

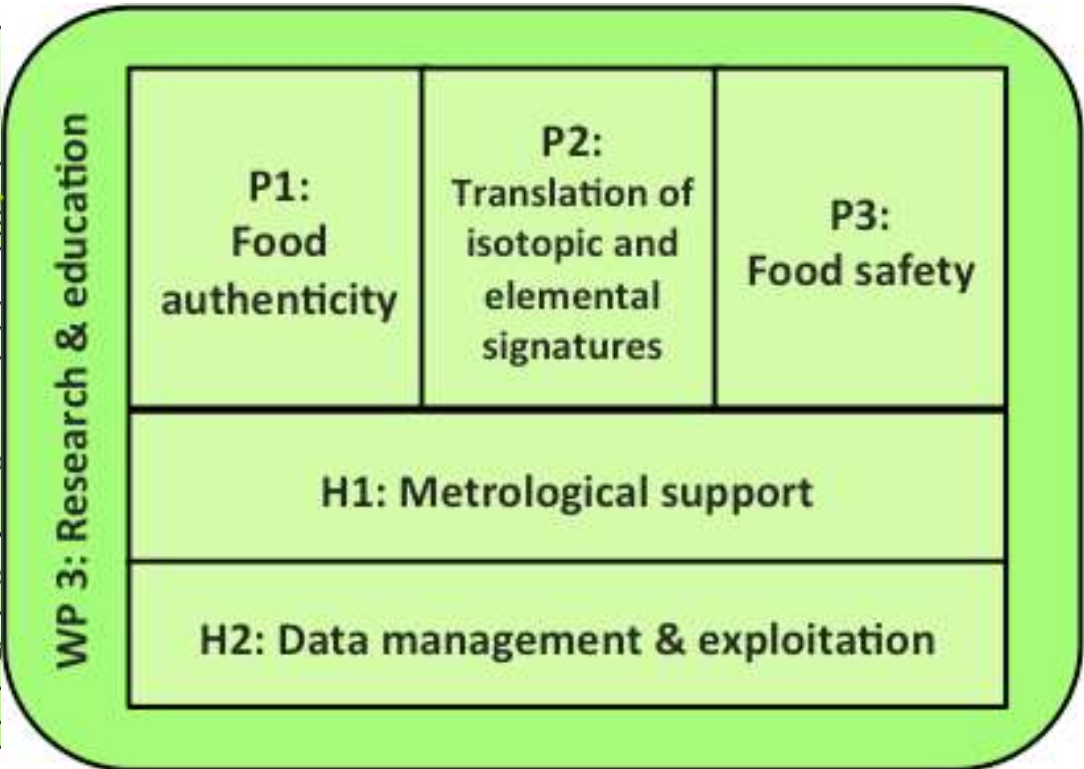
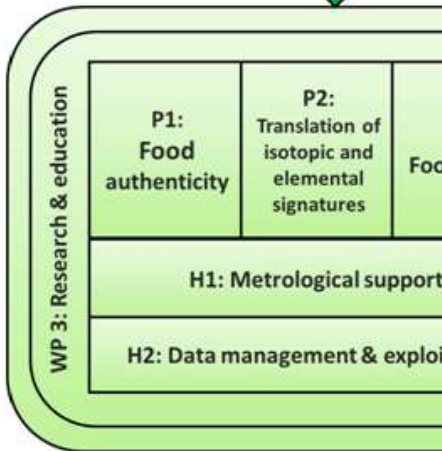
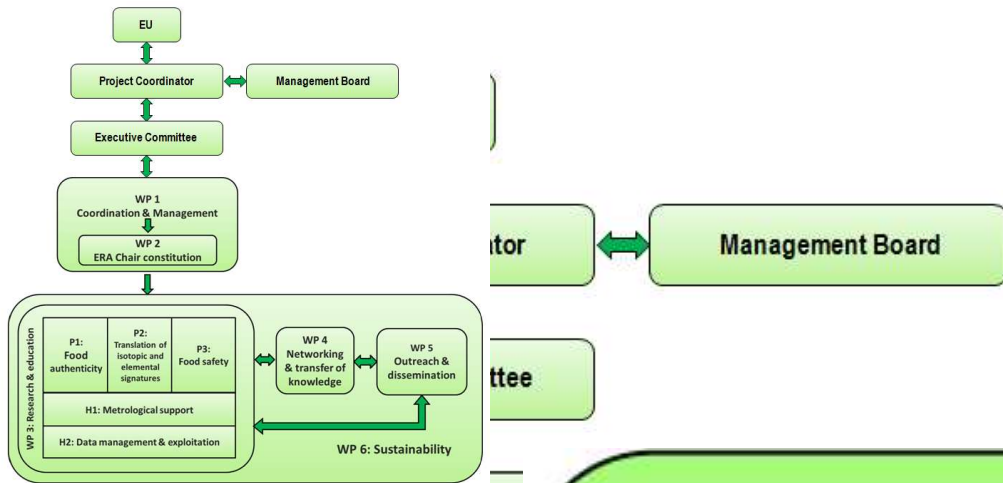


