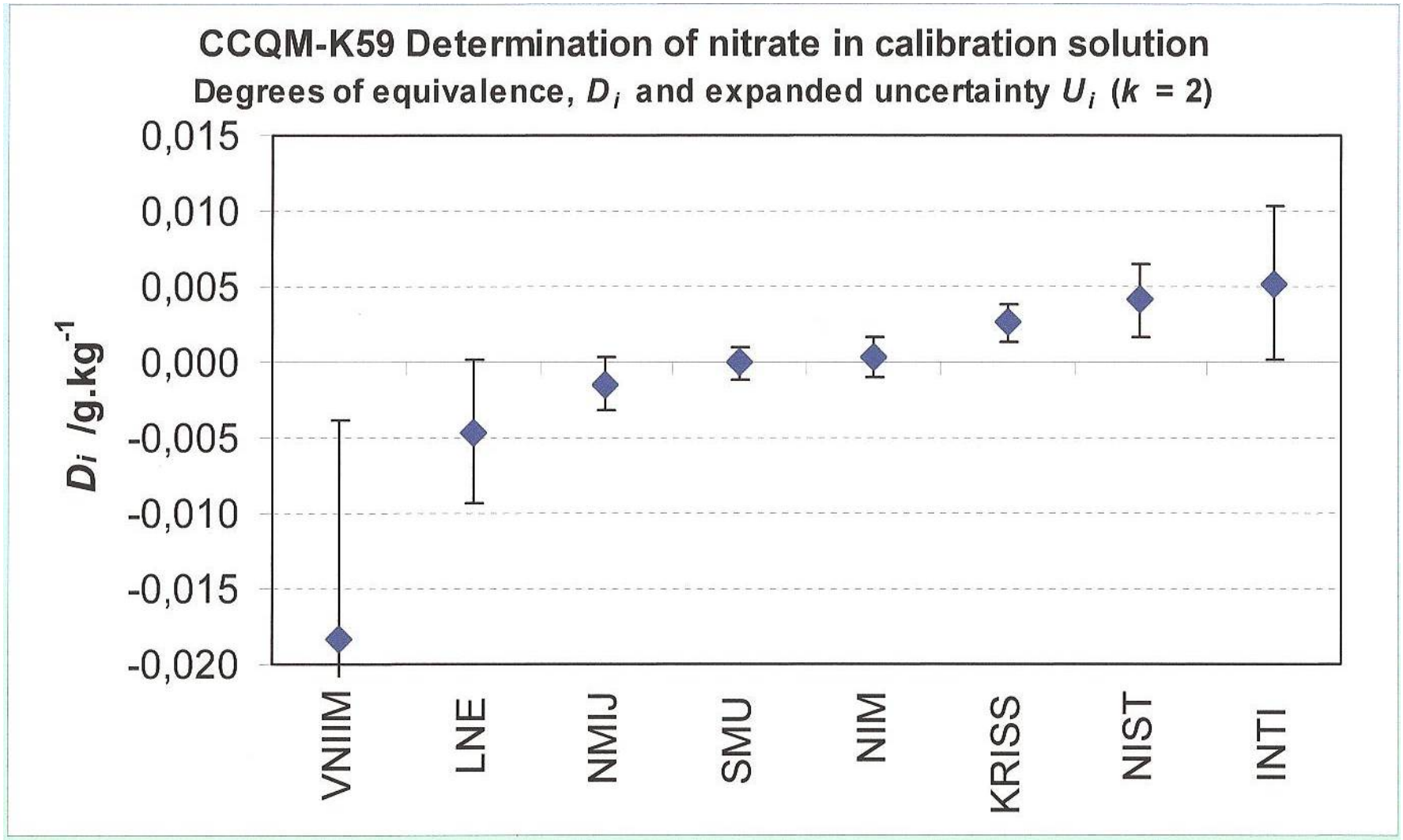
European Metrology Research Programme (FP 7 and art.169)

- Health care
  - Virtual human (modelling system biology)
  - Reference measurements and materials (JCTLM)
  - Quantitative diagnostics (imaging, microscopy, etc.)
  - Diagnostic and therapeutic instrumentation (NMR, ultrasound, etc.)
- Energy
  - New and renewable energy resources
  - Conventional energy system
  - Smart energy networks

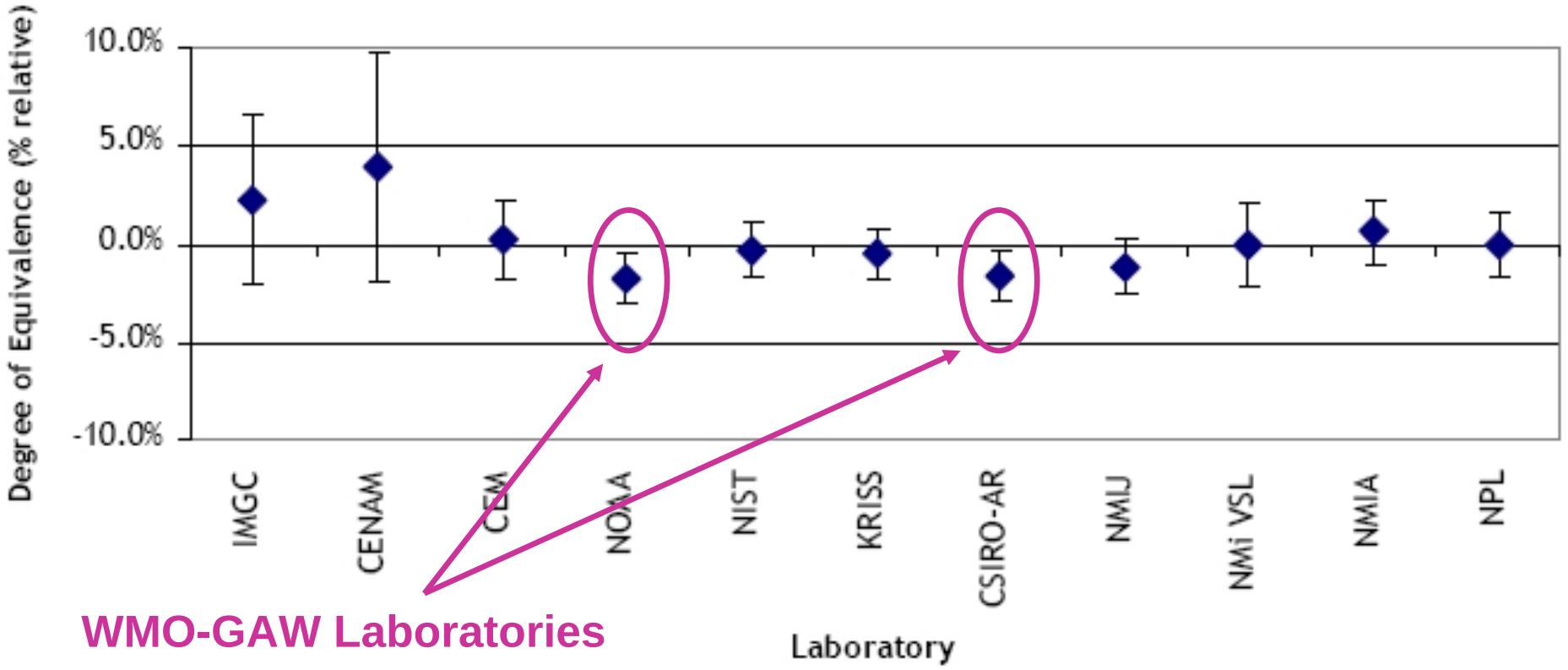
Priority areas in the EU (EURAMET)

European Metrology Research Programme (FP 7 and art.169)

