

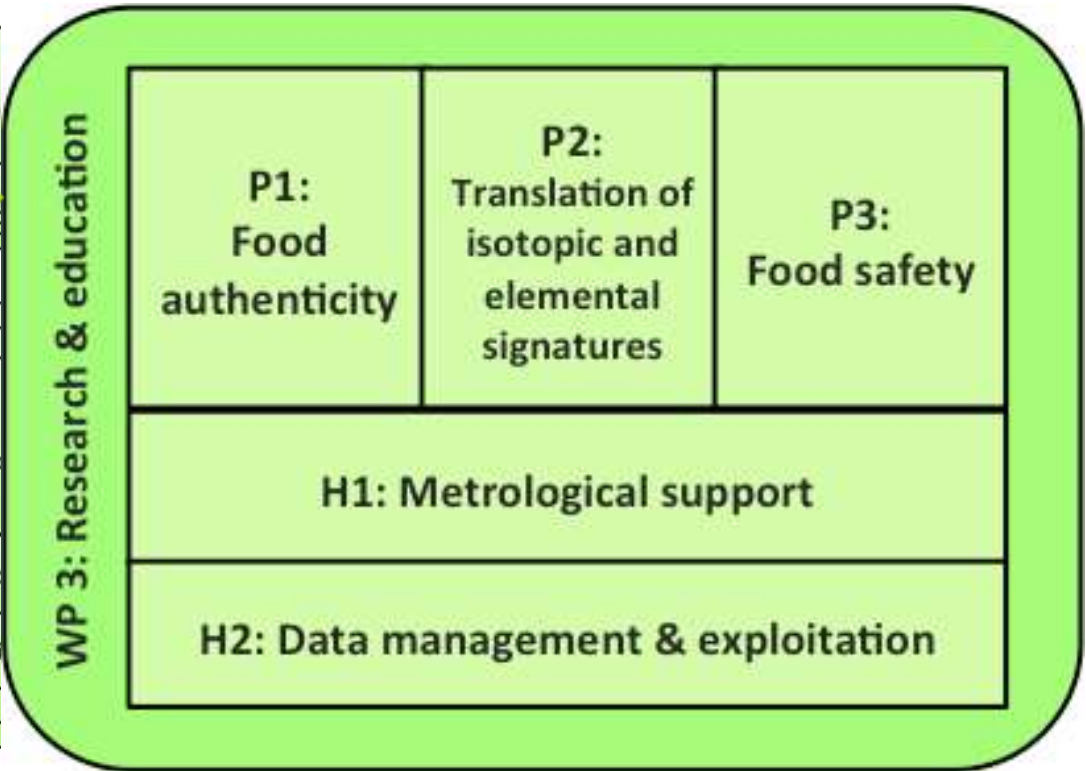
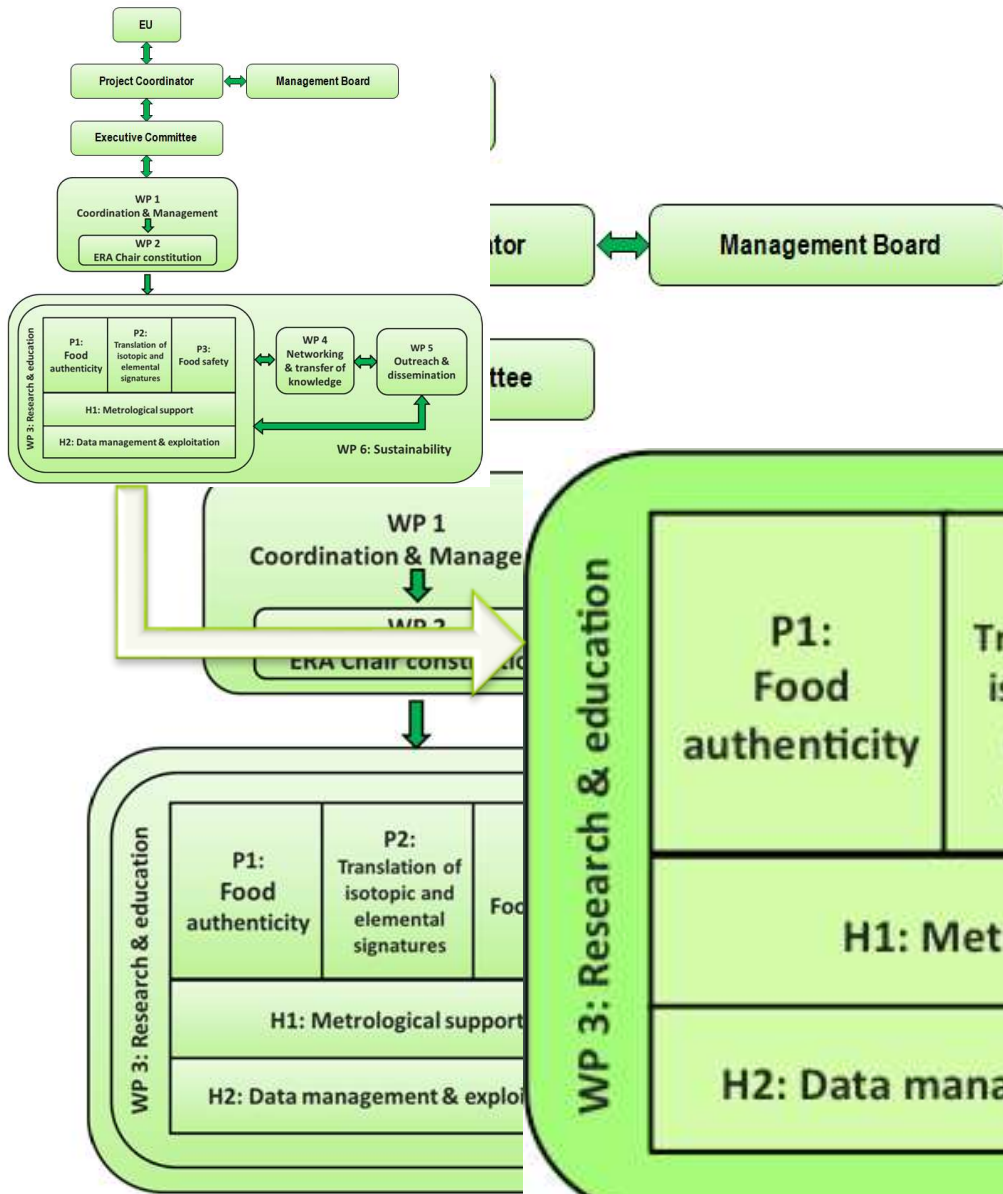


