ISPRA/ARPA/APPA system Reference materials, method harmonization laboratory accreditation

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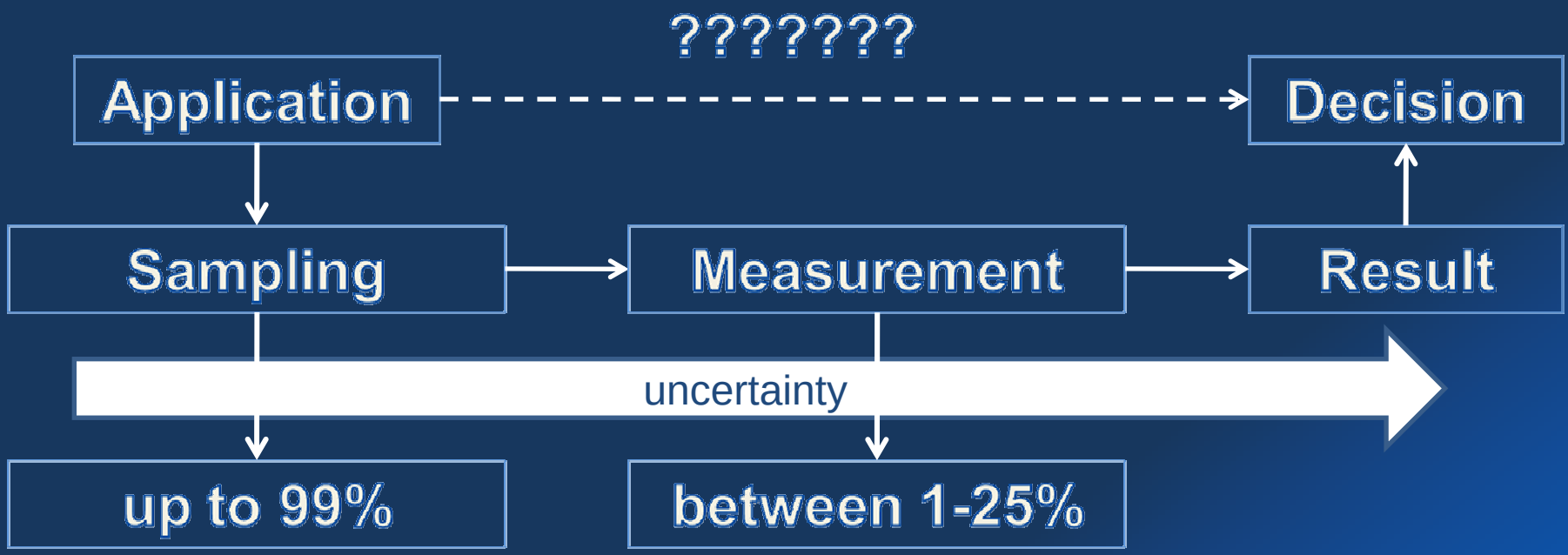
ARPA SICILIA General Director