- Environment and climate change
  - Detecting change and monitoring climate
  - Flow and concentration of substances under regulation
  - Carbon dioxide sequestration
  - Environmental noise
- Fundamental metrology
  - Nanotechnology
  - Security related metrology


CCQM-K59 Determination of nitrate in calibration solution
Degrees of equivalence, D_i and expanded uncertainty U_i (k = 2)
D_i /g.kg^{-1}
0,015
0,010
0,005
0,000
-0,005
-0,010
-0,015
-0,020
VNIIM
LNE
NMIJ
SMU
NIM
KRISS
NIST
INTI

CCQM-P41, Methane 1.8 µmol/mol (2003)



WMO-GAW Laboratories

1.7 % difference from gravimetric value

WMO scale is being revised

CCQM-K43.1As, Hg and Me-Hg in marine fish (swordfish), year 2007

Mercurio

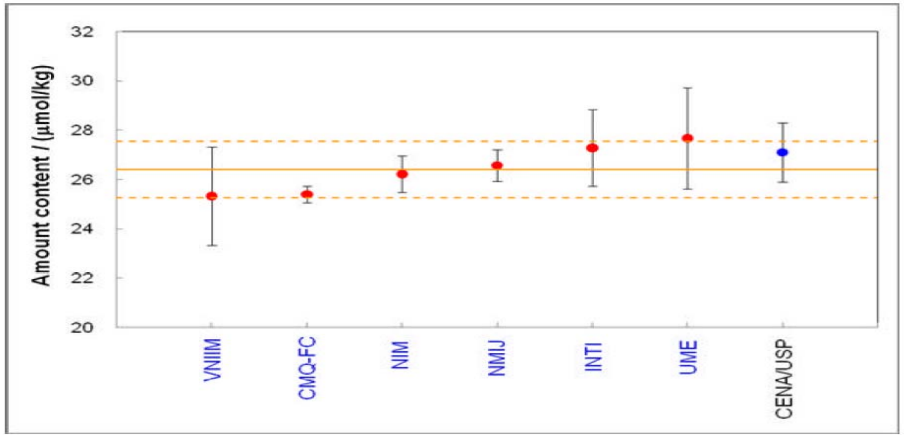


Figure 2 CCQM-K43.1: Hg in marine fish (swordfish), (The results of CMQ-FC: *k=2.18*)

Arsenico

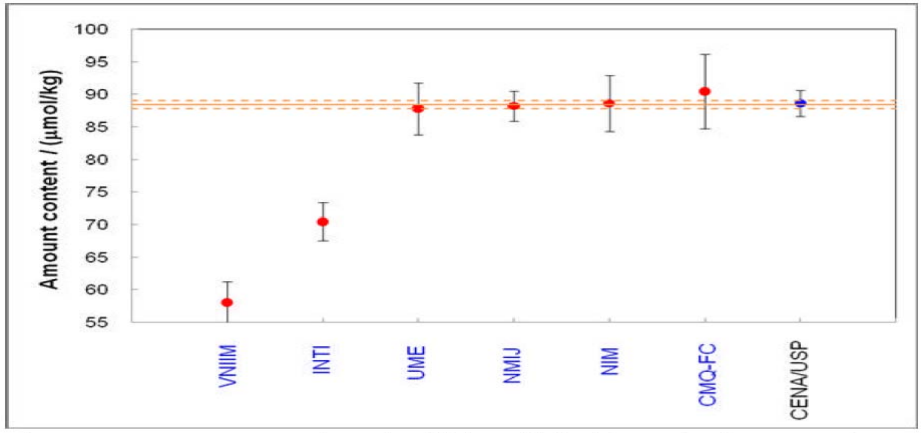


Figure 1 CCQM-K43.1: As in marine fish (swordfish), (The results of CMQ-FC: *k=1.96*)

CCQM-P12.1 Cu, Fe, Pb and Cd in wine, year 2006

Hierro

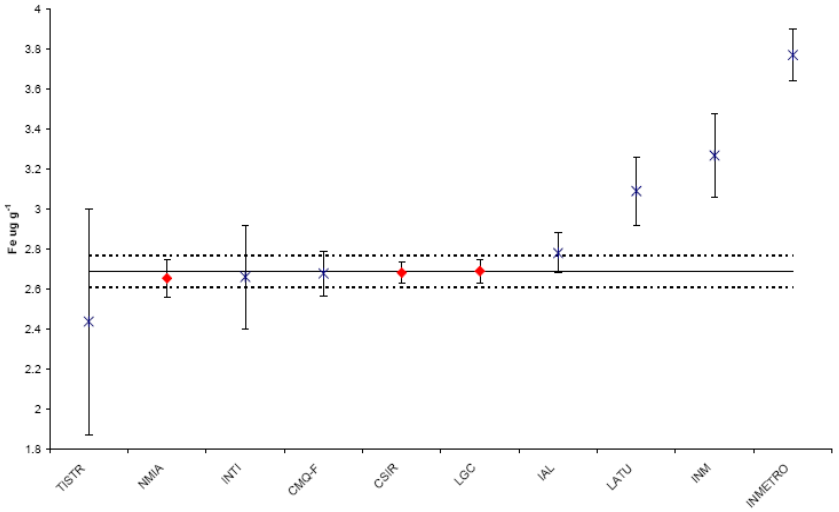


Figure 2: CCQM-P12.1 participants' measurement results for iron

The horizontal lines represent the KCRV and associated uncertainty. Solid, red diamonds represent results obtained using IDMS.

Cobre

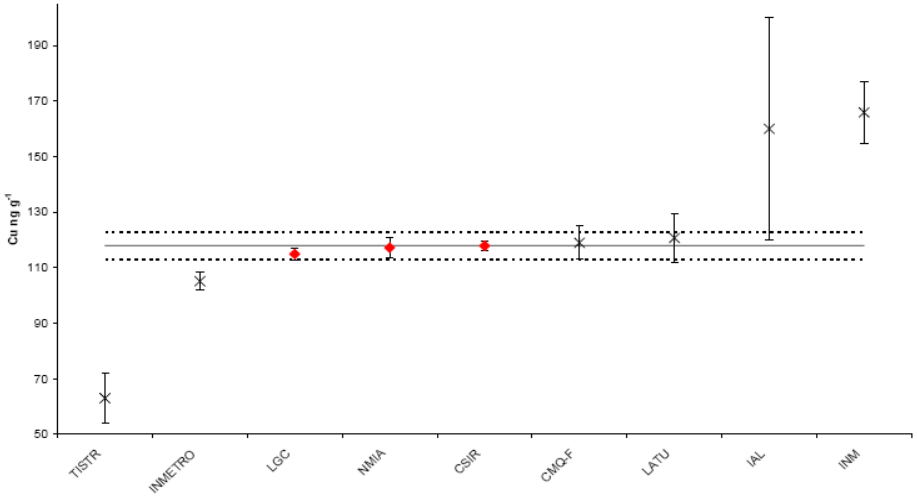


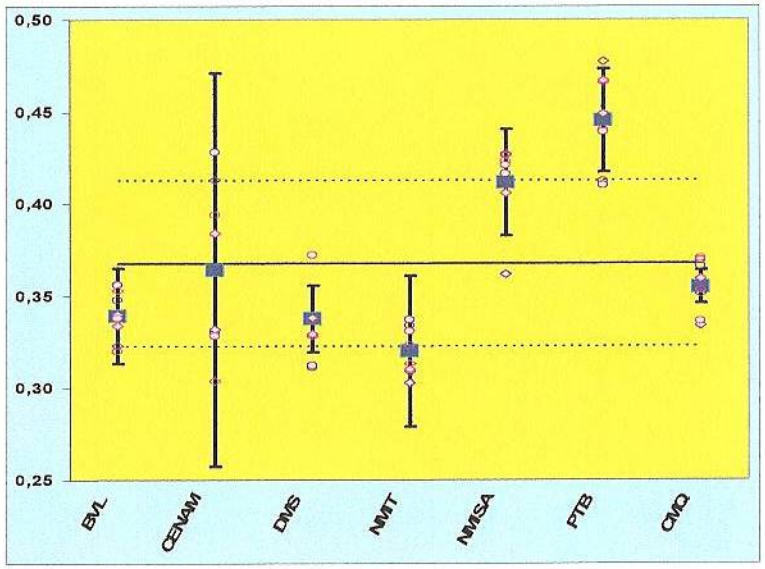
Figure 3: CCQM-P12.1 participants' measurement results for copper

