



Bureau International des Poids et Mesures



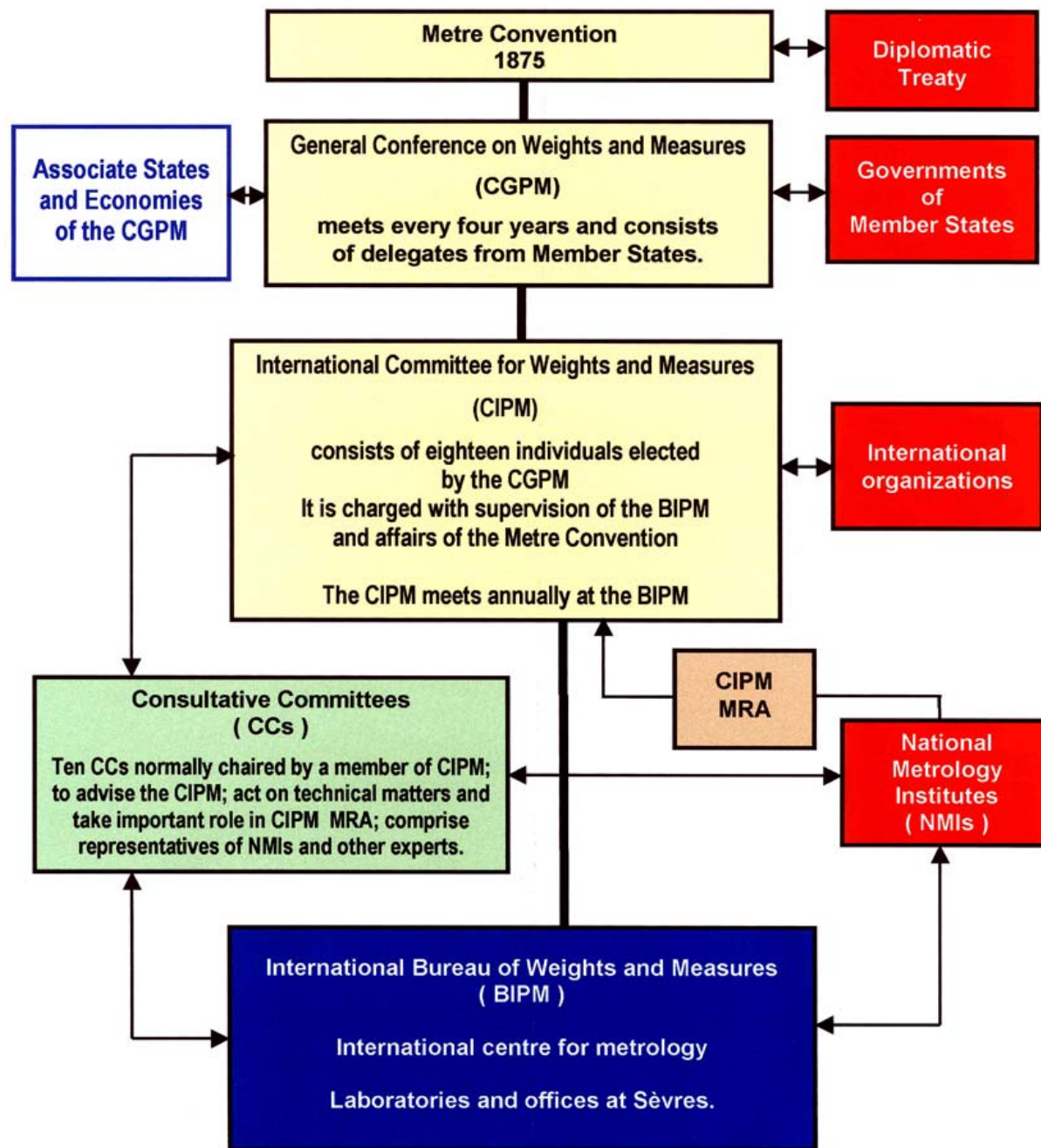
Key Comparisons, the MRA and CMCs: An International Measurement Infrastructure

Robert Wielgosz

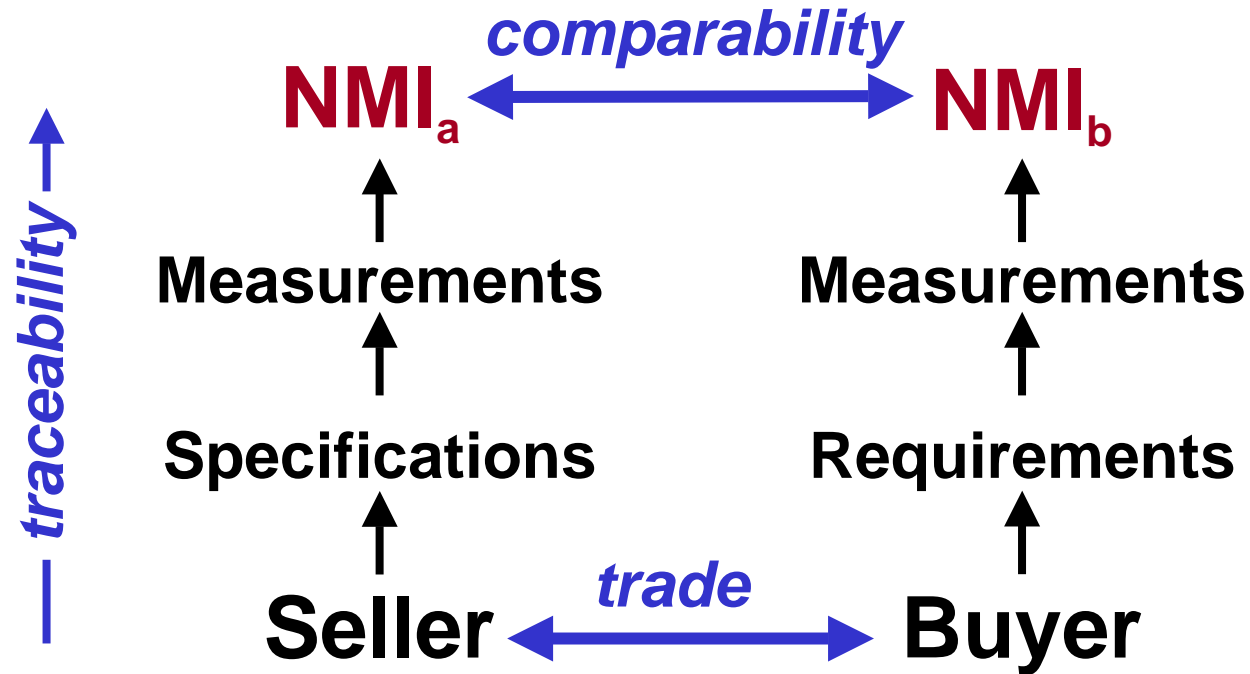
Bureau International des Poids et Mesures

The task of the BIPM is to ensure world-wide uniformity of measurements and their traceability to the International System of Units (SI).





International Trade Requires Traceability *and* Comparability



NMI: National Measurement Institute

Bureau International des Poids et Mesures



Reconnaissance mutuelle

des étalons nationaux de mesure
et des certificats d'étalonnage et de mesurage
émis par les laboratoires nationaux de métrologie

Paris, le 14 octobre 1999



Mutual recognition
of national measurement standards
and of calibration and measurement certificates
issued by national metrology institutes

Paris, 14 October 1999

Comité international des poids et mesures

Bureau
international
des poids
et mesures

Organisation
intergouvernementale
de la Convention
du Mètre

Bureau International des Poids et Mesures



Objectives of the MRA are to:

establish the degree of equivalence of measurement standards maintained by NMIs;

provide for the mutual recognition of calibration and measurement certificates issued by NMIs; thereby to

provide governments and other parties with a secure technical foundation for wider agreements related to international trade, commerce and regulatory affairs

Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes

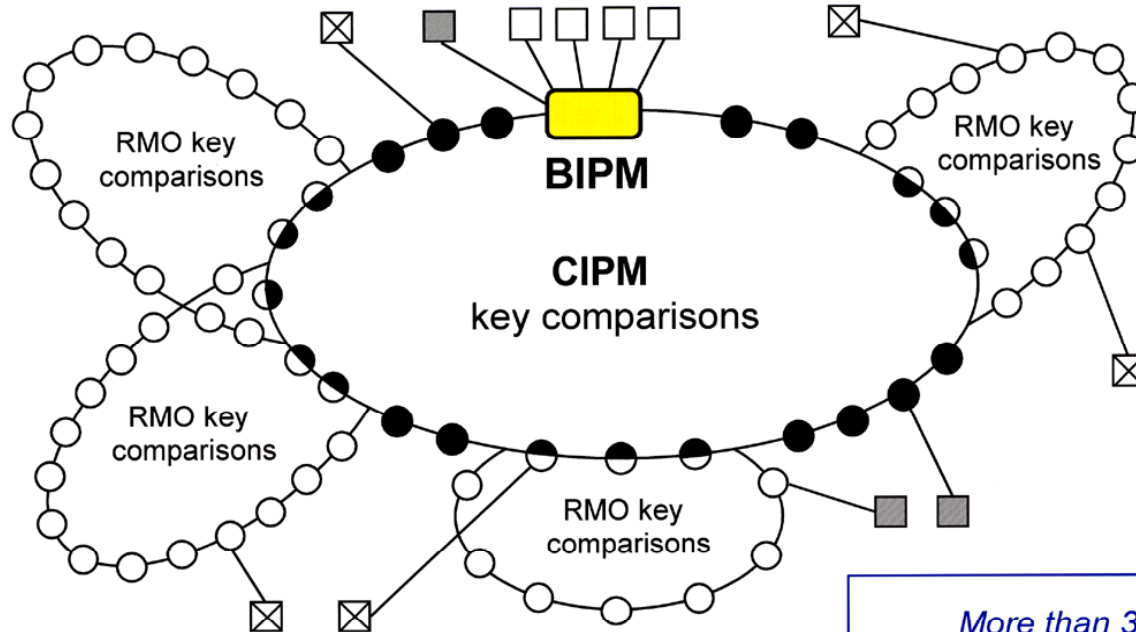
Statements of the measurement capabilities of each NMI in the BIPM key comparisons and calibration database publicly available on the web; included are:

the results of the key comparisons

lists of calibration and measurement capabilities of each participating NMI (some ten thousand lines of data now on the web)

www.bipm.org

Scheme for key comparisons

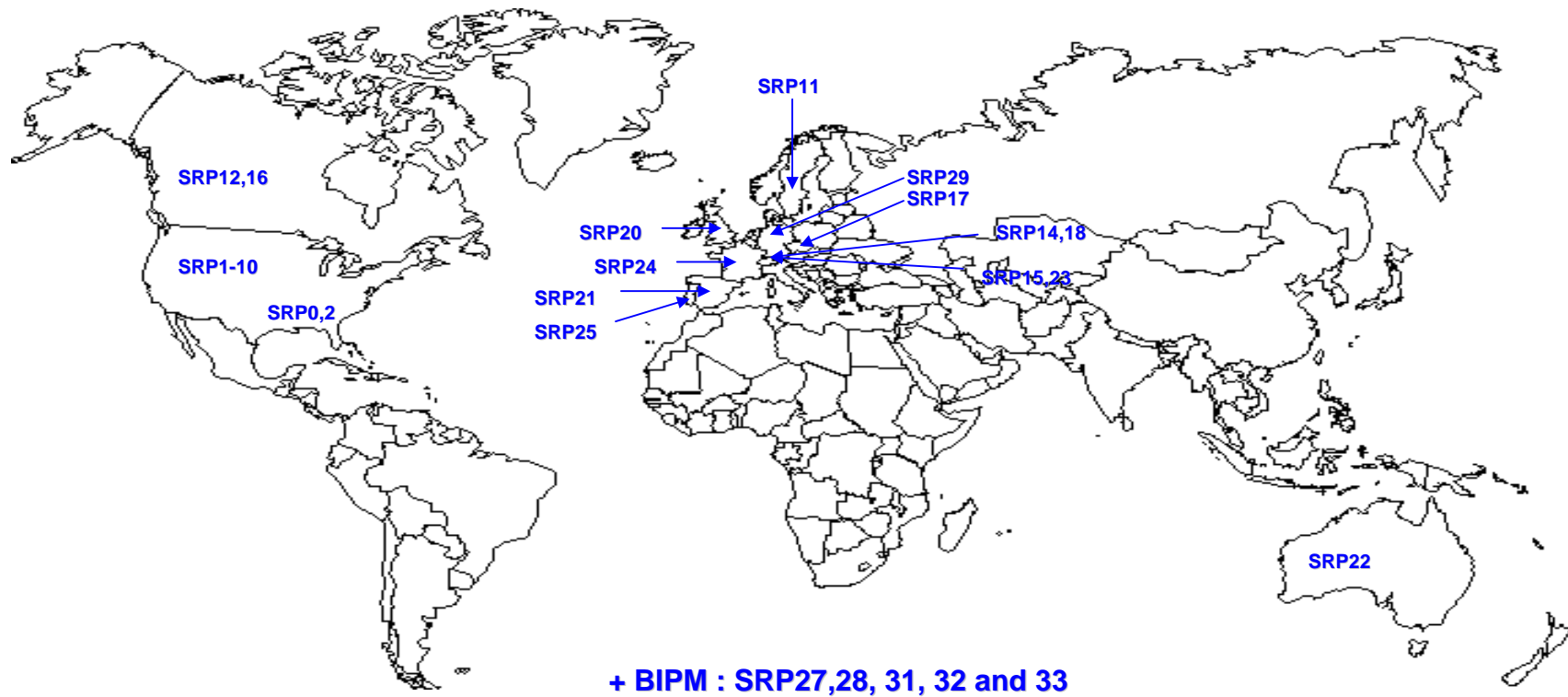


More than 300 key comparisons are in progress

BIPM

- NMI participating in CIPM key comparisons.
- ◐ NMI participating in CIPM key comparisons and in RMO key comparisons.
- NMI participating in RMO key comparisons.
- NMI participating in ongoing BIPM key comparisons.
- ⊗ NMI participating in a bilateral key comparison.
- International organization signatory to MRA.

CCQM-P28 Ozone, ambient level



SRP facilities at the BIPM

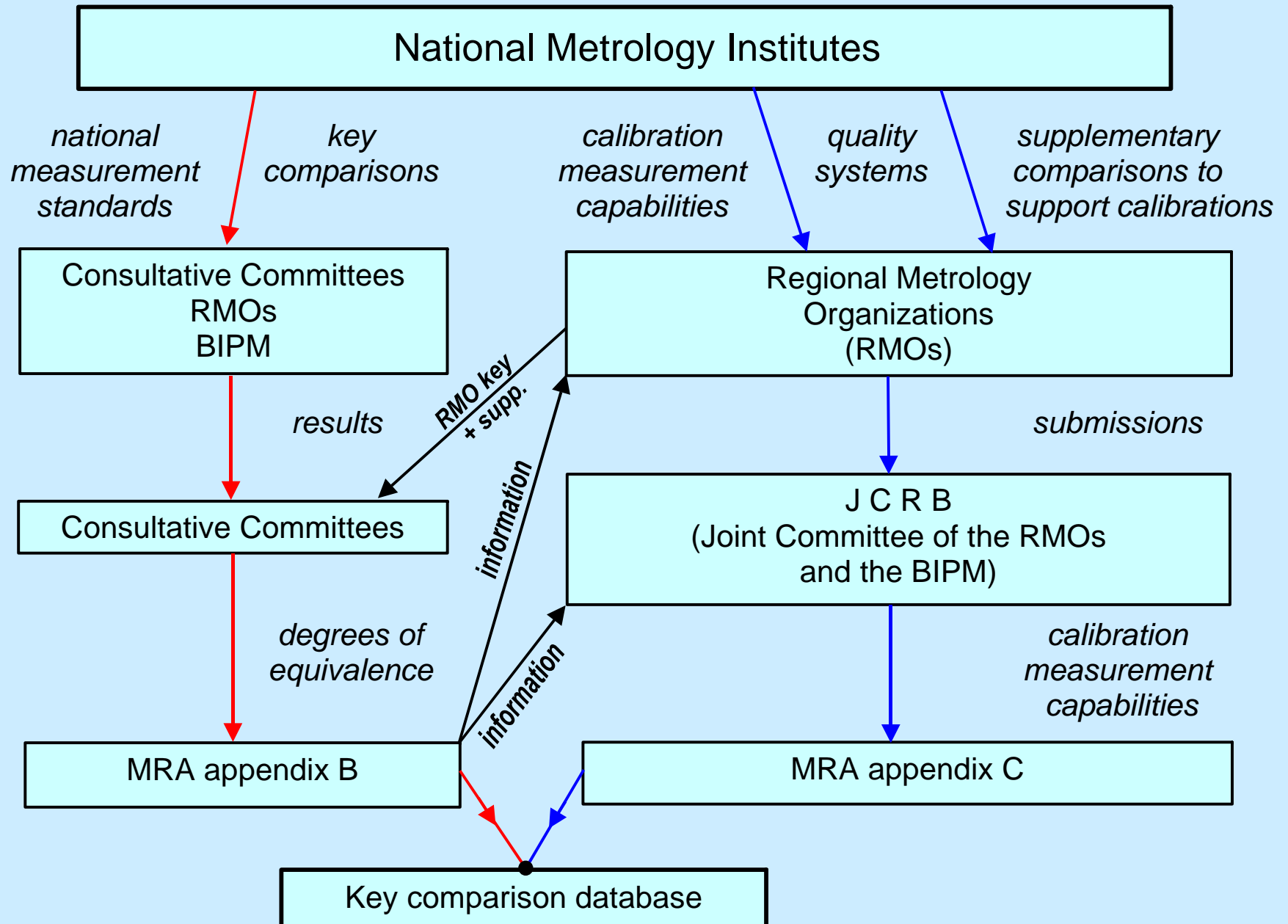


Measurement range: to 500 nmol/mol

Instruments: SRP27, SRP28, SRP31, SRP32, **SRP33**, **SRP19 (PTB, UBA)**

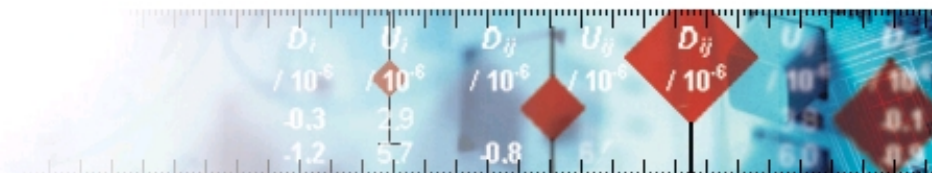
Reference instrument: **SRP27**

Mutual Recognition Arrangement





Bureau International des Poids et Mesures



APPENDIX A

APPENDIX B

APPENDIX C

APPENDIX D

[KCDB home](#)

The BIPM key comparison database



↳ KCDB

- [KCDB home](#)
- [KCDB news](#)
- [MRA](#)
- [JCRB](#)
- [Guidelines for key comparisons](#)
- [Nomenclature](#)
- [Metrologia](#)
- [Version française](#)

↳ What's new ?