**Safety**  
**Quality**  
**Traceability**

# H1: Metrological support

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# 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**

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**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 —

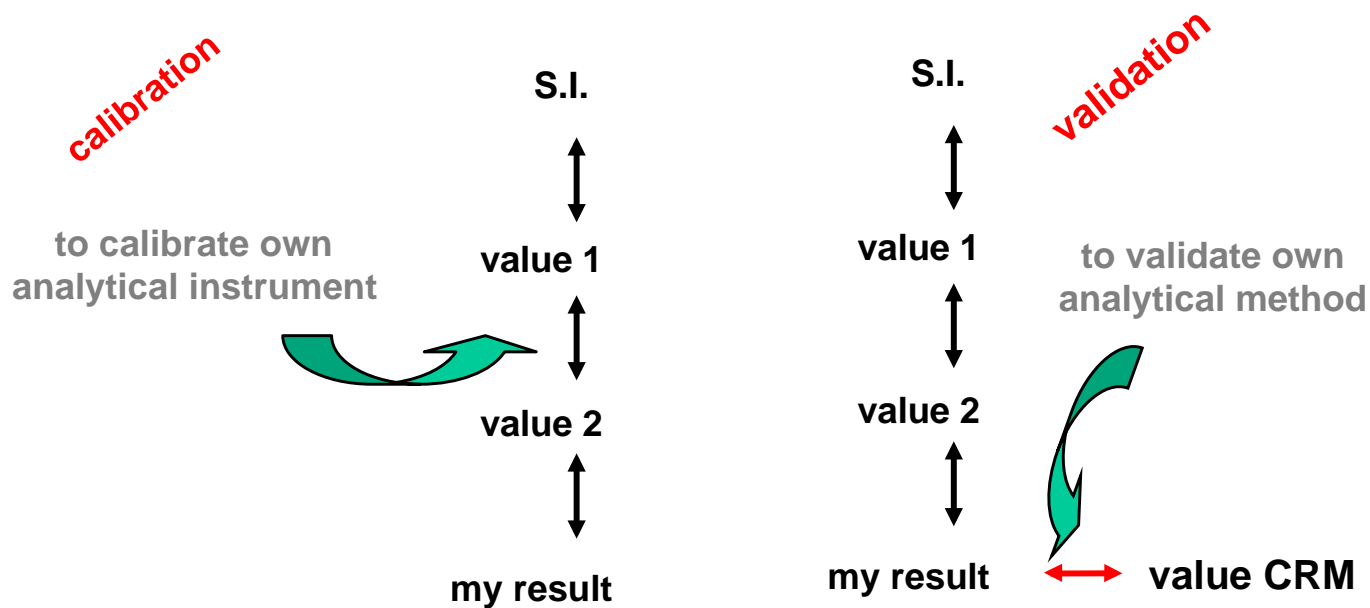
ISO/REMCO definition, 2005

“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