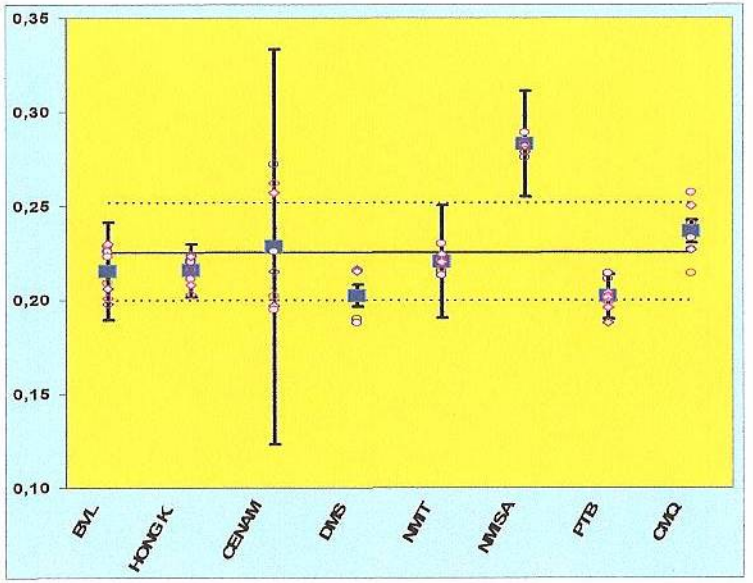
The horizontal lines represent the KCRV and associated uncertainty. Solid, red diamonds represent results obtained using IDMS.

Continuation of discussions from Oct 2007 OAWG meeting

- **CCQM-P90, Chloramphenicol in milk** (Coordinators: BVL, PTB)
  - Draft 1 Summary Report distributed to OAWG Mar 2008

Overview and Participant presentations made and results discussed at Oct 2007 Meeting

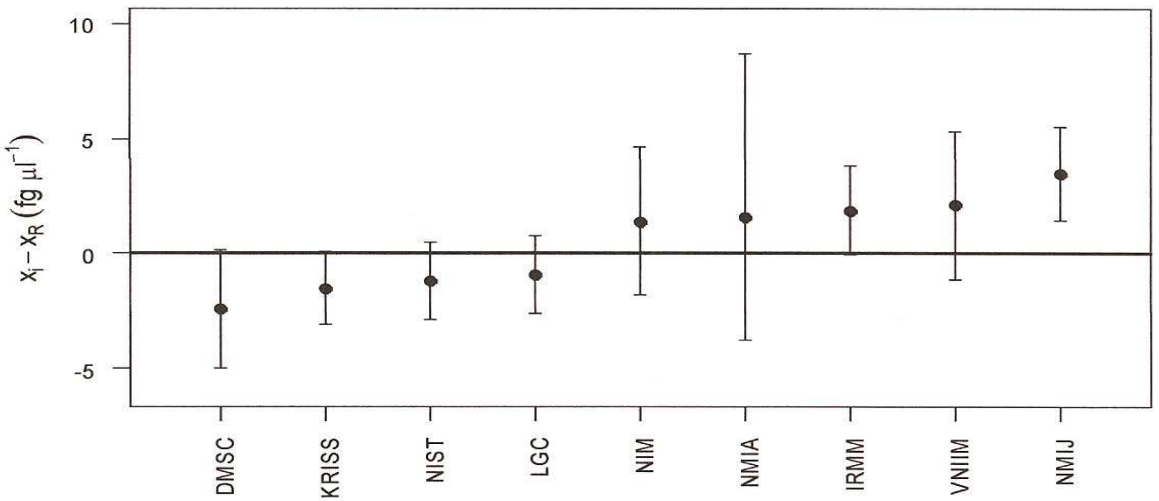




CCQM-K61

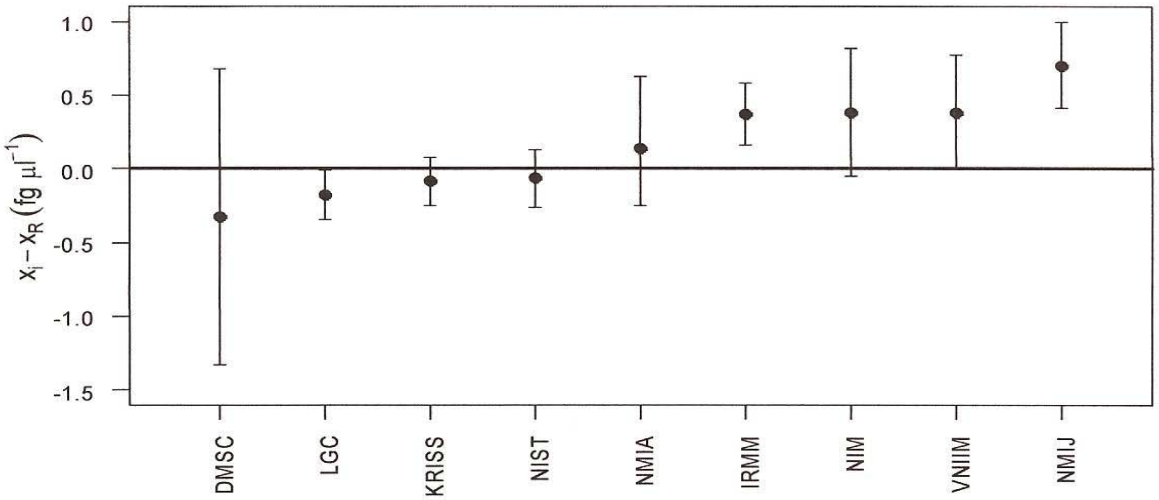
Quantitation of a linearised plasmid DNA

