

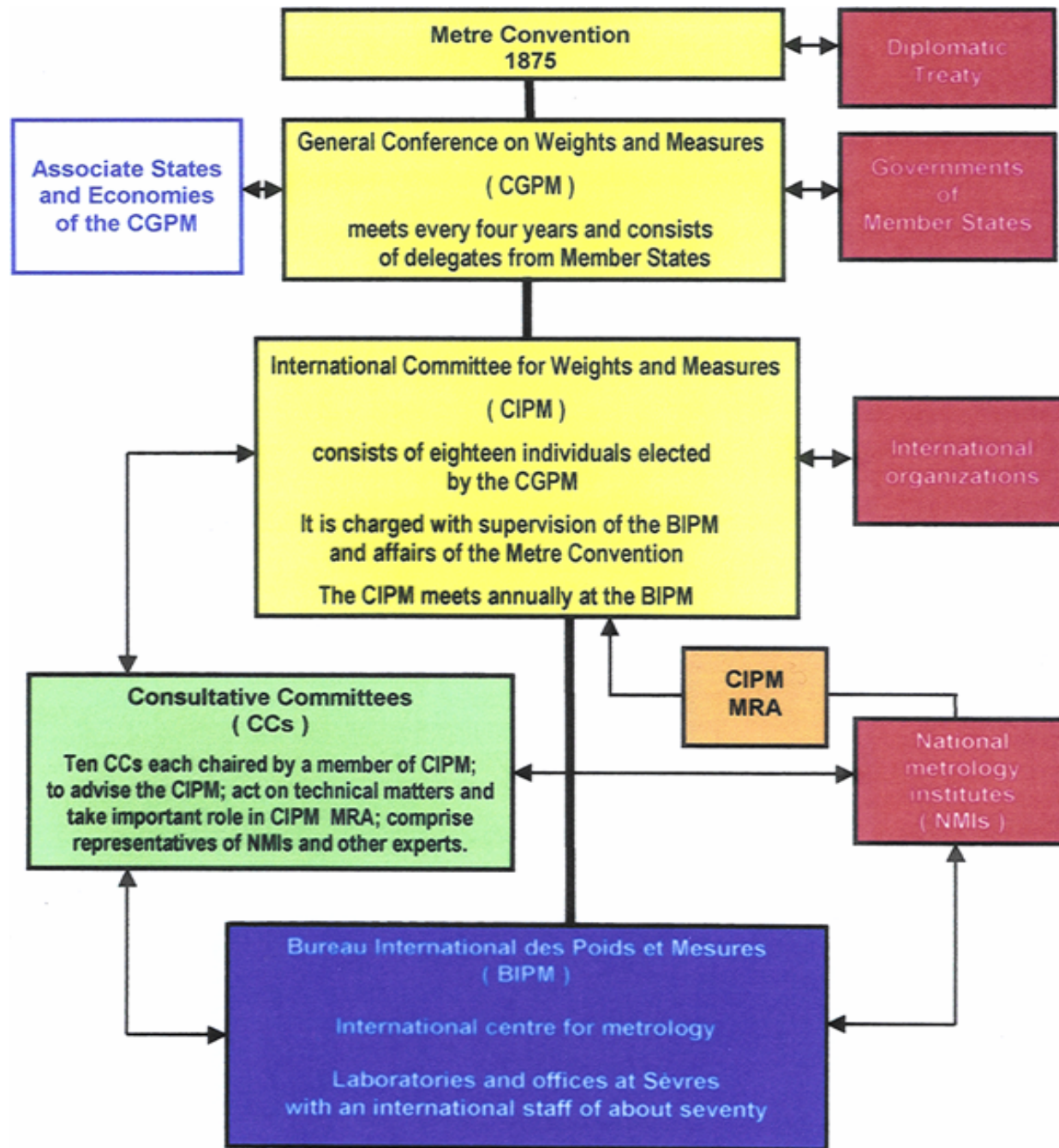
ESTABLISHING INTERNATIONAL TRACEABILITY and COMPARABILITY

Robert Kaarls
Secretary CIPM
President CCQM
Formerly Director NMI – The Netherlands

Workshop on Measurement Traceability
And Uncertainty in Analytical Chemistry
Luzern, 17-18 June 2002

Inter-governmental Treaty “Metre Convention”

- General Conference of Weights and Measures - CGPM
- International Committee of Weights and Measures - CIPM
- 10 Consultative Committees, a.o. CCQM
- International Bureau of Weights and Measures - BIPM at Sevres, France



CCQM Metrology in Chemistry

- Established by CIPM in 1993
- After hypothesis on traceability and comparability had been proven
- Members and observers representing more than 40 institutes from 30 countries/economies
- Including (designated) NMI's and other international organisations (JRC-IRMM, IUPAC, IAEA, IFCC, ISO-REMCO, CITAC, a.o.)