- 16 February 2004 [Appendix C: Mass and Related Quantities \(Fluid Flow\)](#)
- 16 February 2004 [Appendix B: Key comparison CCTF-K2001.UTC](#)

↳ Contact us

- BIPM.KCDB@bipm.org

↳ KCDB Newsletter

- [Subscribe](#)

➔ **in support of the Mutual Recognition Arrangement (MRA) of national measurement standards and of calibration and measurement certificates issued by national metrology institutes**

The BIPM key comparison database is defined in the text of the [MRA](#) as "the database maintained by the BIPM (Bureau International des Poids et Mesures) which contains Appendices A, B, C and D of the Mutual Recognition Arrangement".

Appendix A: MRA signatories

List of national metrology institutes that are signatories to the arrangement.

Appendix B: Key and supplementary comparisons

Information on CIPM (Comité International des Poids et Mesures) and RMO (Regional Metrology Organization) key and supplementary comparisons, together with results when they become available.

Appendix C: Calibration and Measurement Capabilities (CMCs)

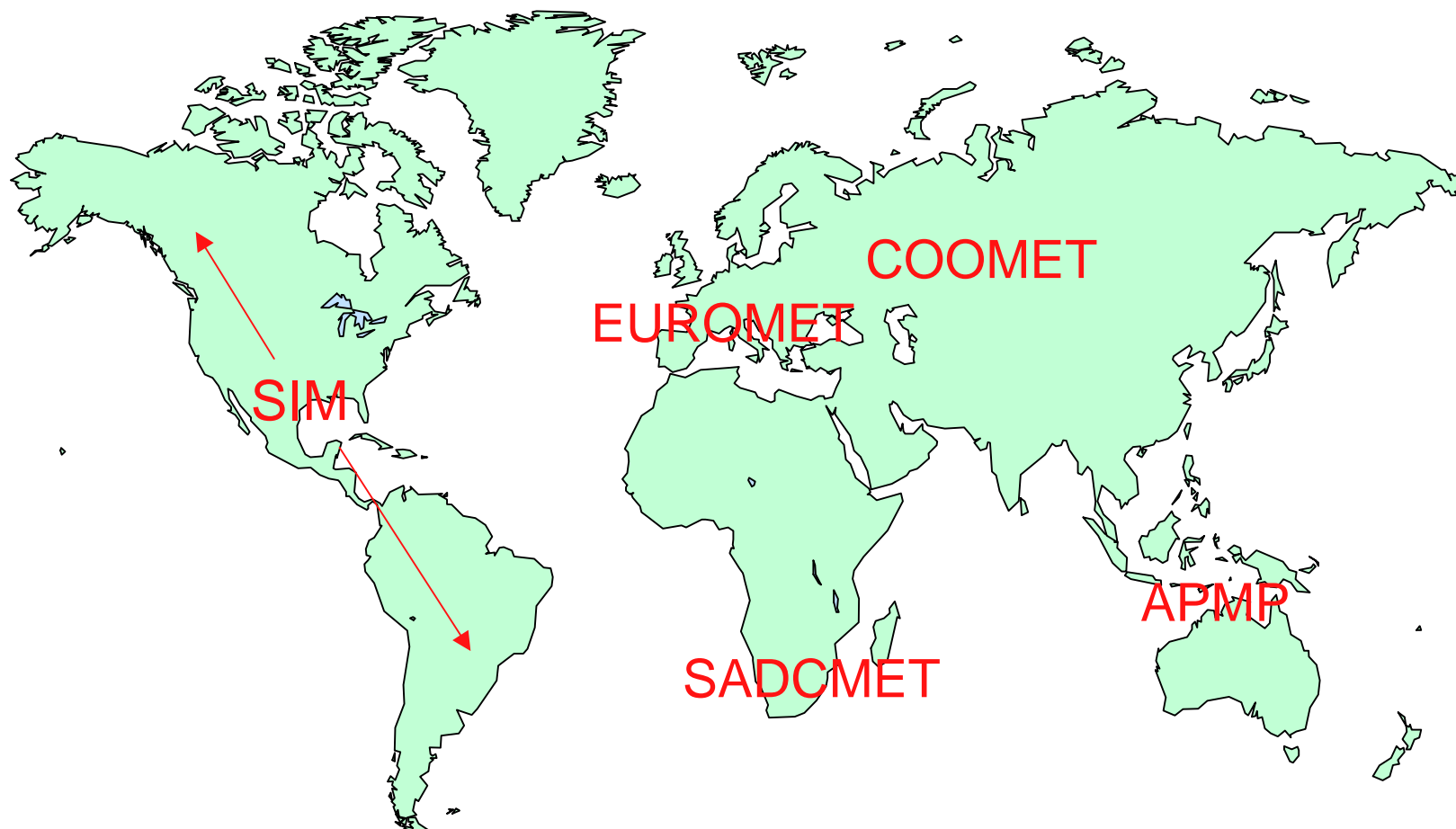
Quantities for which calibration and measurements certificates are recognized by institutes participating in part two of the arrangement.

Appendix D: List of key comparisons

The KCDB website is best viewed using version 7.0 of [Netscape](#) or version 5.0 or higher of [IE](#).



Regional Metrology Organizations



Consultative Committee for Amount of Substance – CCQM - Metrology in Chemistry

Aim

- Establishing worldwide comparability
- Traceability to SI, or if not (yet) possible to other internationally agreed references
- Development of primary methods and databases

Thus

- Scientific component
- Interlaboratory comparisons and proficiency testing component

CCQM

CCQM Working Groups

Key Comparisons and CMC Quality

Organic Analysis

Inorganic Analysis

Gas Analysis

Electro-chemical Analysis

Surface Analysis

Bio-Analysis

NRC-INMS

NIST

LGC

NMi

SMU

NPL

LGC/NIST

CCQM WG on Organic Analysis

- P,p' DDT in fish oil
- LSD in human urine
- Cholesterol in human serum
- Glucose and creatinine in human serum
- PCB's in sediment
- Organic purity assessment series
- Ethanol in aqueous matrix (forensic level)
- Organic calibration solutions (PCB, PAH, pesticides, etc.)
- Drugs of abuse in urine

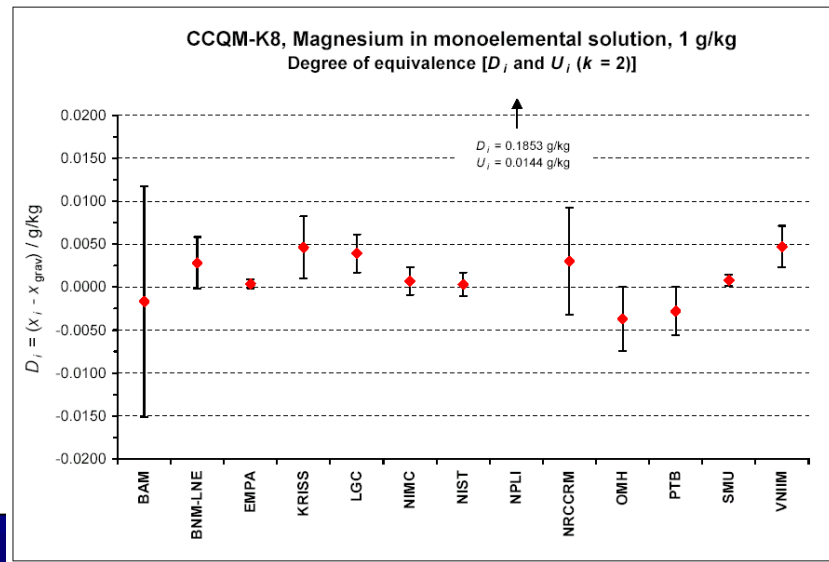
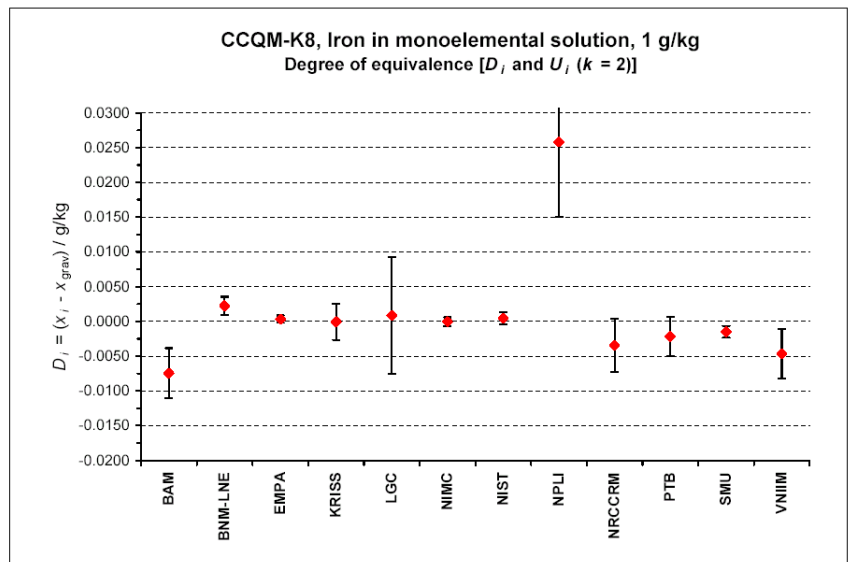
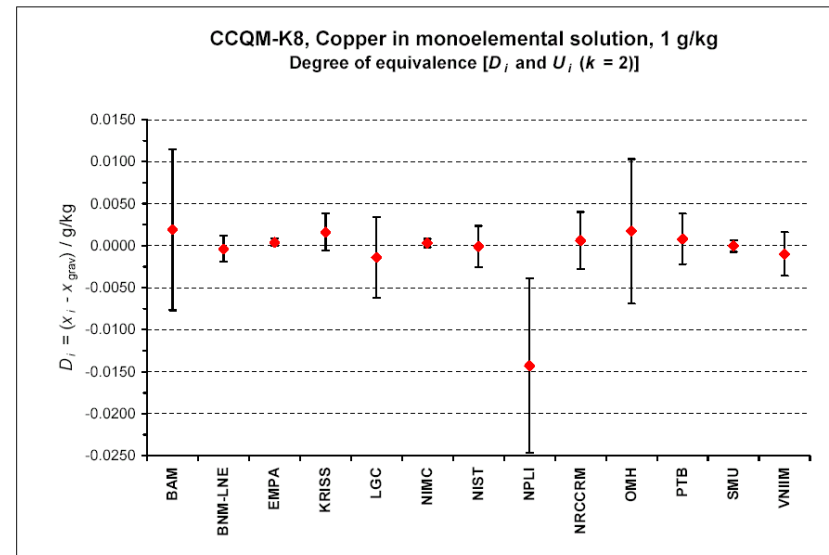
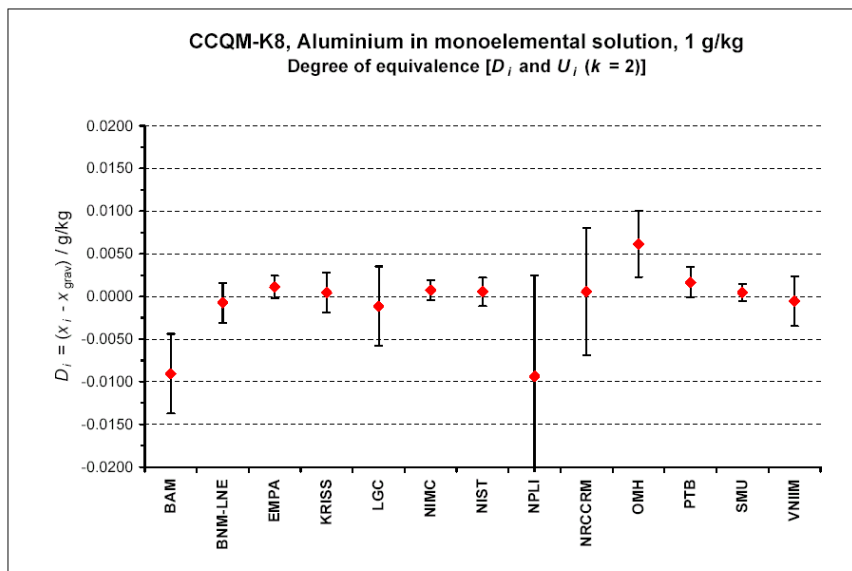
CCQM WG on Inorganic Analysis