Figure 3: Degrees of equivalence - Unknown 1



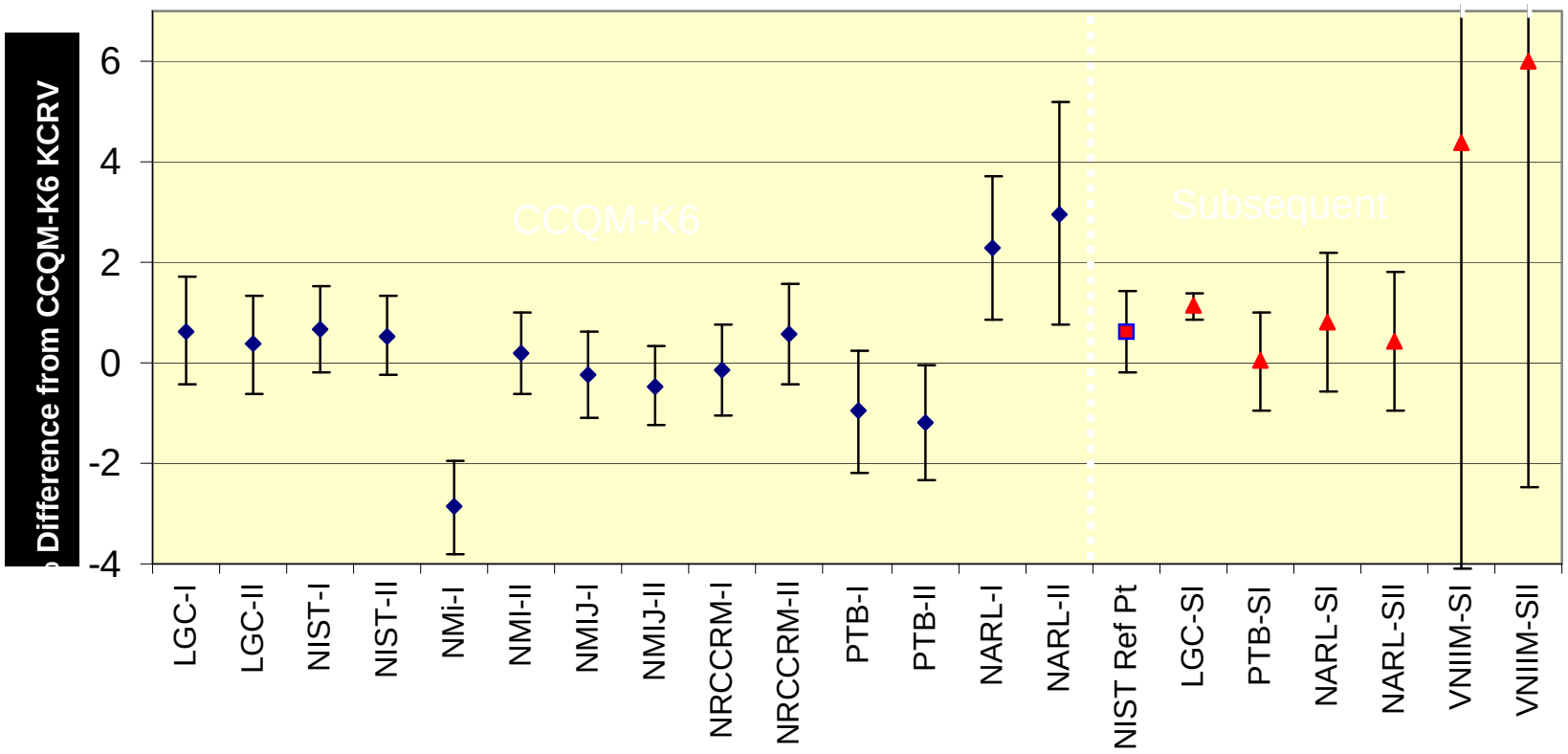
The graph shows the degrees of equivalence between participant results and KCRV. Error bars show uncertainties at k=2 or (for NMIA) the same level of confidence assuming a lognormal distribution.

Figure 4: Degrees of equivalence - Unknown 2



The graph shows the degrees of equivalence between participant results and KCRV. Error bars show uncertainties at k=2 or (for NMIA) the same level of confidence assuming a lognormal distribution.

CCQM-K6 and Subsequent Study - Relative Results



K6 cholesterol in serum results are plotted as % differences from KCRVs

Subsequent results are plotted relative to NIST results in K6S and are offset by average (NIST-KCRV) result from K6 (NIST Ref Pt)

NIST
National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

Joint Committee on Traceability in Laboratory Medicine - JCTLM

Principal promotors

- CIPM/BIPM
- IFCC
- ILAC

Supported by

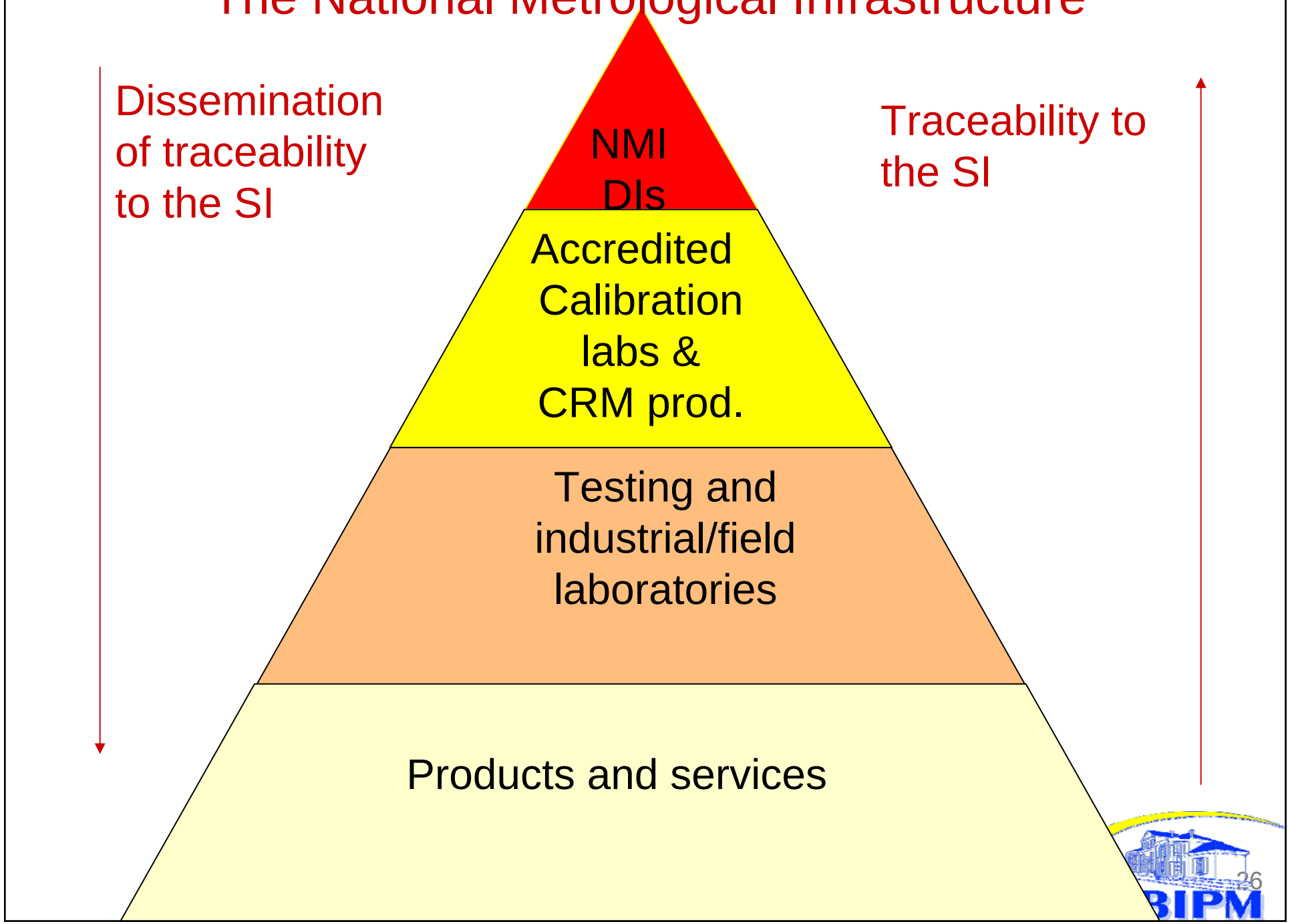
- WHO
- Regulators (FDA, EC, Japan)
- CRM producers (NIST, IRMM, a.o.)
- Reference laboratories (CDC, DGKS, etc.)
- PT and QA organisations (CAP, EQA, etc)
- Written Standards (NCCLS, JCCLS, ISO)
- IVD industry (ADVAMED, EDMA, JARC)