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



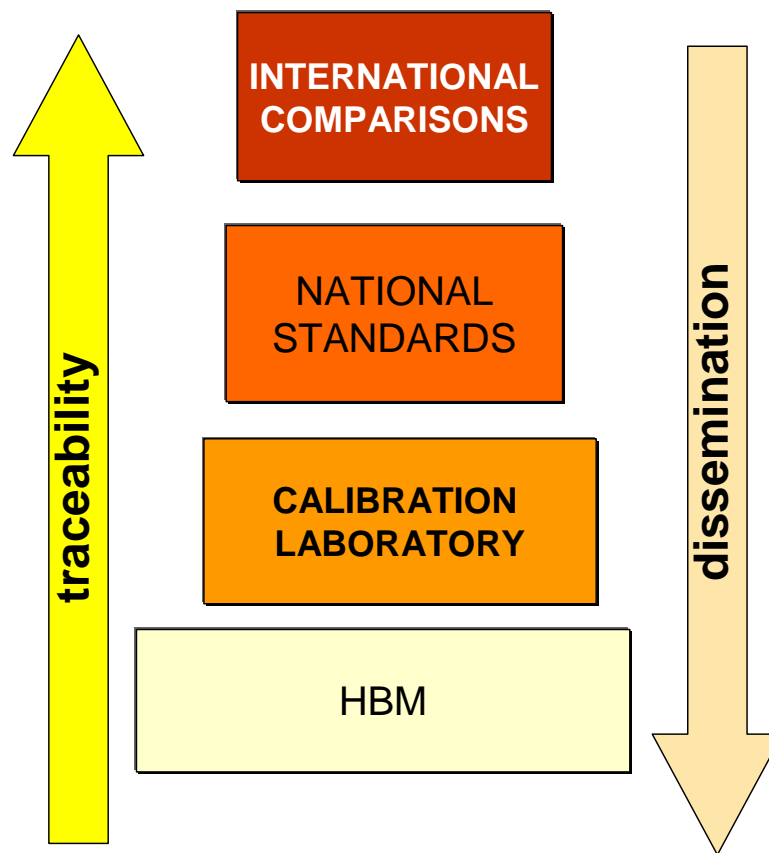
***ONLY direct matrix and concentration match of the sample and CRM (and CRM as calibrant) can be used to demonstrate accuracy and traceability***



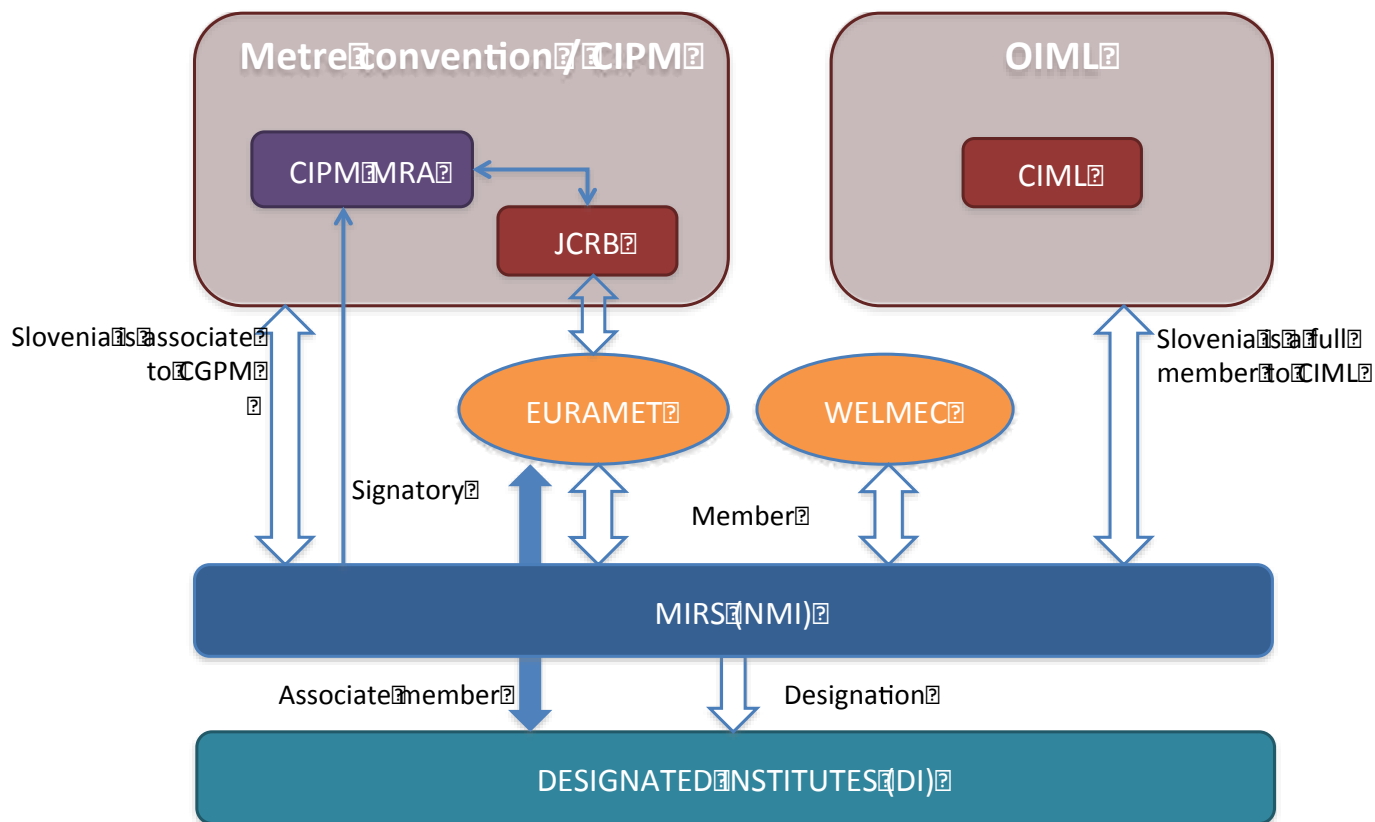
***CRM producer MUST take intended use of CRM into consideration in the planning and preparation stages and PROVIDE instructions on how to establish traceability to the stated reference***