Safety
Quality
Traceability

H1: Metrological support

Milena Horvat, Polona Vreča,
Tea Zuliani, Radojko
Jačimovič, Radmila Milačič





Basic requirement

How to ensure appropriate measurements and monitor changes in time and space?

Analytical measurements need to be **comparable** in time and space

traceability is the best way to achieve this

Chemical metrology

Sampling + Processing + Measurement = Result

**Representative
Appropriate
Contamination
Stability
Handling**

**Dissolution
Extraction
Dilution**

**Comparison to SI units
or conventional scale**

± uncertainty

Measurement traceability

VIM definition

“... the 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**.”

Reference Materials – ISO/REMCO definition, 2005

“Material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process.”

- *generic term*
- *property can be quantitative or qualitative*
- *uses: calibration, assessment of a measurement procedure, assigning values to other materials and QC*
- *can be used for a single purpose in a given measurement.*



Certified Reference Materials —

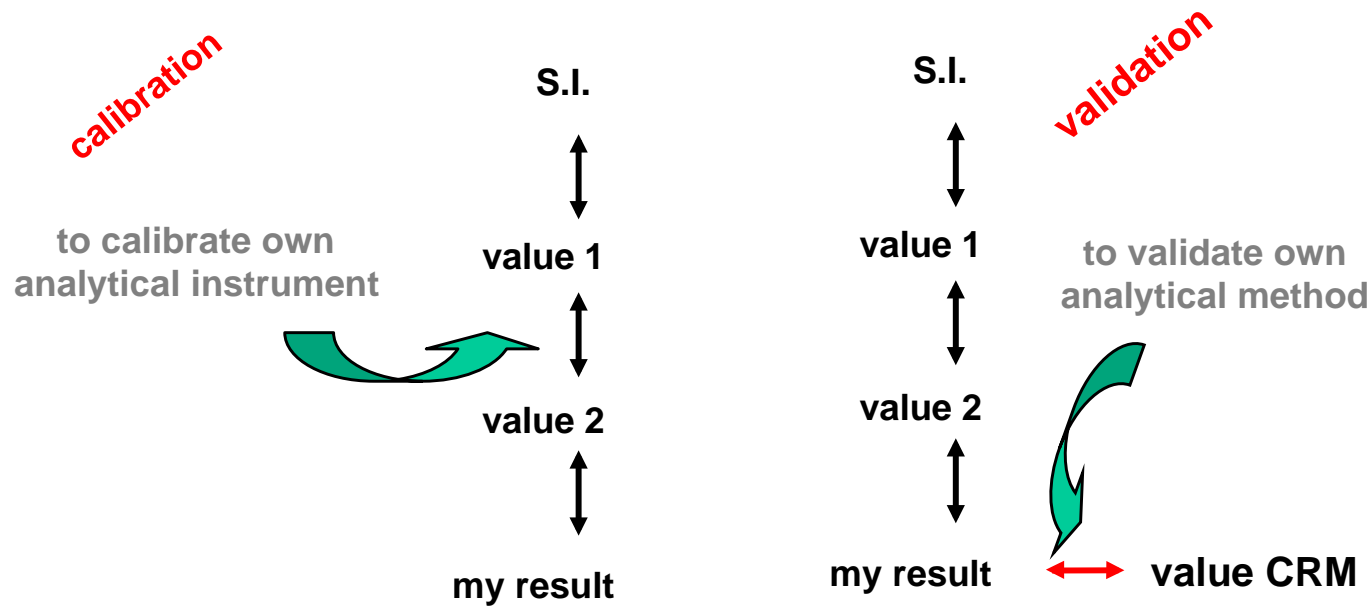
ISO/REMCO definition, 2005

“An RM characterized by a **metrologically valid procedure** for one or more specified properties, accompanied by a certificate that states the **value** of the specified property, its associated **uncertainty**, and a statement of metrological **traceability**.”

Metrologically valid procedures: ISO Guide 34 and 35

Contents in the certificate: ISO Guide 31.

How CRMs are Used in Terms of Traceability?



ASSURANCE OF MEASUREMENT COMPARABILITY (I)

- Certified value in CRM is:
the property value and its uncertainty


User:

$$U_c = \sqrt{U_m^2 + U_{mat}^2 + u_{CRM}^2}$$


ASSURANCE OF MEASUREMENT COMPARABILITY (II)

Uncertainty of the CRM is one of the strong component in the combined uncertainty of the user`s results

$$U_{\text{user's results}} > U_{\text{CRM}}$$



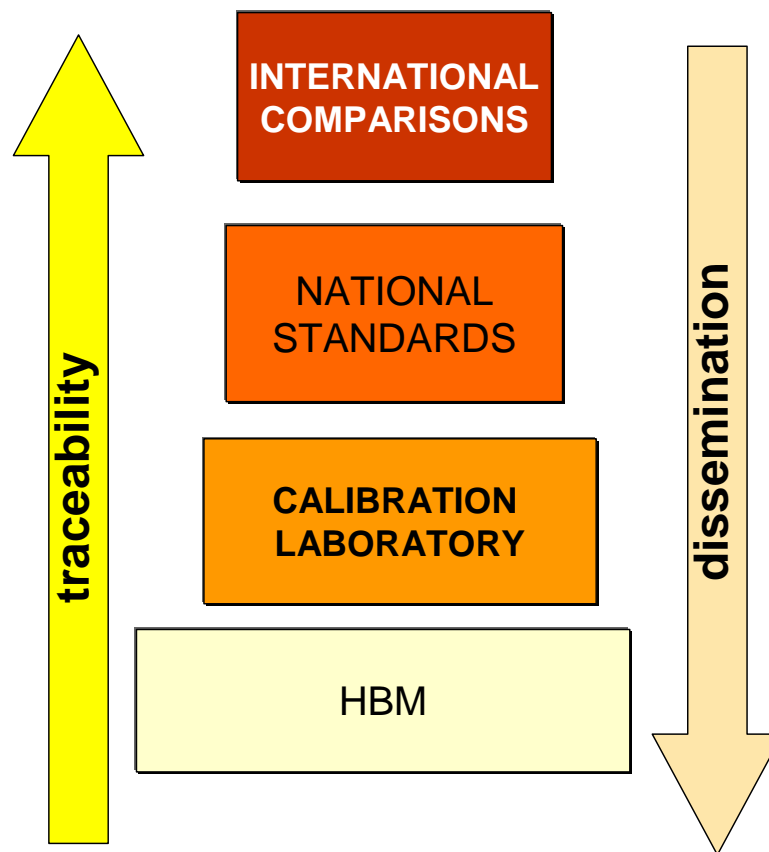
ONLY direct matrix and concentration match of the sample and CRM (and CRM as calibrant) can be used to demonstrate accuracy and traceability