JCTLM WG 1 Measurand/Analyte-Based Review Teams

Coagulation Factors	**Elaine Gray**, **NIBSC,** United Kingdom
Drugs	**Andre Henrion, PTB,** Germany
Electrolytes/Blood Gases	**Gerhard schumann, Med. School,** Germany
Enzymes	**Mauro Panteghini**, **University of Milan**, Italy
Metabolites/Substrates	**Michael Welch**, **NIST**, United States
Nucleic Acids	**Helen Parkes, LGC,** United Kingdom
Non-Peptide Hormones	**Heinz Schimmel**, **IRMM,** European Union
Proteins	**David Bunk, NIST,** United States
Blood Groupings	**Susan Thorpe**, **NIBSC,** United Kingdom
Microbial Serology	**Morag Ferguson**, **NIBSC,** United Kingdom
Vitamins	**Katherine Sharpless, NIST**, United States
Non-electrolyte Metals	**Lee Yu, NIST,** United States
Blood cell counting	**Keiji Fujimoto, Sysmex Corp.,** United States
Quality System	**Craig M Jackson**, **HDC,** United States

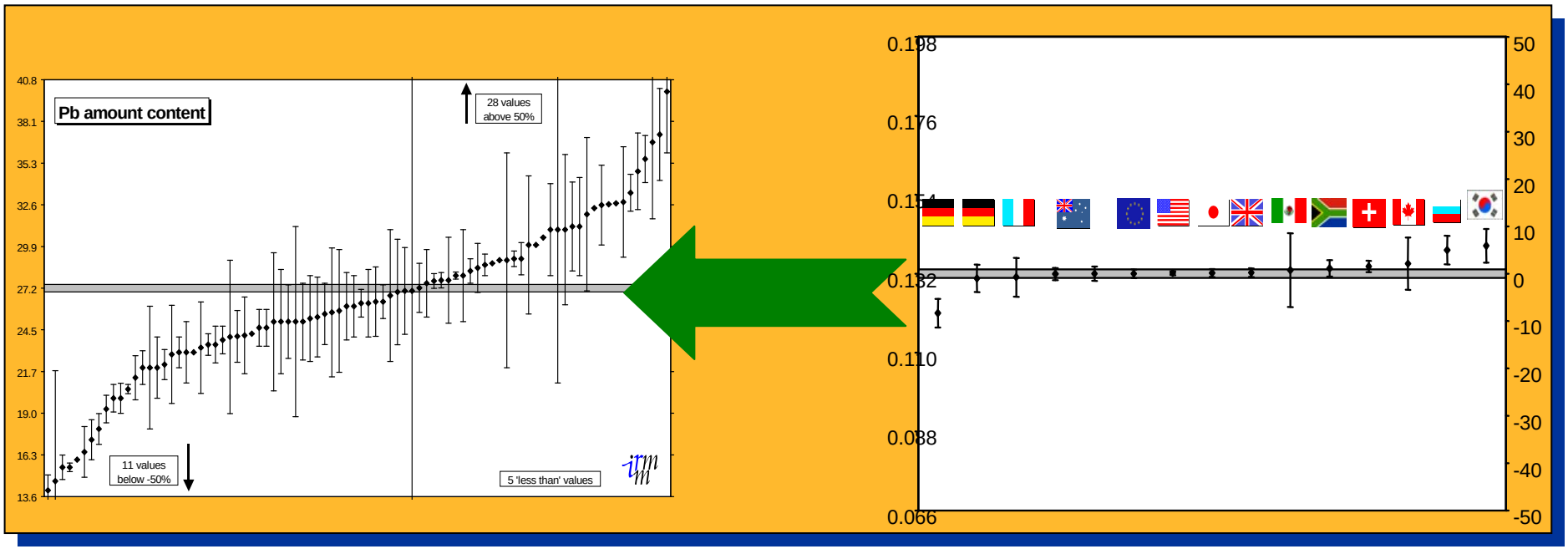
Review Teams established with worldwide representation from Laboratory Accreditation Organizations, National Metrology Institutes, Professional Societies, and IVD Industry in order to facilitate a fair and transparent review process.

The National Metrological Infrastructure



OIVV – CCQM comparison Pb in wine coordinated by the IRMM (IMEP programme)

OIVV likes to use assigned reference values

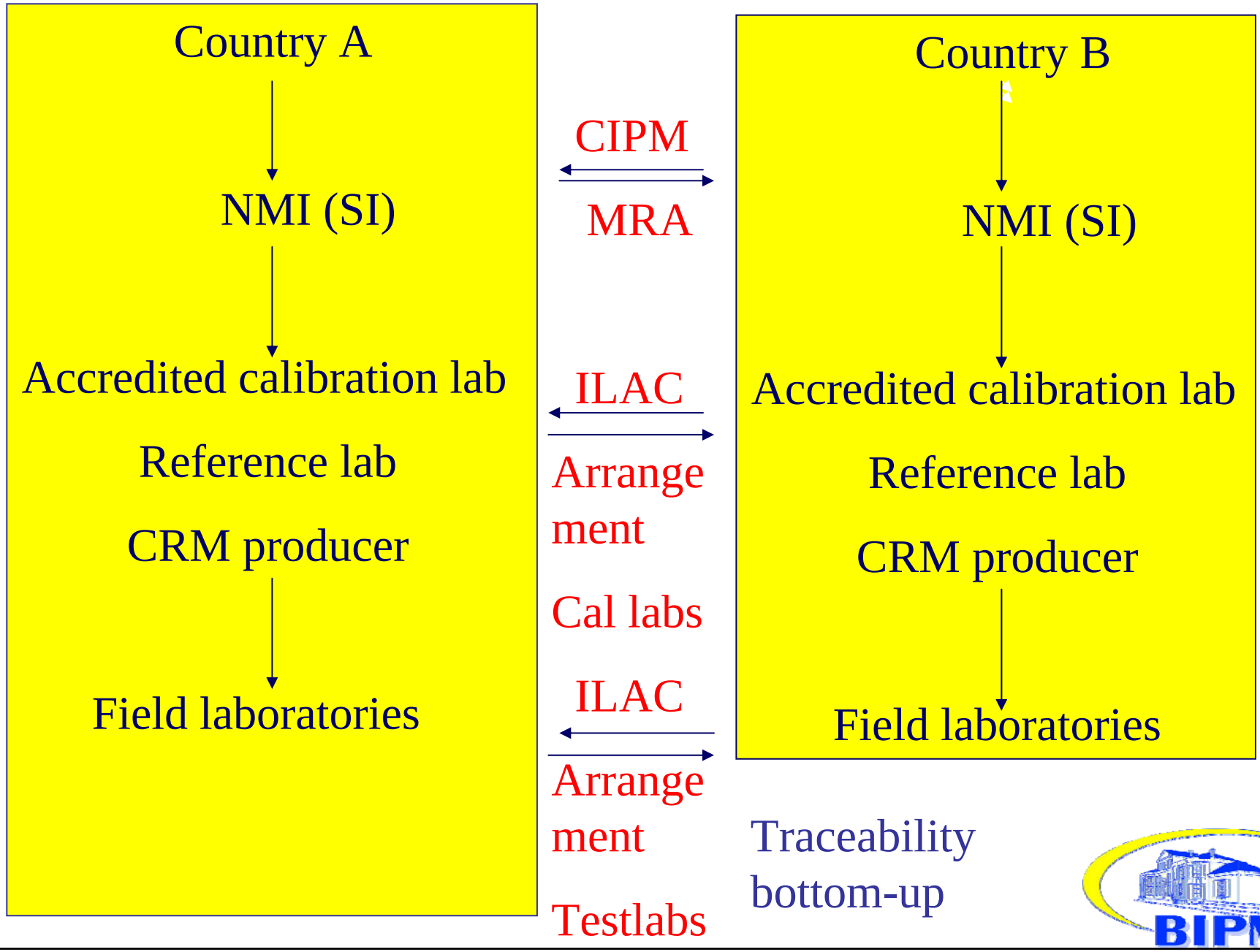


IMEP-16 CCQM-P12

Services to be delivered by National Metrology Institutes and other Designated Institutes