- Mono-elemental solutions
- Pb and Cd in sediment
- Zn and Cd in rice ; Pb in wine
- Ca in serum
- As in fish or shellfish
- Metals in food digest
- S in fuel
- Minor elements in steel
- Boron in Si
- Purity of HCl

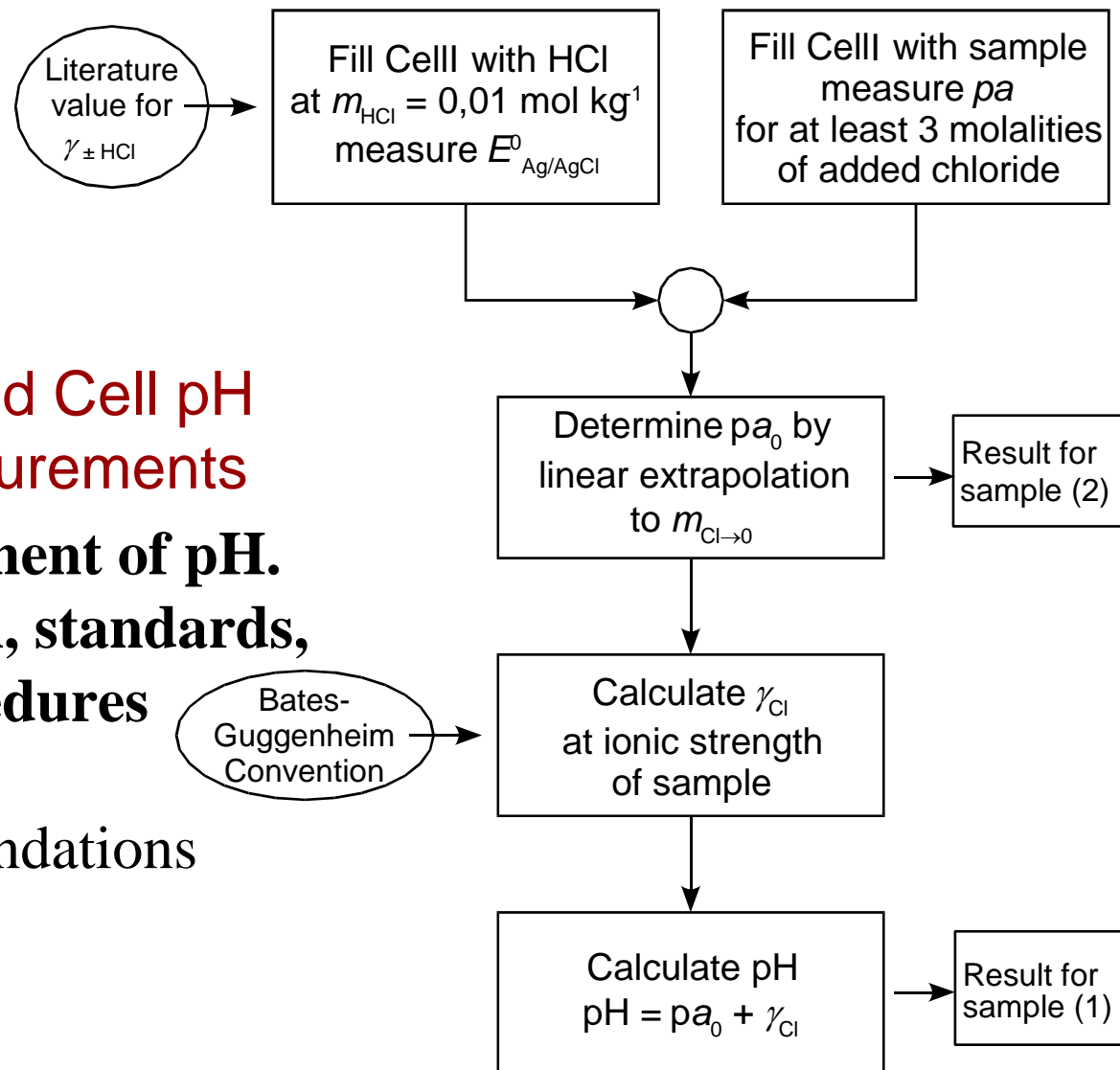
CCQM-K8: Elemental Calibration Solution Standards

[Al, Cu, Fe, Mg - each at nominal value of 1 g/kg]

Dates of Study: 1999-2000



Harned Cell pH measurements
Measurement of pH.
Definition, standards,
and procedures
(IUPAC
Recommendations
2002)



CCQM-K9: pH measurement comparison

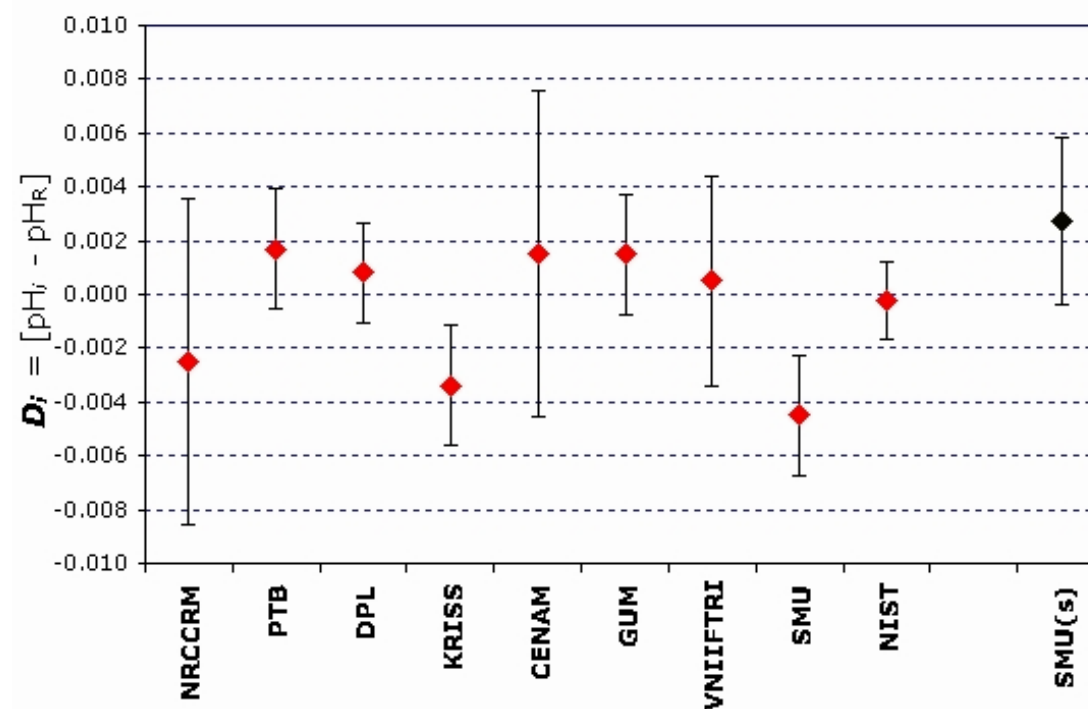
MEASURAND : pH value of phosphate buffer

Sample 1 : [0.025 mol kg⁻¹ KH₂PO₄ + 0.025 mol kg⁻¹ Na₂HPO₄]

Measurements at 15 °C

NOMINAL VALUE : pH = 6.9 at 25 °C

Degrees of equivalence D_i and expanded uncertainty U_i ($k = 2$)



◆ indicates the degree of equivalence for SMU resulting from the subsequent bilateral comparison between SMU and PTB.