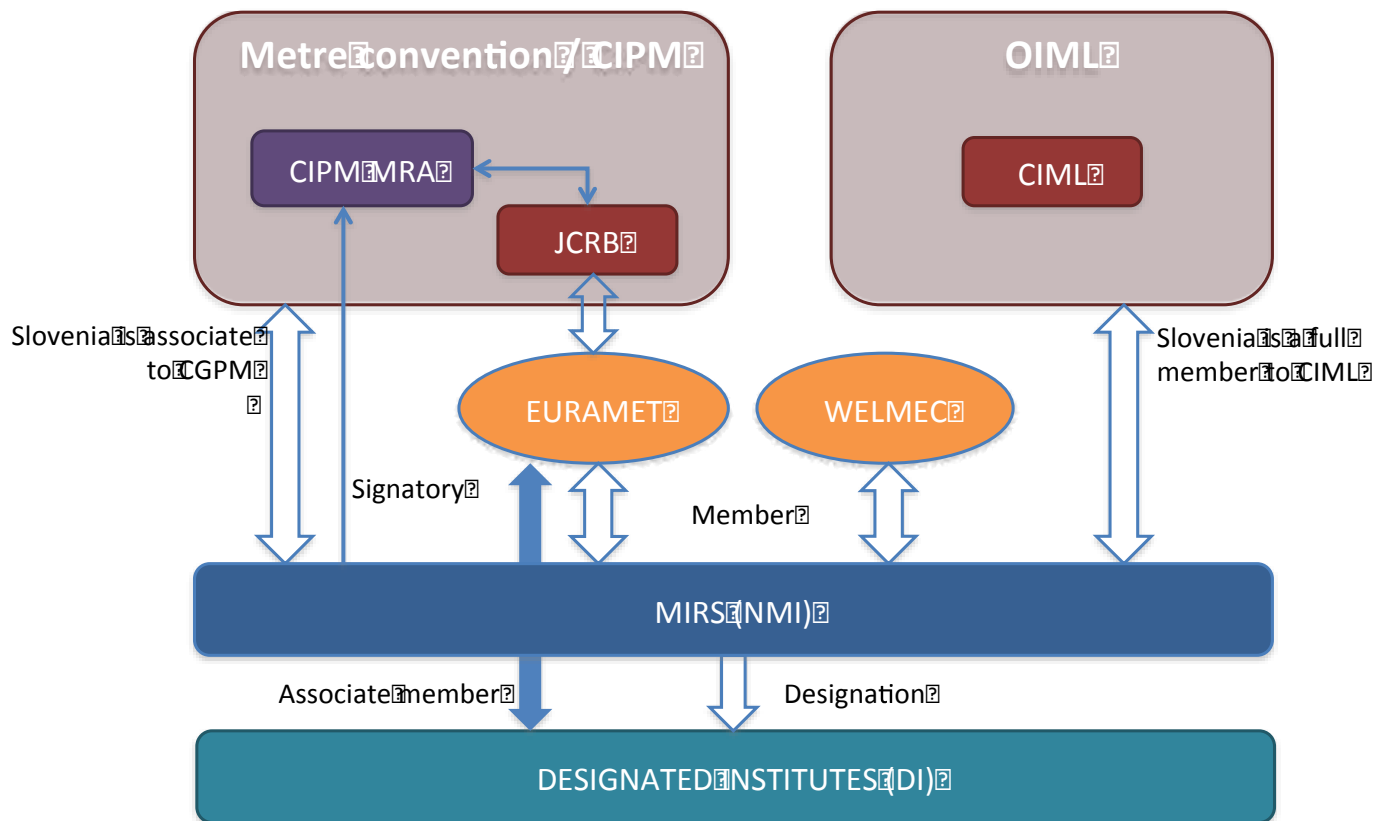
CRM producer MUST take intended use of CRM into consideration in the planning and preparation stages and PROVIDE instructions on how to establish traceability to the stated reference

What do we need?