- Calibration and capability to assign values to samples
- Certified Reference Materials (production, certification)
- Reference value assignment of Proficiency Testing samples (own PT schemes and/or third party PT schemes)
- Validation of measurement methods/procedures
- Delivering traceability to industry and ILAC Arrangement accredited “calibration” and testing laboratories, CRM producers and PT providers
- Delivering traceability to sector specific reference laboratories (clinical and food reference laboratories)

Dissemination of traceability top-down

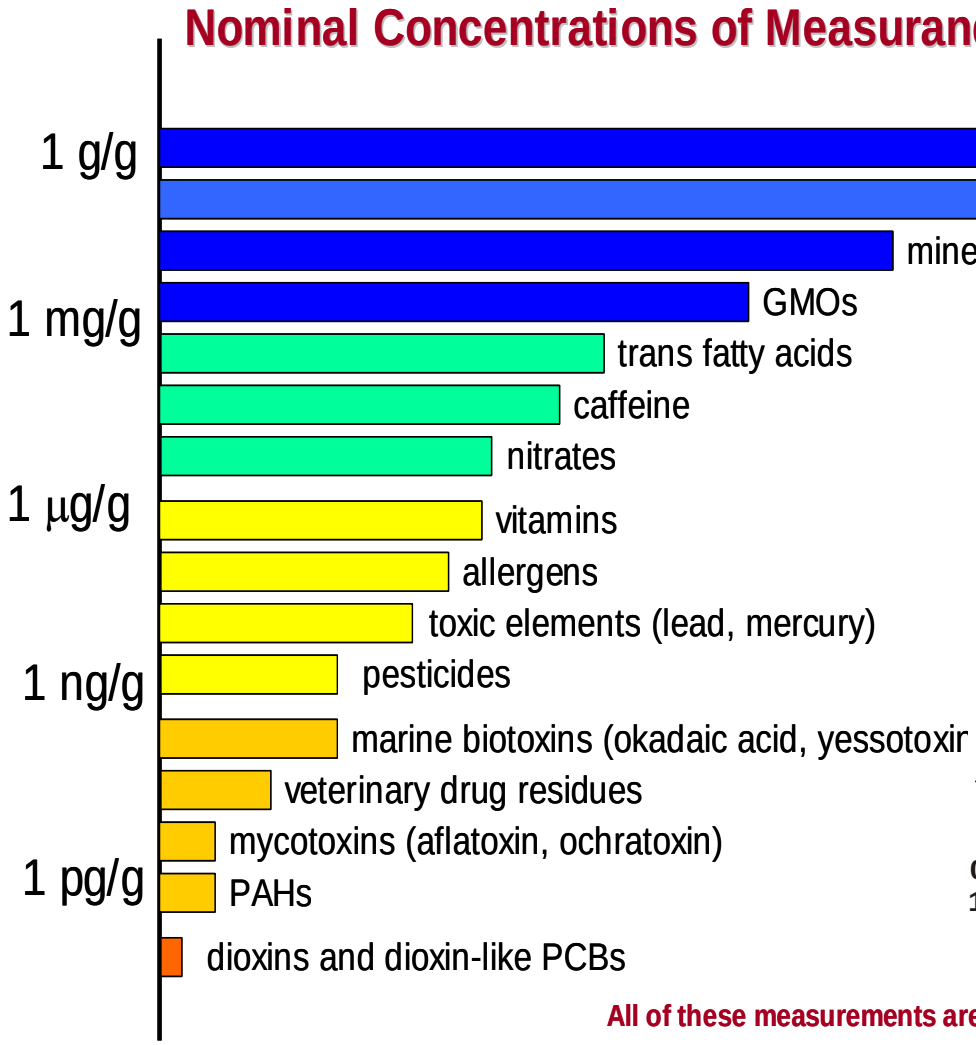


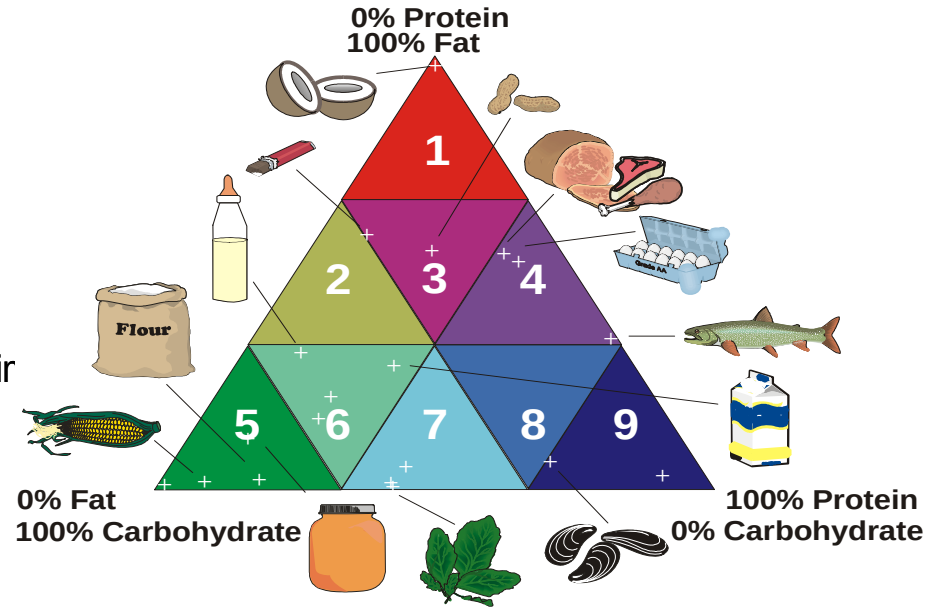
Trade, Health and Food Safety

Recent examples of temporary closure of markets due to the presence of residues

- Antibiotics in pork, Japan
- Antibiotics in meat, Korea
- Antibiotics in salmon, Japan
- Crystal violet in salmon, EU
- Leucomalachite green in salmon, Chinese Taipei
- Amphenicol in salmon, Canada
- Dioxin in pig meat, South Korea
- Melamine in milk
- Carbaryl in wine
- Cd in mussels
- Hg and nitrate in swordfish
- Patulin in apple and azinphos-methyl in pears and grapes
- Etc.

Examples of Regulated Classes of Chemicals in Foods





All of these measurements are impacted by legislation.