Calibration and Measurement Capabilities (CMCs)

Appendix C Search Form

↘ Keyword search in one or several pre-selected categories

Please type part or full name of analyte. Select chemical categories in which the search is to be performed. Clicking on 'Search' returns the full list of analytes matching the entry and the category selection.

Analyte or component
keyword search :

Match exactly:

Yes
 No

All categories

Advanced materials

Food

Gases

Inorganic solutions

Organic solutions

Sediments, soils, ores, and particulates

Biological fluids and materials

Fuels

High purity chemicals

Metals and metal alloys

Other materials

Water

→ Search

Cholesterol in human serum

United Kingdom, LGC (Laboratory of the Government Chemist)

Complete CMCs in Amount of Substance for Biological fluids and materials for United Kingdom

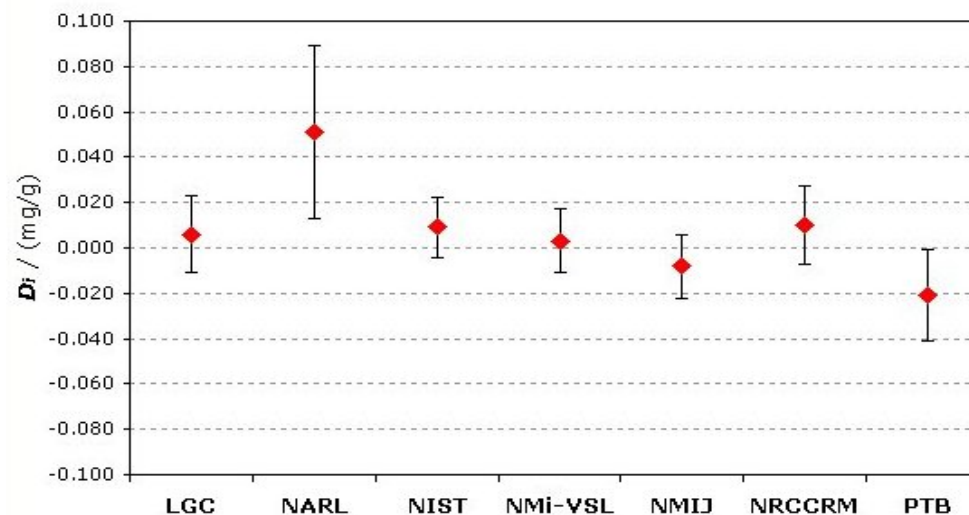
(.pdf file)

Matrix or material	Analyte or component	Dissemination range of measurement capability	
		Mass fraction in mg/g	Relative expanded uncertainty in %
blood serum	cholesterol	1 to 3	0.2 to 1

Mechanism(s) for measurement service delivery: Calibration

MEASURAND : Mass fraction of cholesterol in human serum
Material B : physiological range, ~1.7 mg/g

Degrees of equivalence D_i , and expanded uncertainty U_i , (95% level of confidence) expressed in mg/g ($x_R = 1.726$ mg/g, $U_R = 0.013$ mg/g)



Automotive Emission Gases: CO in N₂

Gases, Environmental

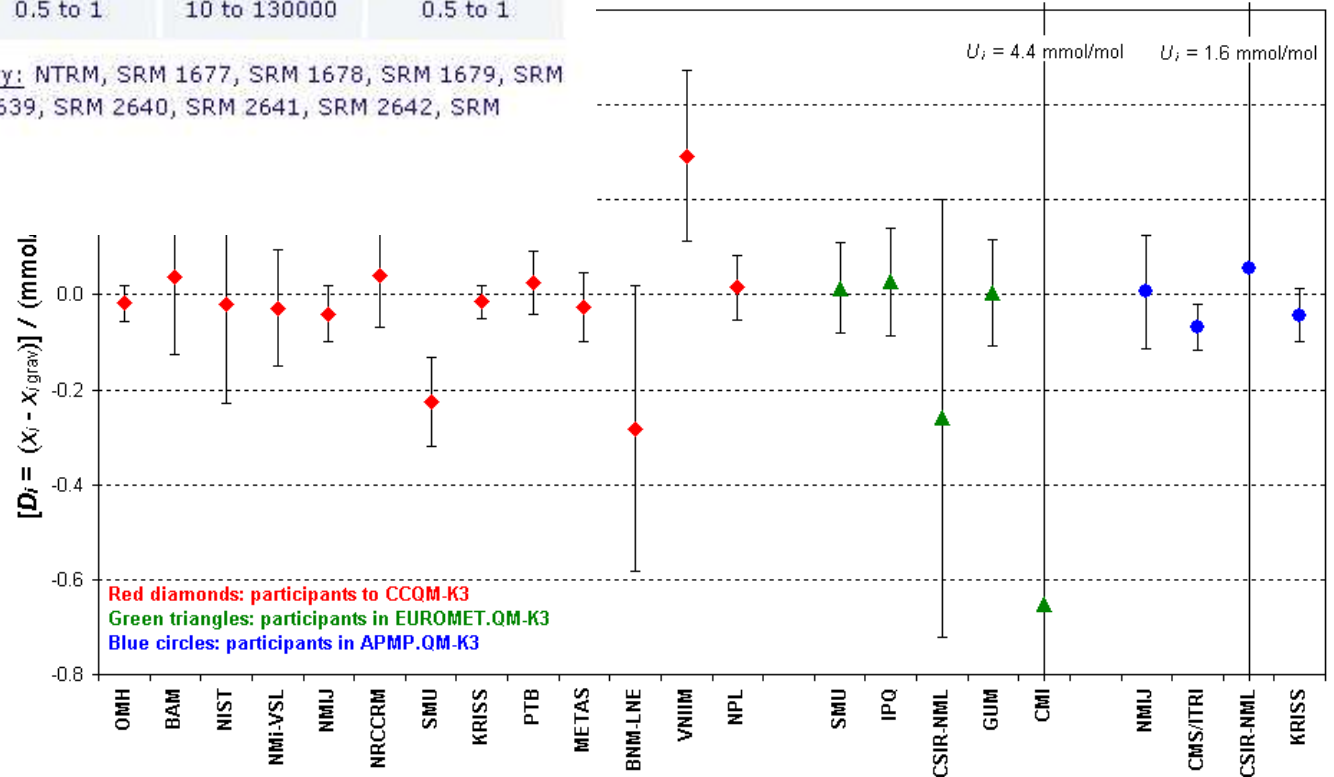
United States, NIST (National Institute of Standards and Technology)

Complete CMCs in Amount of Substance for Gases 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 fraction in $\mu\text{mol/mol}$	Relative expanded uncertainty in %	Amount-of-substance fraction in $\mu\text{mol/mol}$	Relative expanded uncertainty in %
nitrogen	carbon monoxide	1 to 1.5E+05	0.5 to 1	10 to 130000	0.5 to 1