- Production of accurate data **in time and space**
- Current reference materials are **not sufficient** to cover the needs (matrix, concentration range, uncertainty, etc...)
- **Hierarchical metrology structure of technical infrastructure** needs to be developed in practise for the succesfull HBM (national, regional, global)



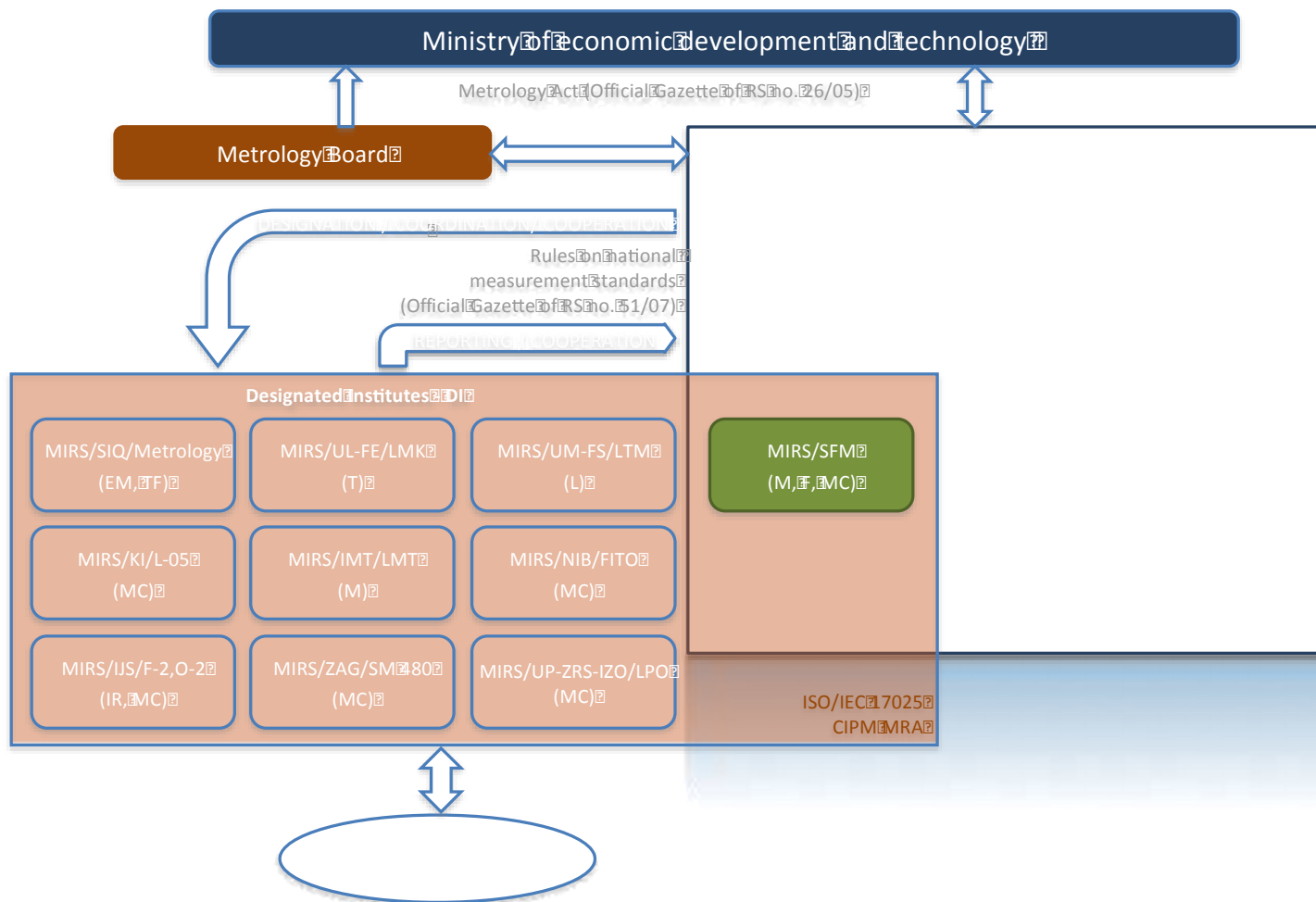
NMS – Traceability International cooperation