CCQM Metrology in Chemistry

- **Activities**
- **Studies**
- **Primary methods (traceability)**
- **Key comparisons (comparability)**
- **How far does the light shine?**
- **Measurement uncertainty (ISO GUM, EURACHEM-CITAC Guide)**
- **Vocabulary (ISO VIM)**

CCQM Metrology in Chemistry

- **Areas defined in overall framework (1)**
- **Health (clinical diagnostic markers)**
- **Food (pesticides, toxins, drinking water)**
- **Environment (water, air, global warming, contaminants in soil)**
- **Advanced materials (semiconductors, alloys, plastics)**
- **Commodities (oil, cement, precious metals, alcohol content)**

CCQM Metrology in Chemistry

- **Areas defined in overall framework (2)**
- **Forensics (drugs, explosives, breath analysis, DNA)**
- **Pharmaceuticals**
- **Bio-technology (GMO's, DNA profiling, diagnostics)**
- **General analytical applications (purity, pH, isotopic standards)**

- CCQM
- CCQM Working Groups
- Key comparisons NIST
- Organic Analysis NIST
- Inorganic Analysis LGC
- Gas Analysis NMI
- Electro-chemical Analysis SMU
- Surface Analysis NPL
- Bio-technology LGC/NIST

Traceability

- Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties
- To SI, whenever possible; mol, kg, m, etc. or any combination (multiple, sub-multiple)
- To “next best” internationally agreed reference if SI is not possible

Primary method

- A primary method of measurement is a method having the highest metrological qualities, whose operation can be completely described and understood, for which a complete uncertainty statement can be written down in terms of SI units
- A primary direct method measures the value of an unknown without reference to a standard of the same quantity

Primary ratio method

- A primary ratio method measures the value of an unknown to a standard of the same quantity; its operation must be completely described by a measurement equation
- In this case almost always use is made of a high purity material (a “primary reference material”)
- Thus, high purity analysis are crucial

Definition and study of primary methods

- IDMS
- Coulometry
- Gravimetry
- Titrimetry
- Calorimetry (DSC)
- INAA
- Cavity Ring Down Spectroscopy
- ID-ICP-MS

Certified Reference Materials

- Calibration
- Validation - Recovery factor (calibration)
- In general **NOT** the top of the chain
- Importance of pure reference materials
- Role when traceability to SI can not be realized
- Limited availability
- Matrix problems

CCQM

- Studies
- Key Comparisons
- Supplementary and bilateral comparisons
- **How far does the light shine?**
- Review of calibration and measurement capabilities, including traceability delivered via CRM's, based on own characterizations and value assignment
- Reviewed Quality Assurance Systems

CCQM

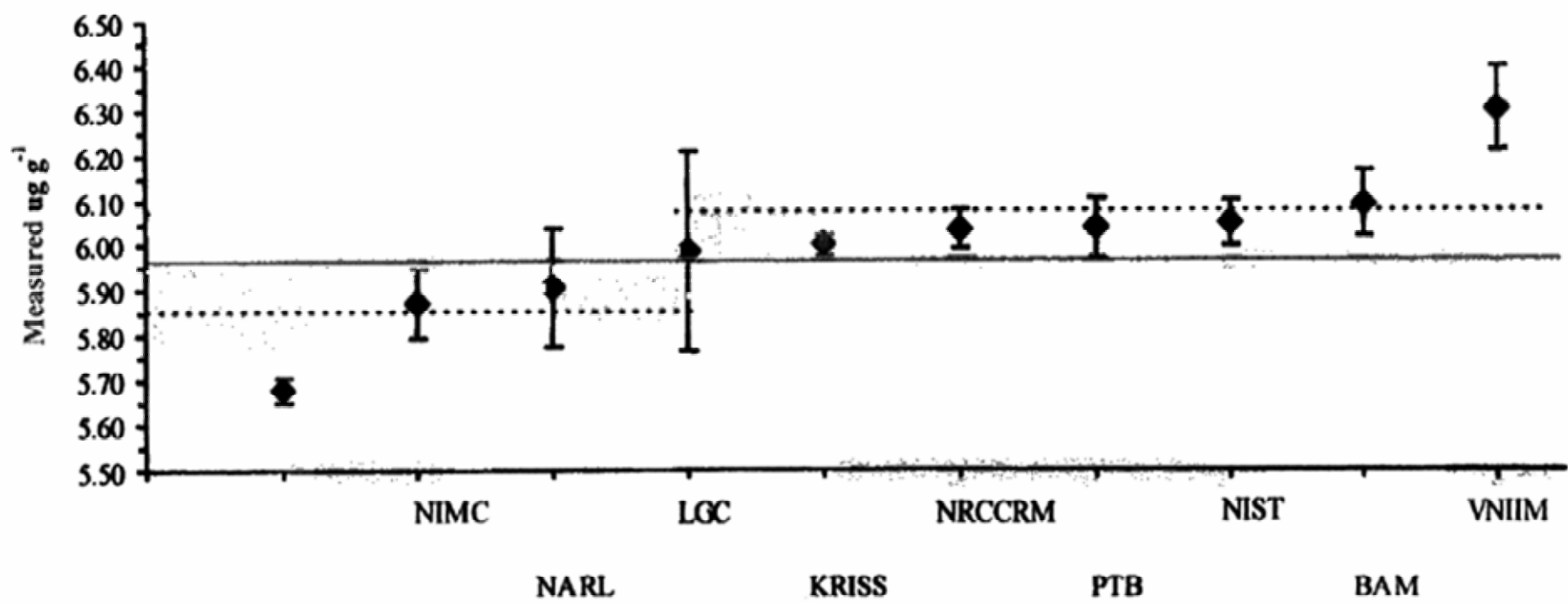
- **Studies**
- Development primary methods, procedures
- Try out comparisons
- How well can we do it ?
- How far does the light shine ?
- **TRACEABILITY** * primary method
 - * through “calibrated” artifact measuring instrument, CRM