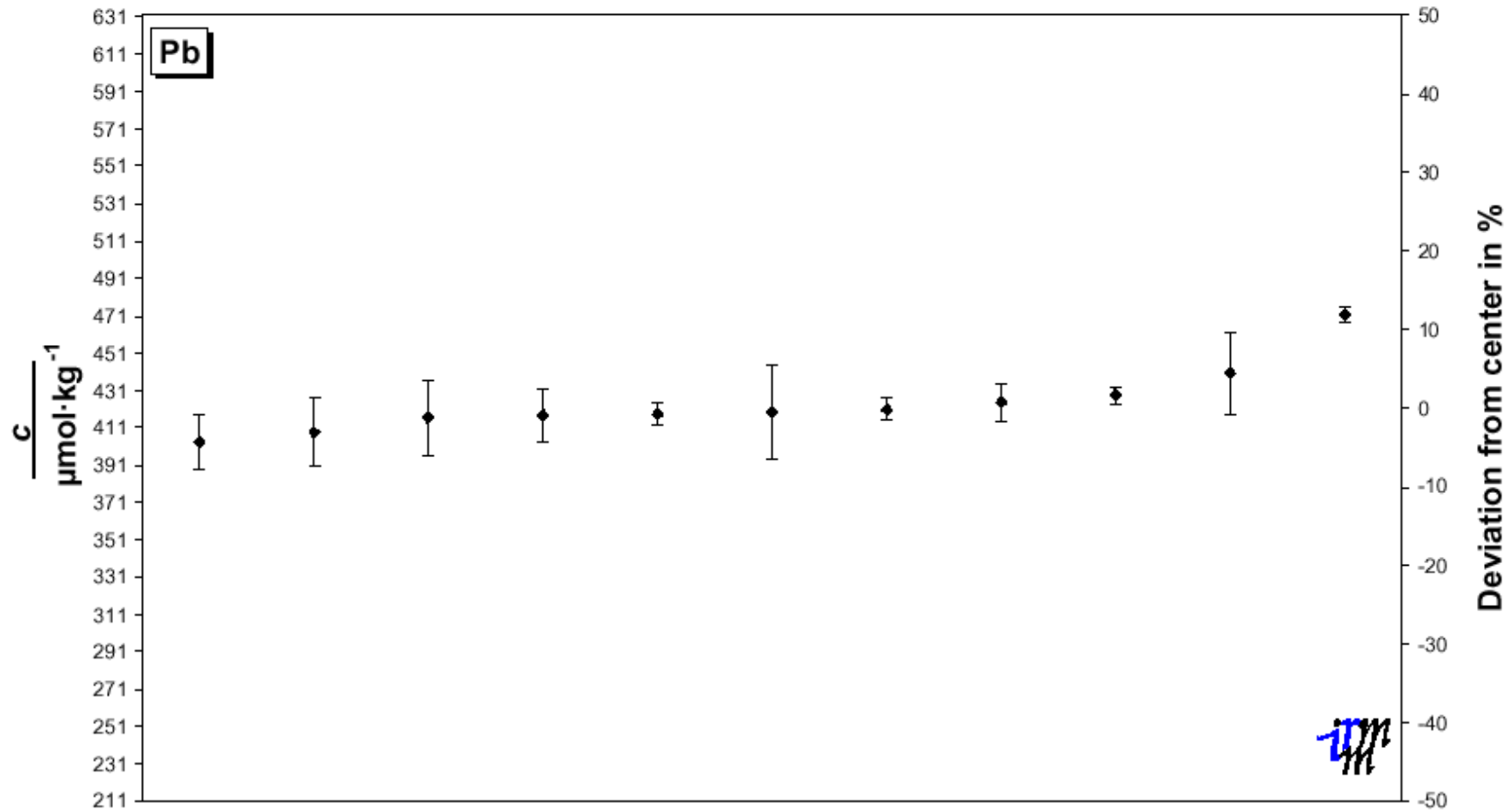
Mechanism(s) for measurement service delivery: NTRM, SRM 1677, SRM 1678, SRM 1679, SRM 1680, SRM 2636, SRM 2637, SRM 2638, SRM 2639, SRM 2640, SRM 2641, SRM 2642, SRM 2740, SRM 2741

and APMP.QM-K3 Automotive emission gases
for Carbon monoxide, CCQM-K3 nominal value: 32 mmol/mol



CCQM Pilot Study CCQM-P15, Pb and Cd in Sediment

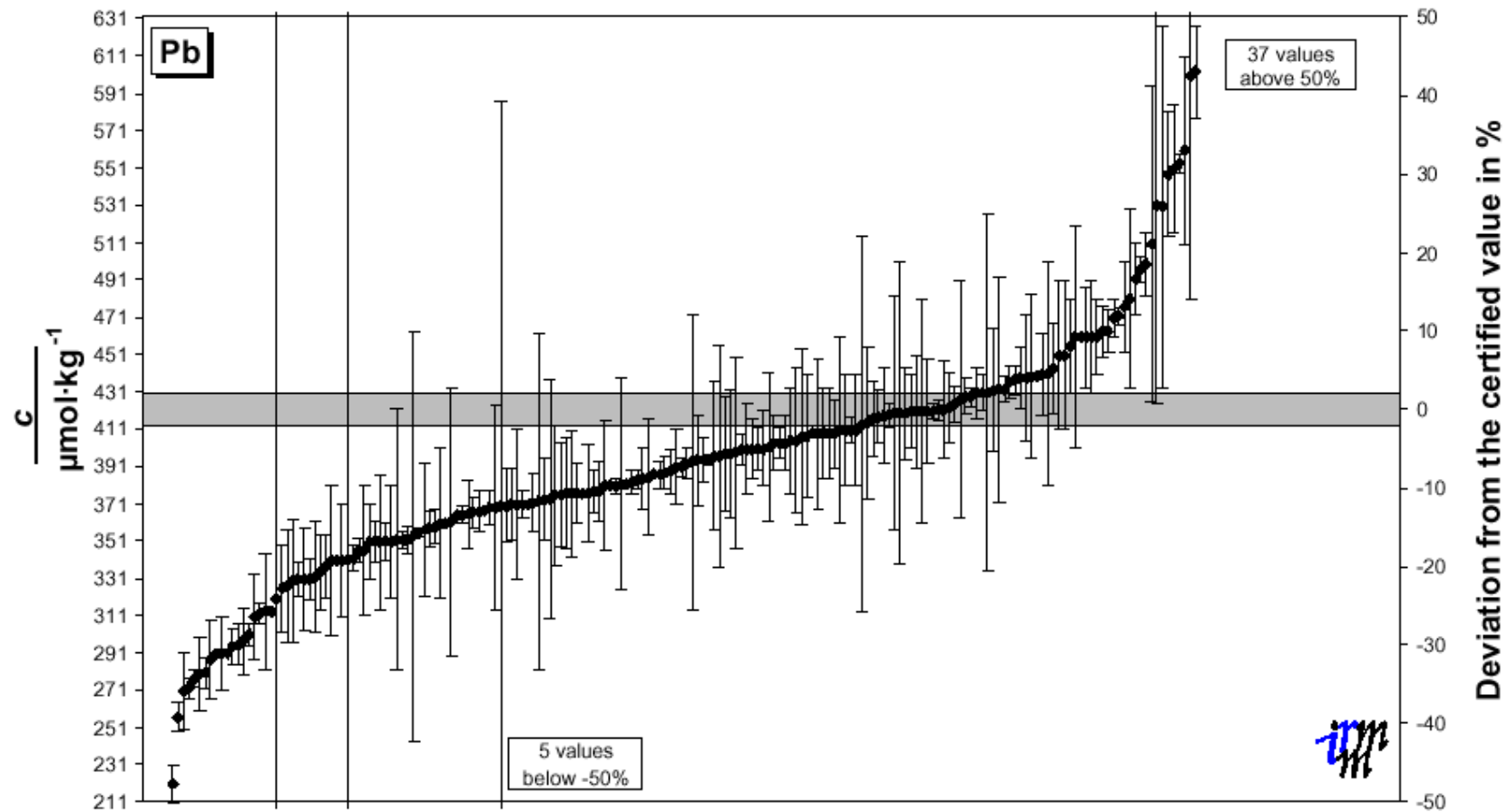
CCQM-P15 : Pb and Cd in sediment



CCQM Pilot Study CCQM-P15, Pb and Cd in Sediment

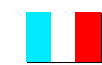
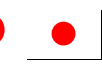
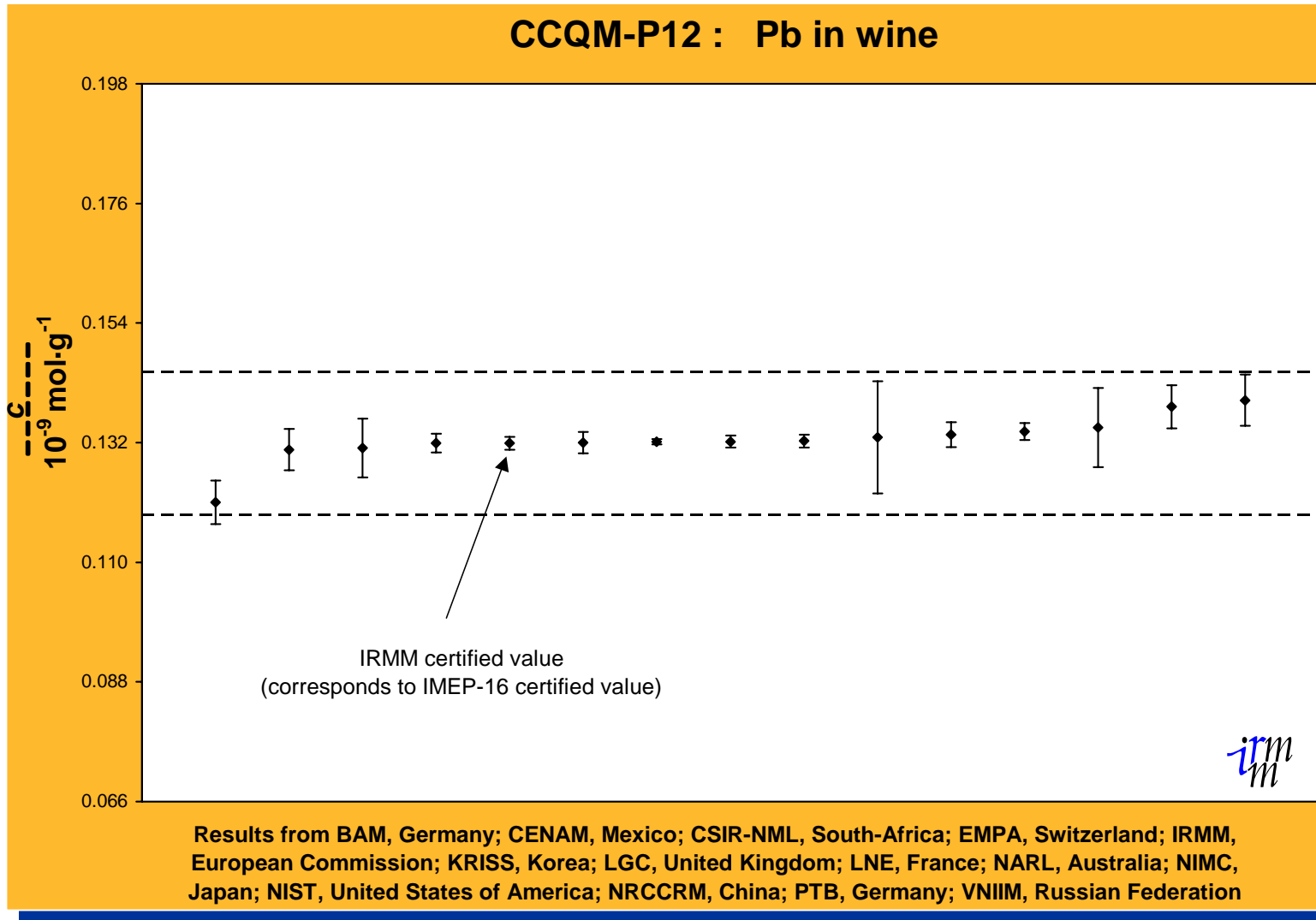
IMEP- 14 : Trace elements in sediment