REPUBLIC OF SLOVENIA
MINISTRY OF ECONOMIC DEVELOPMENT AND TECHNOLOGY
METROLOGY INSTITUTE OF THE REPUBLIC OF SLOVENIA

NMS – Traceability National hierarchy



Slovenian metrology system

Fields of measurements



| Institution | Acronym | Field |
|---|---------------------|---|
| Metrology institute of the Republic of Slovenia | MIRS | Mass Volume Amount of Substance, Metal and Metal Alloys |
| Slovenian institute of Quality and Metrology | MIRS/SIQ/Metrology | Electricity and Magnetism Time and Frequency |
| University of Ljubljana, Faculty of Electrical Engineering | MIRS/UL-FE/LMK | Thermometry |
| University of Maribor, Faculty of Mechanical Engineering | MIRS/UM-FS/LTM | Length |
| Institute of metals and technology | MIRS/IMT/LMT | Mass and related quantities, Pressure |
| Jožef Stefan Institute | MIRS/IJS/F-2,O-2 | Ionising Radiation Amount of Substance, Sediments, soils, ores, and particulates |
| National institute of Chemistry | MIRS/KI/L05 | Amount of Substance, Water |
| National Institute of Biology | MIRS/NIB/FITO | Amount of Substance, Food, GMO |
| University of Primorska, Science and Research Centre of Koper | MIRS/UP-ZRS-IZO/LPO | Amount of Substance, Food, Nutritional constituents |
| Slovenian National Building and Civil Engineering Institute | MIRS/ZAG/SM 480 | Amount of Substance, Other materials |

Quality Management System at JSI/O-2



- September 2007: Implementation of QMS according to SIST EN ISO/IEC 17025
- October 2007: Application for accreditation of 7 methods to the Slovenian accreditation (SA):
 - Determination of strontium by beta counting
 - Determination of tritium with liquid scintillation counting
 - Determination of ^{14}C in alkaline solution
 - Determining elemental composition of environmental samples using k0-INAA
 - Determination of mercury in water
 - Determination of elements in water by ICP-MS
 - Determination of organotin compounds in water with gas chromatography

Quality Management System at JSI/O-2



- February 2009: QMS presented at EURAMET TC-Q
- April 2009: QMS confirmed by EURAMET TC-Q
- June 2009: 7 methods accredited according to SIST EN ISO/IEC 17025 – Accreditation certificate LP-090



Quality Management System at JSI/O-2



- December 2009: Application for DI for amount of substance
- February 2010: Designated as DI by MIRS in for:
 - Amount of substance (mass fraction of elements): sediments, soils, ores, and particulates; sewage sludge
- 2009 – 2014: Regular annual assessments (internal, external) passed
- April 2014: Re-evaluation of QMS at EURAMET TC-Q – successful

CMC - Calibration and Measurement Capability

- *CMC is a calibration and measurement capability available to customers under normal conditions:*
 - *(a) as published in the BIPM key comparison database (KCDB) of the CIPM MRA; or*
 - *(b) as described in the laboratory's scope of accreditation granted by a signatory to the ILAC Arrangement."*

Participation in Key comparisons and Pilot studies during last 3 years

- CCQM-K30.1 Pb *in Wine*
- CCQM-K89 Trace and Essential Elements *in Herba Ecliptae* (As, Ca, Cd, Pb and Zn)
- APMP.QM-S5 Essential and Toxic Elements *in Seafood* (As, Cd, Fe and Zn)
- CCQM-K108 Determination of arsenic species, total As and Cd *in brown rice flour*
- CCQM-K106 Pb, As and Hg measurements in cosmetic (cream)
- CCQM-P12.2 Analysis of Pb, Fe, Cu and Cd *in Wine*
- CCQM-P128 Cosmetic (cream)



Problem

- Limited scope of designation in the BIPM database (only amount of substance in sediments, soils, ores, and particulates)

Solution

- Enlarge the scope of designation in the BIPM database to allow the expertise in metrology in chemistry, amount of substance, mass fraction of elements (***to add at least biological fluids and materials, and food***) of O-2 to be more effective in areas of need at the national and international level
- Organisation of ***KC and PT***

Organisation of Key Comparison and Pilot Study

○ Motivation

- Lack of soil matrix KC; particularly from non-perturbed/non-contaminated areas

○ KC (CCQM-K127) and PS (CCQM-P162) for toxic and trace element analysis in soil

○ Time schedule

- Call for participation: October, 2014
- Deadline for registration of participation: December, 2014
- Distribution of samples: January, 2015
- Deadline for delivery of results: August, 2015
- First discussion on results: October, 2015