# 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)

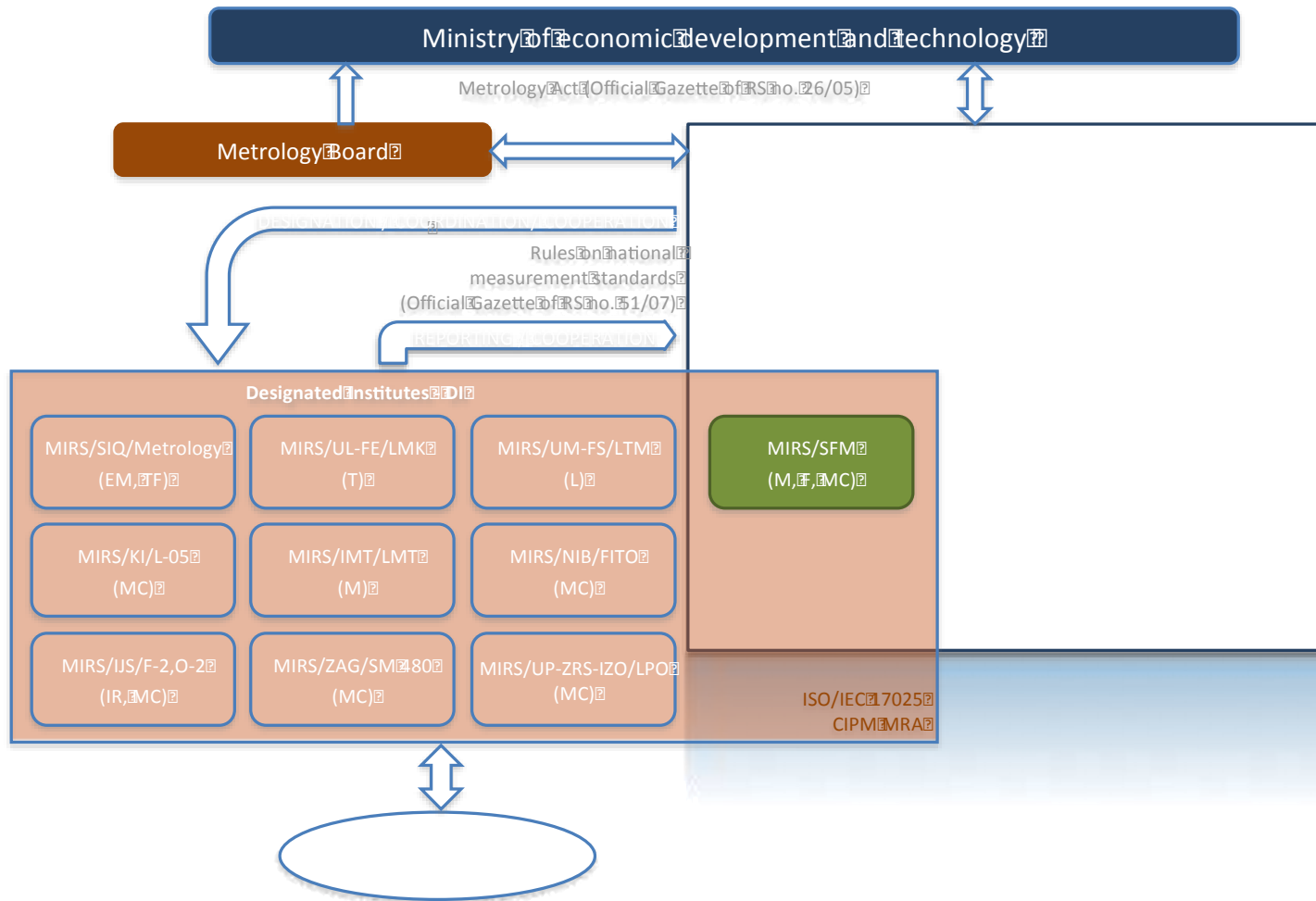


# NMS – Traceability International cooperation





# 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  $^{14}\text{C}$  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