Certified value : $421.1 \pm 8.4 \mu\text{mol}\cdot\text{kg}^{-1}$ [$U=k\cdot u_c$ ($k=2$)]



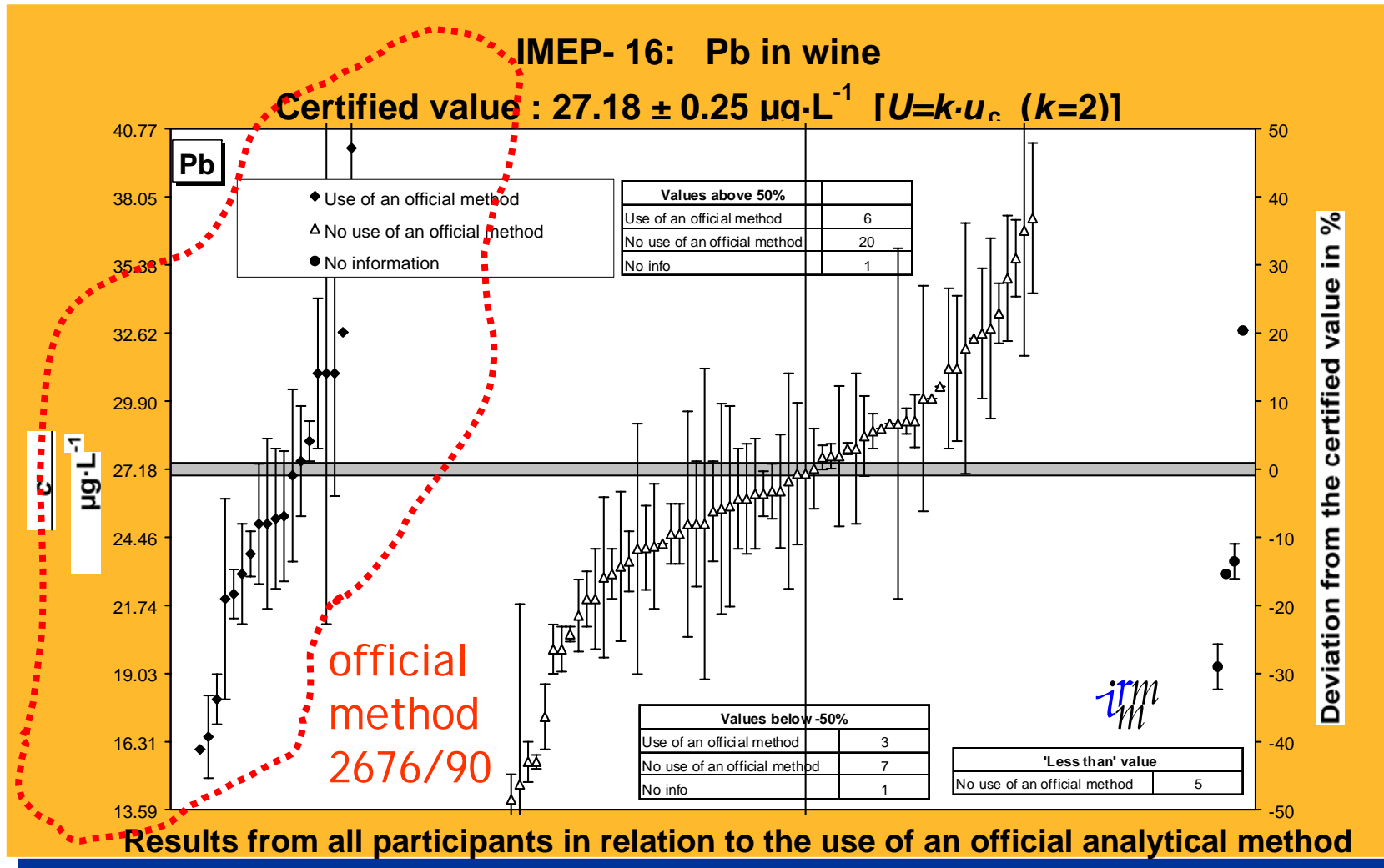
Results from all participants.

CCQM-P12 Pb in Wine

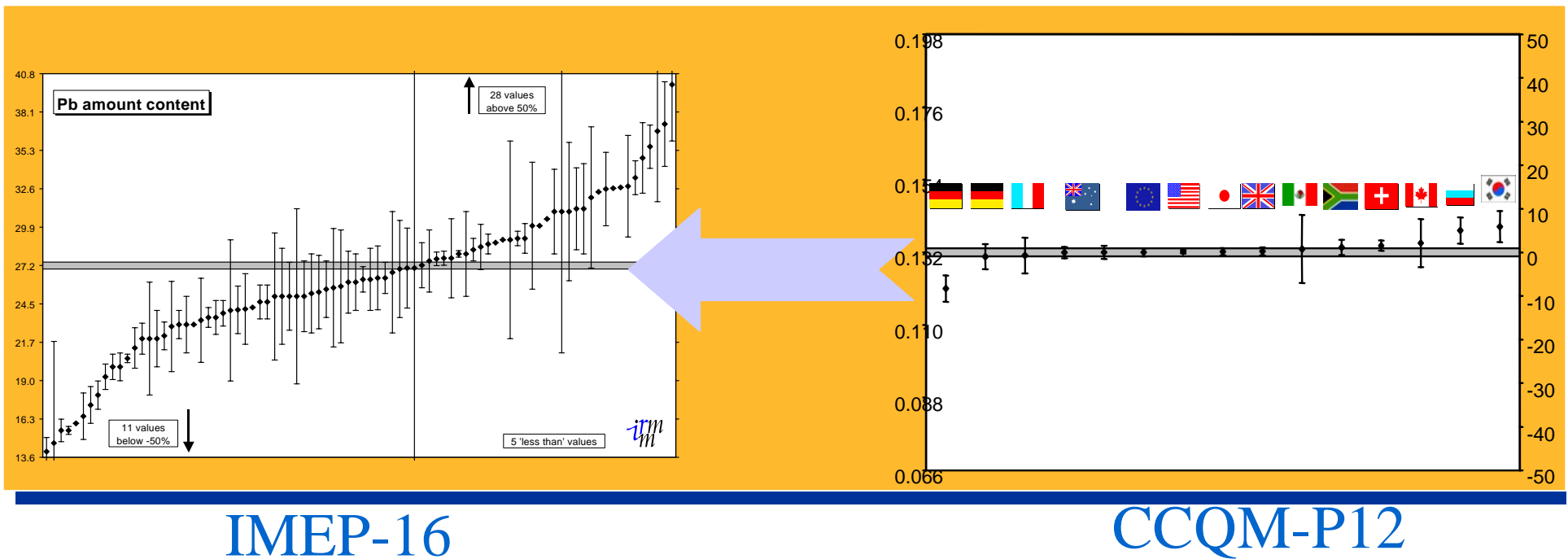


±50%

Assessing the quality of results of measurements : IMEP-16



Reference value, demonstrated to be equivalent at global level



JOINT COMMITTEE on TRACEABILITY in LABORATORY MEDICINE (JCTLM)

Declaration of co-operation
establishing

A framework for the international recognition of available higher-order reference materials, measurement procedures and reference measurement laboratories



Mission Statement

“The aim of the Joint Committee is to support world-wide comparability, reliability and equivalence of measurement results in Laboratory Medicine, for the purpose of improving health care, by:

- promoting the concept of traceability of measurement results to the *Système International d’Unités* (SI) or, where necessary, to other internationally agreed references;
- promoting close links between Reference Laboratories in Laboratory Medicine and National Metrology Institutes;
- co-ordinating and giving guidance in the establishment of Reference Measurement Systems with respect to medical needs;

Implementation of

- EC-IVD Directive (98/79/EC)

EC-IVD Directive, Annex I, Essential requirements A.3

'...The traceability of values assigned to calibrators and/or control materials must be assured through available reference measurement procedures and/or available reference materials of a higher order.'

Organization Structure

JCTLM Executive

- Priority setting
- Decision on projects
- Identification of appropriate organizations

2002-2004:

Chairman - IFCC : Prof J H H Thijssen

Secretariat - BIPM

Executive Secretary: Dr R I Wielgosz

IFCC, ILAC, BIPM and JCTLM WG Chairmen

Working Groups:

WG 1: Reference Materials and Reference Procedures

WG 2: Reference Measurement Laboratories

JCTLM Working Group on Reference Materials and Reference Measurement Procedures

Charge:

- establishing a process for identifying, and reviewing against agreed upon criteria
- publishing a List of “higher order” Certified Reference Materials and Reference Measurement Procedures required for IVD industry compliance with the EC IVD Directive regarding in vitro diagnostic medical devices.