CCQM

- **Key comparisons**
- Key Comparison Reference Value (KCRV)
- Assessing competence
- Assessing capabilities
- Verifying equivalence
- **Comparability**

- **Comparability through Traceability**

Figure 2 Sample B Results showing Mean and Upper and Lower Limits of the 95% C. I. of the KCRV (NARL and VNIIM excluded)



PP DDE in Fish Oil

New areas

- Environment
- Food
- Laboratory medicine (in-vitro diagnostics)
- Forensics, drugs, doping
- Bio analysis
- Surface analysis

New networks

- WMO/Global Atmospheric Watch
- WHO/IFCC
- Codex Alimentarius/Global Harmonization Task Force
- EU Reference Laboratories
- Anti-doping Laboratories
- Intensified co-operation with ISO/IEC, WTO, OIML, IMEKO, IUPAC, IUPAP, etc

New networks in metrology

- Covering environmental area, laboratory medicine, food safety, anti-doping, forensics, etc.
- EU is creating EU reference laboratories (should be connected to the NMI's!)
- BIPM establishes networks with ISO/IEC, WTO, ILAC, WMO, WHO/IFCC, Codex Alimentarius, apart from existing contacts with OIML, IMEKO, IUPAP, IUPAC, a.o.

New networks in metrology

- Society requires metrology in much more areas, like environment, health-care, food
- Most NMI's have no wide capabilities and competence in chemistry and biotechnology
- Virtual/decentralized NMI's have to be established, by designating other key institutes as an NMI with responsibilities in a certain field (for specified quantities and measurement ranges)
- These NMI's must join in international activities under the RMO's and the Metre Convention

Role and tasks of the BIPM

- International measurement system
- Traceability, comparability, recognition
- International co-ordination in metrology
- Promotion, stimulation
- Unique international and unique transfer standards (e.g. ozone standard, may be some essential pure materials)

CIPM - MRA

- Signed in October 1999
- By NMI's of Metre Convention Member States
- Internatl. Organizations (IAEA, IRMM)
- Possibilities for Associates to the CGPM
- Appendix B (key and supplementary comparisons)
- Appendix C (best calibration and measurement capabilities, underpinned by results of comparisons and reviewed quality systems)

CIPM – MRA www.bipm.org

- International recognition of national measurement standards and of calibration and measurement certificates issued by national metrology organizations
- Based on key- and supplementary comparisons **Appendix B**
- Quality assurance **(ISO 17025, 34, 35)**
- (Peer-) assessments **(accreditation)**
- NMI capabilities **Appendix C**

CCQM

Problems in metrology in chemistry

- Matrix structure
- Instabilities and differences in composition of artifacts (reference materials) used in comparisons
- Lack of precise definition of measurand
- Need for precise prescription for treatment and measurement of artifact (sample, CRM)
- Measurement uncertainty calculations
- How far does the light shine?

Why comparability through Traceability ?

- Need for reliable measurements
- Take away Technical Barriers to Trade
- Trade, Accreditation, Metrology agreements, incl. internal markets
- Environment (long term stable reference points)
- Health care
- Food safety
- Forensics, Customs, Anti-Doping, etc.