By courtesy of NIST

The CIPM Mutual Recognition Arrangement

- Mutual recognition of national measurement standards and of calibration and measurement certificates issued by NMI's (and other designated institutes) (covers >96% of world trade)
- Now signed by a large and increasing number of NMI's and other designated institutes (some 207), acting as NMI's for certain quantities and measurement ranges, of about 84 Member States and Associate Economies and 2 international organizations (IAEA and EU – JRC IRMM and JRC Ispra) (See Appendix A)

(soon also to be signed by the WMO)

The CIPM Mutual Recognition Arrangement

- Based on results of key-, supplementary- and bilateral comparisons (Appendix B)
- Quality system in place in conformity with ISO/IEC 17025 and ISO Guide 34
- Quality system assessment by international RMO review, accreditation and/or on-site peer review
- Regional and inter-regional review of claimed calibration and measurement capabilities

The CIPM Mutual Recognition Arrangement

- Published are the Calibration and Measurement Capabilities (CMCs), that are the services of the NMIs and other designated institutes, which are normally delivered to the customers

  * Analysing/measurement/calibration capabilities and/or

  * CRM's delivered/sold to customers

  * Delivering assigned reference values for PT schemes

  Some 21400 CMCs of which 4400 chemical, 3800 ionizing radiation, 13200 physical (Appendix C)
- Data Base – KCDB on www.bipm.org/kcdb

BIPM and the Mutual Recognition Arrangement

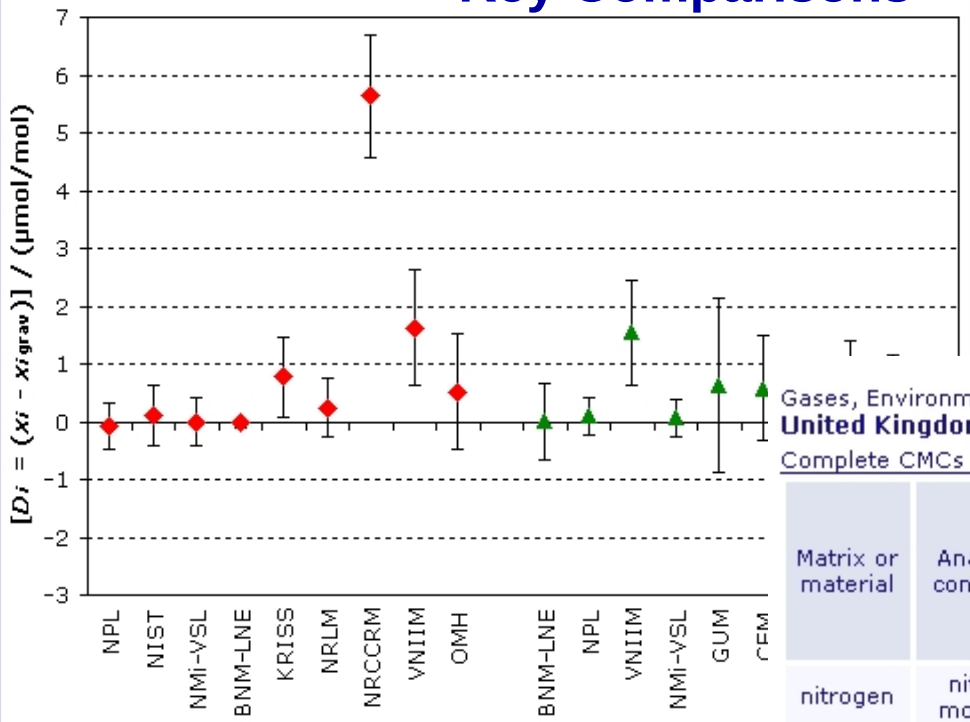
Laboratory individual measurements | Equivalence statements | Degrees of equivalence | **Graph(s) of equivalence**

CCQM-K1.c and EUROMET.QM-K1.c

MEASURAND : Amount-of-substance fraction of Nitrogen monoxide in Nitrogen
NOMINAL VALUE : 100 µmol/mol

Degrees of equivalence D_i and expanded uncertainty U_i (k = 2) expressed in µmol/mol

Key Comparisons



Red diamonds: participants in CCQM-K1.c
Green triangles: participants in EUROMET.QM-K1.c

Calibration and Measurement Capabilities

Gases, Environmental
United Kingdom, NPL (National Physical Laboratory)
Complete CMCs in Amount of Substance for Gases for United Kingdom (.pdf file)

Matrix or material	Analyte or component	Dissemination range of measurement capability		Range of certified values in reference materials	
		Amount-of-substance fraction in mmol/mol	Relative expanded uncertainty in %	Amount-of-substance fraction in mmol/mol	Relative expanded uncertainty in %
nitrogen	nitrogen monoxide	1 to 10	0.2 to 0.1	1 to 10	0.2 to 0.1

Mechanism(s) for measurement service delivery: PRGM, SGS and calibration
Uncertainty convention 2
Internal NMI service identifier: NPL/15

Mechanisms for measurement service delivery: CRMs

Biological fluids and materials, Blood serum

United States, NIST (National Institute of Standards and Technology)

Complete CMCs in Amount of Substance for Biological fluids and materials for United States (.pdf file)

Matrix or material	Analyte or component	Dissemination range of measurement capability		Range of certified values in reference materials	
		Amount-of-substance concentration in mmol/l	Relative expanded uncertainty in %	Amount-of-substance concentration in mmol/l	Relative expanded uncertainty in %
human serum	cholesterol	3 to 10	0.2 to 1.5	3.453 to 8.61	0.20 to 1.3

Mechanism(s) for measurement service delivery: SRM 1589a, SRM 1951a, SRM 1952a, SRM 909b, SRM 968c

Uncertainty convention 1. The expanded uncertainty for certified values in reference materials is given at a 95% level of confidence, but the coverage factor is not explicitly equal to 2

Internal NMI service identifier: NIST/8392169

High purity chemicals, Organic compounds

United States, NIST (National Institute of Standards and Technology)

Complete CMCs in Amount of Substance for High purity chemicals for United States (.pdf file)

Matrix or material	Analyte or component	Dissemination range of measurement capability		Range of certified values in reference materials	
		Mass fraction in %	Relative expanded uncertainty in %	Mass fraction in %	Absolute expanded uncertainty in %
high purity cholesterol	cholesterol	95 to 100	0.2 to 0.1	99.8	0.1