Co-Chairs: Willie E. May (NIST)

Heinz Schimmel (EU Institute for Reference Materials & Measurements)

JCTLM Working Group on Reference Laboratory Networks

Charge:

- Collecting information on existing and candidate reference measurement laboratories (RMLs)
- Encouraging and facilitating the formation of networks of RMLs for different groups of measurable quantities (concerning electrolytes, substrates/metabolites, enzymes, HbA1c, low molecular hormones, etc.)
- Developing comparisons to demonstrate the competence of RMLs

Co-Chairs: Professor Dr. Lothar Siekmann, University of Bonn (Germany)
Professor Dr. Linda Thienpont, University of Gent (Belgium)

Relevant ISO Standards

ISO 17511 In vitro diagnostic medical devices - Measurement of quantities in biological samples - Metrological traceability of values assigned to calibrators and control materials

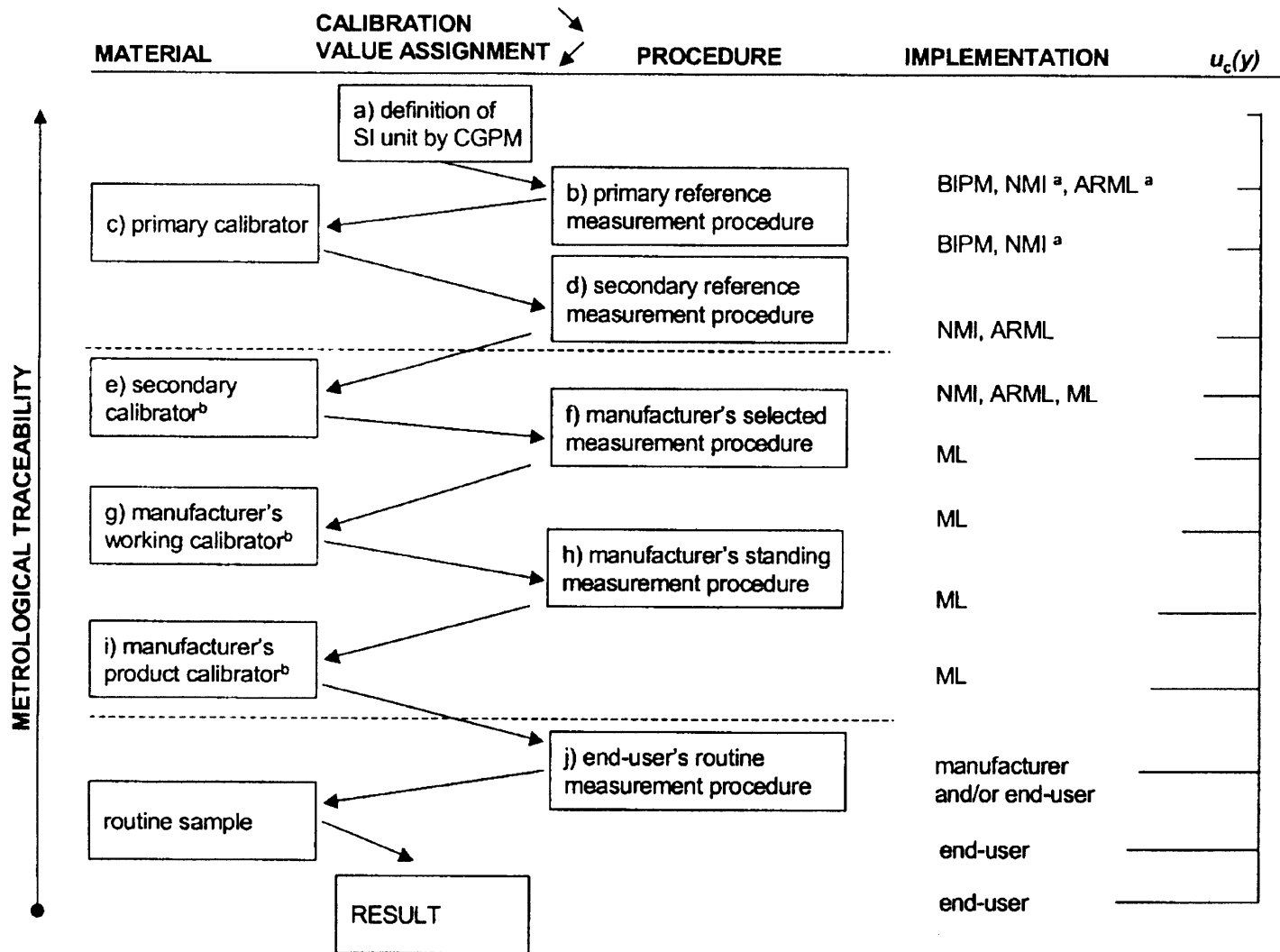
ISO 15193 Presentation of reference measurement procedures

ISO 15194 Description of reference materials

ISO 15195 Reference Measurement Laboratories

ISO 18153 Metrological traceability of values for catalytic concentration of enzymes assigned to calibrators and control materials

Primary Calibrators (ISO 17511)



Initial Provisional List of Higher Order Reference Materials and Reference Measurement Procedures -

Two categories:

- I. Certified Reference Materials and Reference Measurement Procedures for well-defined chemical entities or internationally recognized reference method-defined measurands, such as enzymes. Reference Materials included in this category are those that are traceable to the SI units. [Electrolytes, Enzymes, Drugs, Metabolites and Substrates, Non-Peptide Hormones, Proteins (some)]
- II. International Conventional Reference Materials, i.e., where the measurand(s) is/are not completely defined and/or no internationally recognized reference measurement procedure is available [e.g., WHO reference materials for Coagulation Factors, Nucleic Acids, some Proteins]

Joint Committee on Traceability in Laboratory Medicine

JCTLM

Review Teams for Highest Priority Analyte Areas

Worldwide representation from Lab Accreditation Organizations, NMIs, Professional Societies , and IVD Industry

Electrolytes

Enzymes

Metabolites and Substrates

Proteins

Nucleic Acids

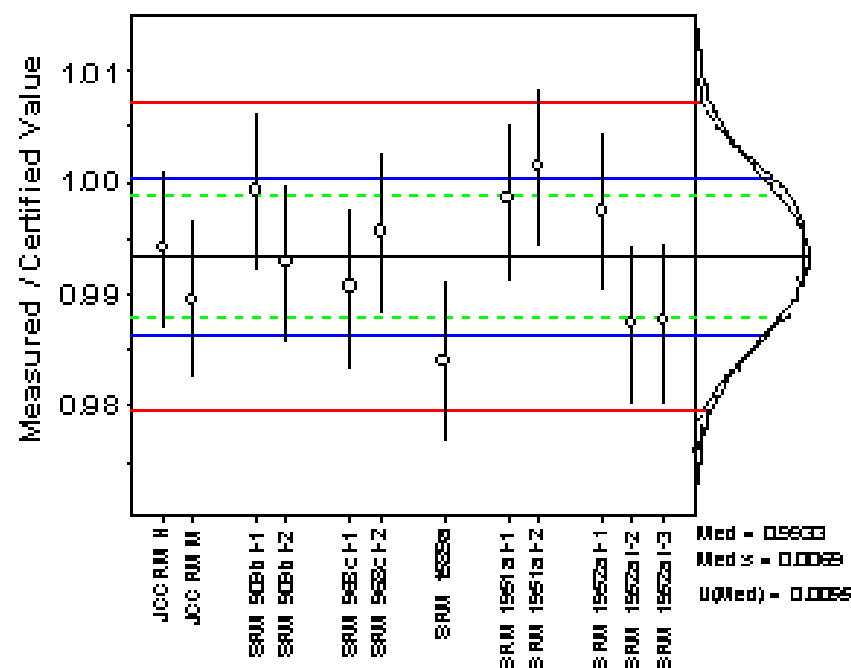
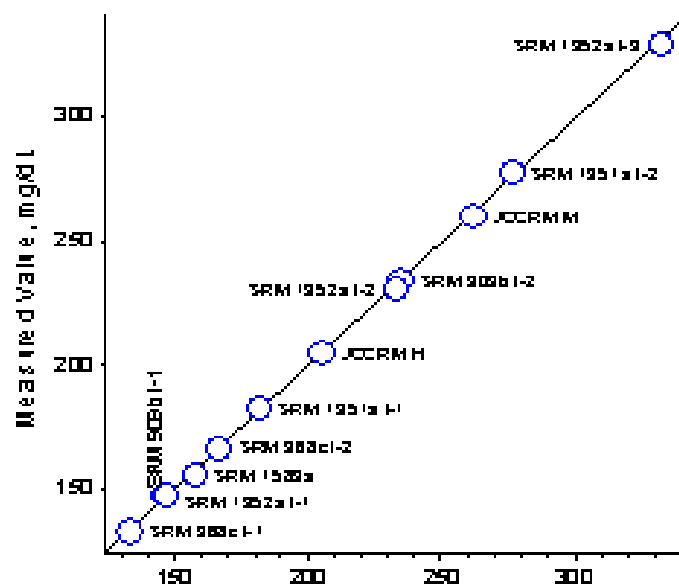
Drugs

Hormones

Coagulation Factors

CRM comparability studies

Comparison of “higher order” Cholesterol in Serum CRMs



⇒ CRM comparability independent of analyte level

The measured/certified ratios for this set of CRMs are:

- ~ normally distributed
- with a standard deviation of ~0.7 %