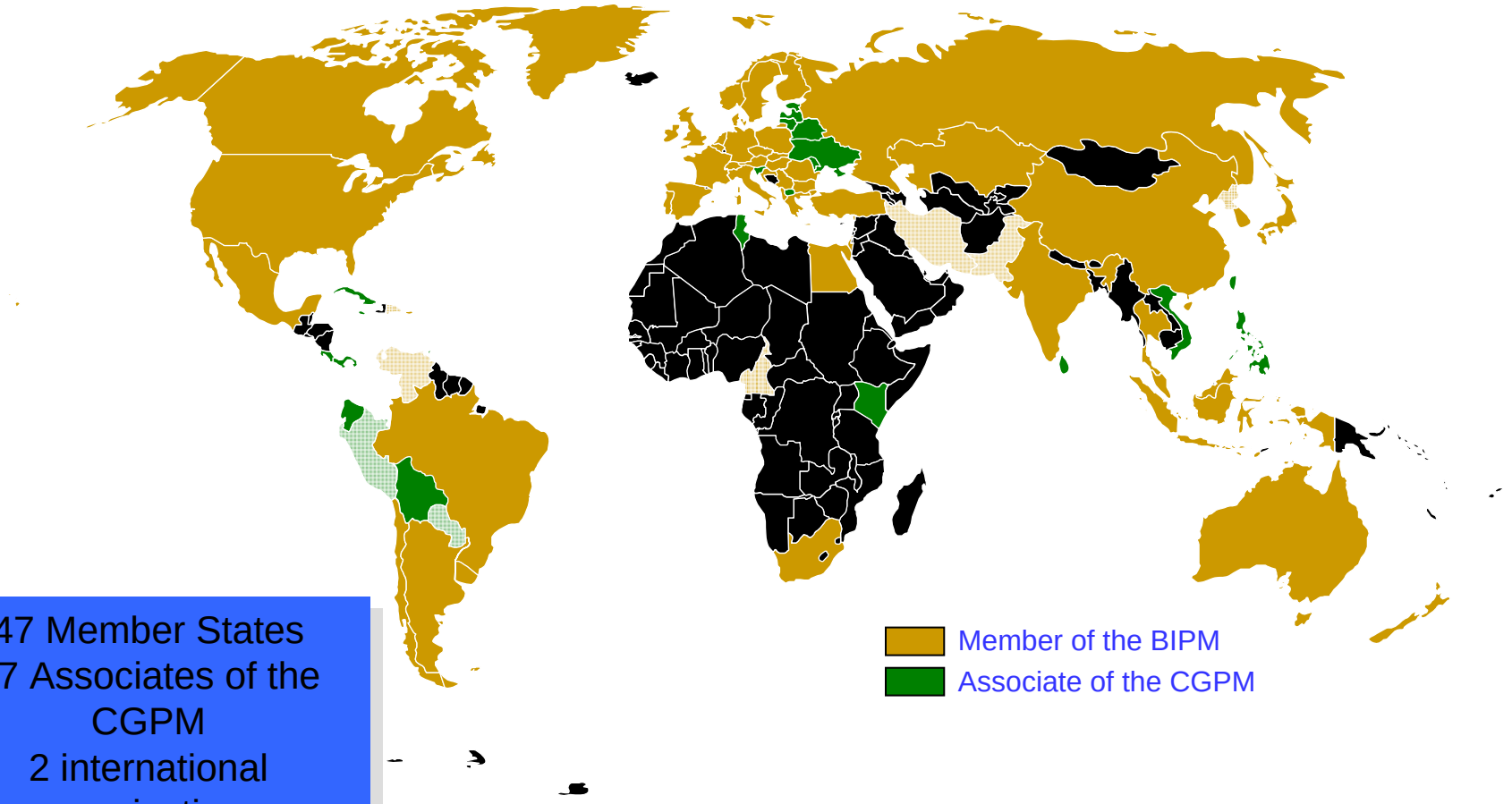
Mechanism(s) for measurement service delivery: SRM 911c

Approved on 24 June 2008.

Uncertainty convention 2

Internal NMI service identifier: NIST/8392005

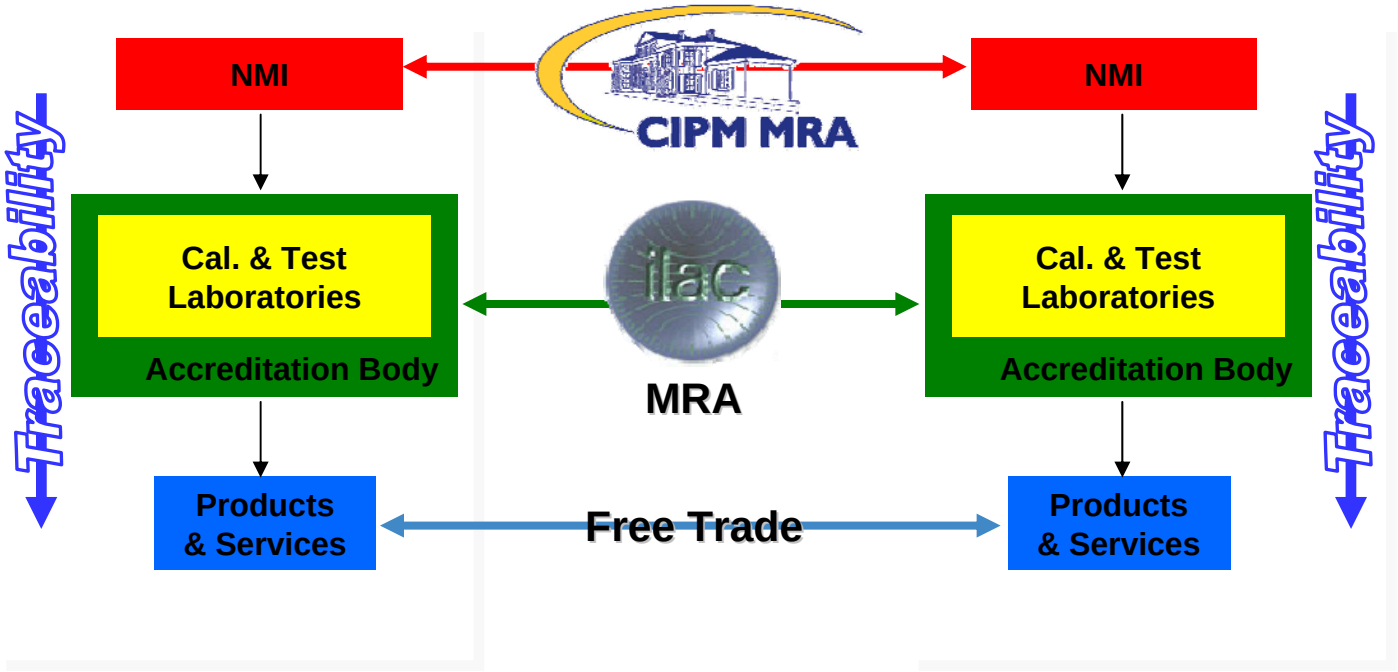
CIPM MRA Participation



47 Member States
27 Associates of the CGPM
2 international organizations
(207 NMIs + DIs)
96% of the 2008 World GDP

THE IMPORTANCE OF MEASUREMENT

A sound measurement system is fundamental in fields of science, production of goods and services, health, commerce, communications,…It creates the framework in which suppliers of products and services can demonstrate compliance with specifications within an internationally standardized system.



MSTQ is essential for strengthening export

Metrology: A Key Success Factor

- ✓ Lake Victoria fish (EU ban caused damage of 100 million US$ p.a. and 150 000 people jobless)
- ✓ Sri Lanka tea export (90 billion Rs p.a. (800 million US$) hindered due to inability to measure pesticides and lack of international recognition)
- ✓ Chilean export of marine, fish, meat, milk and agricultural products (10.5 billion USD p.a.) vulnerable due to lack of sufficient credible traceable testing
- ✓ Australian export of horticulture products based on traceability and international recognition

Metrology: A Key Success Factor

- ✓ Copper production in Chile about 2 x 10^9 kg per year; 0.05% measurement error may lead to a loss of more than 100 million US$ per year
- ✓ Sony electronics lost 110 million euros in sales (52 million euros profit) due to debate on the credibility of the level of Cd in Sony play station cables, exceding maximum admissible limits
- ✓ Global CO_2 trading based on traceability to the SI and international recognition

Metrology: A Key Success factor

- Gas and oil chemical composition and volume essential parameters
- 993 billion dollar international trade in 2004
- In Western Europe a 1% measurement error in natural gas measurements equals a commercial value of more than 1 billion euros per year
- New energy sources (bio fuels, hydrogen fuels)
- Comparable measurements traceable to the SI basis for fair trade, industrial innovation, sustainable economy, better quality of life
- The CIPM MRA is essential

Conclusion

- Clear need for comparability through traceability to SI or if not yet feasible to other internationally agreed references
- Metrology essential in value chain, natl quality system
- Under the CIPM MRA internationally recognized NMI essential for the countries economy and wellfare
- Essential for accreditation and certification
- Sustainable competitiveness, facilitating export, strengthening the economy, improving quality of life
- Strengthening society, industry, SMEs by internatl. recognition of measurement and testing capabilities

And how was your dinner ?

- How was the food and the wine ?
  - color
  - bouquet, smell
  - rare, medium, well cooked
  - plain on your tongue, taste
  - after-taste

- Need for “soft” metrology
  - color
  - taste
  - smell
  - glance

THANK YOU

www.bipm.org