Monitoring and control activities, in support of the governamental decisional process, are a priority within the Environmental Agency system

Analytical result accuracy (trueness and precision) is an indispensable factor assuring decision adequacy

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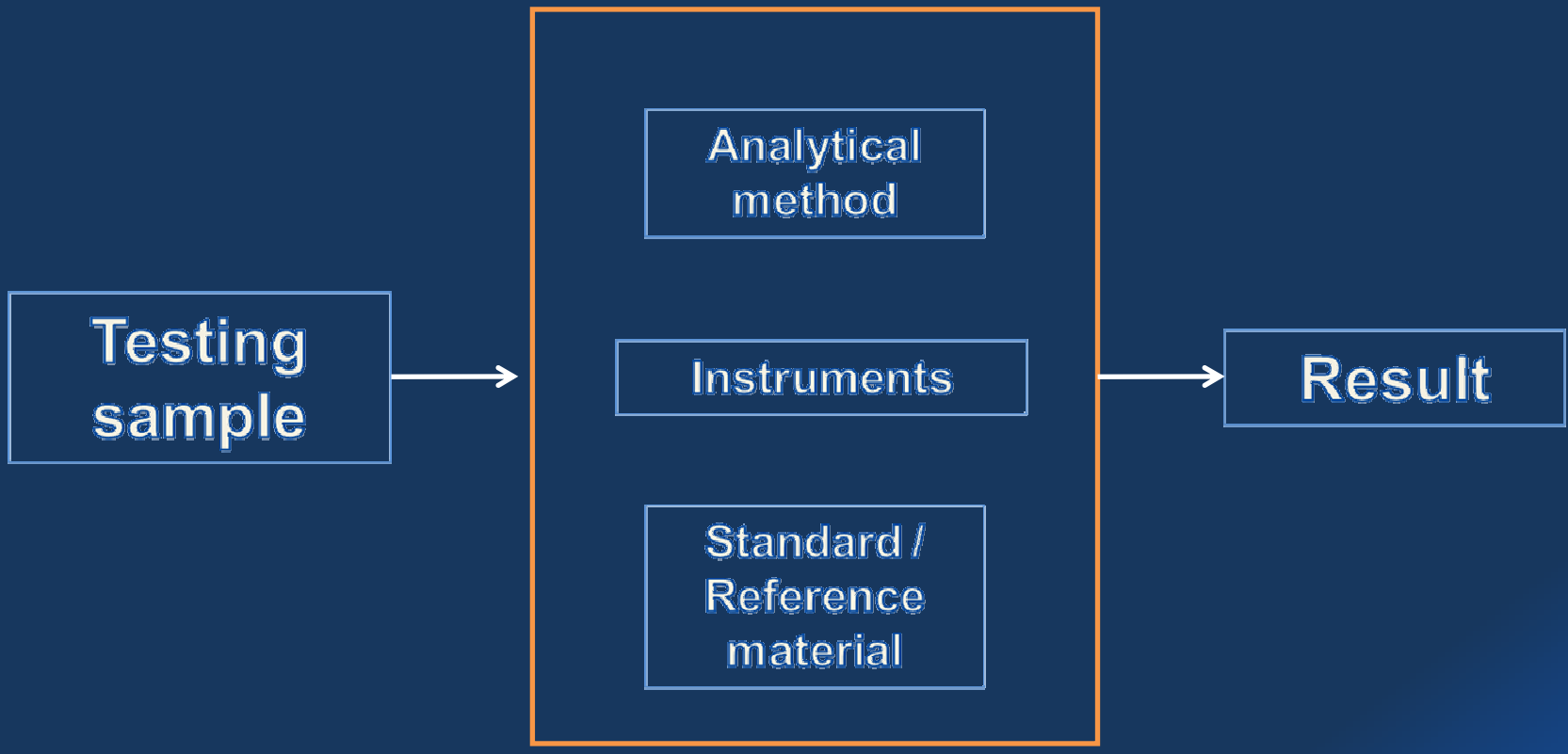
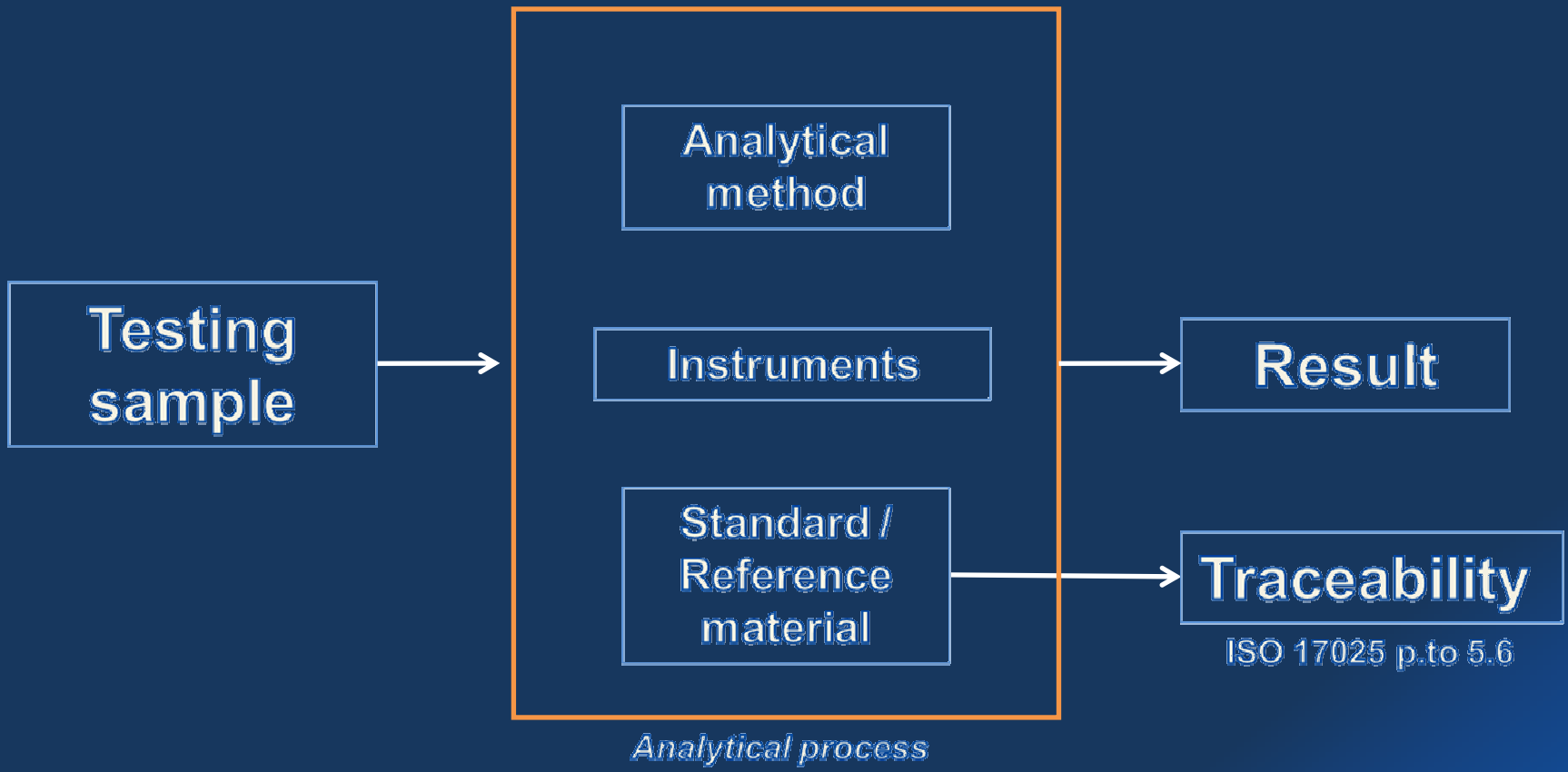
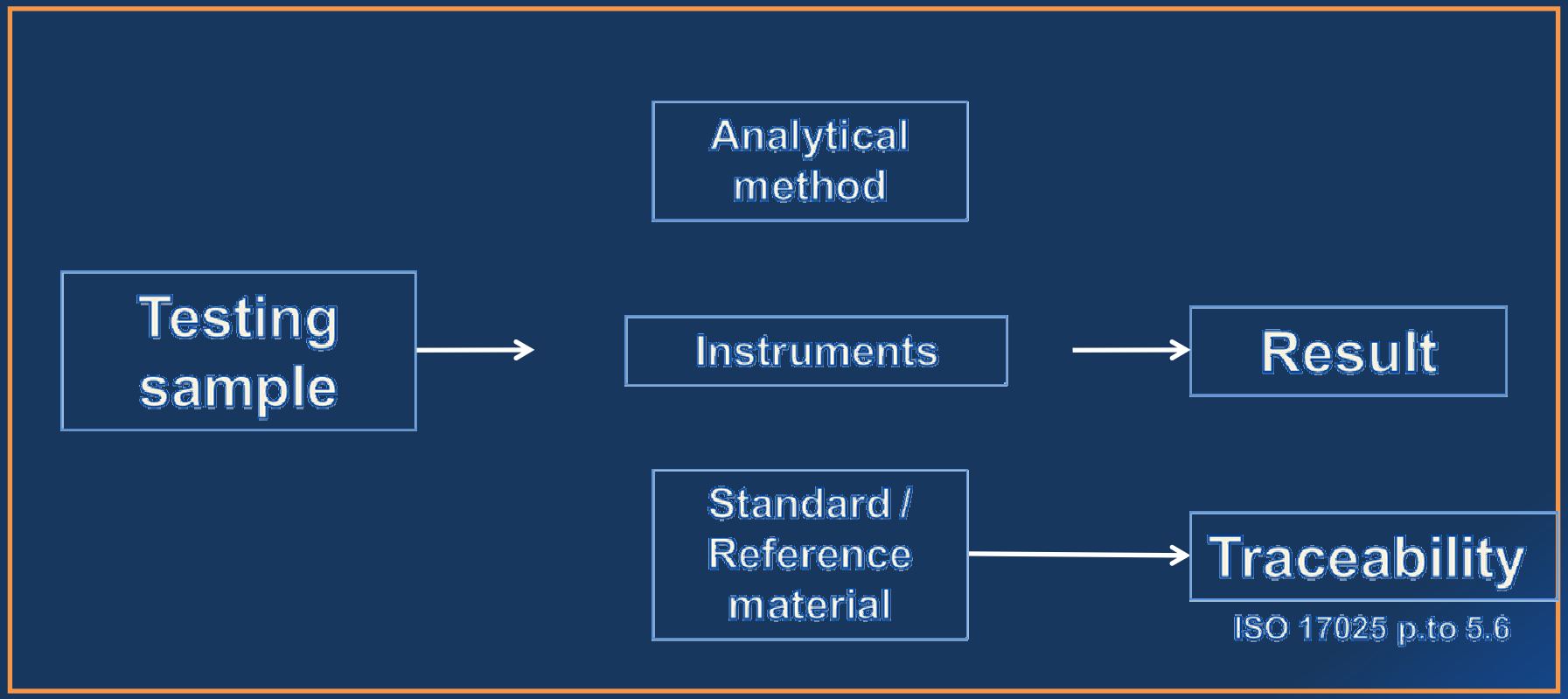
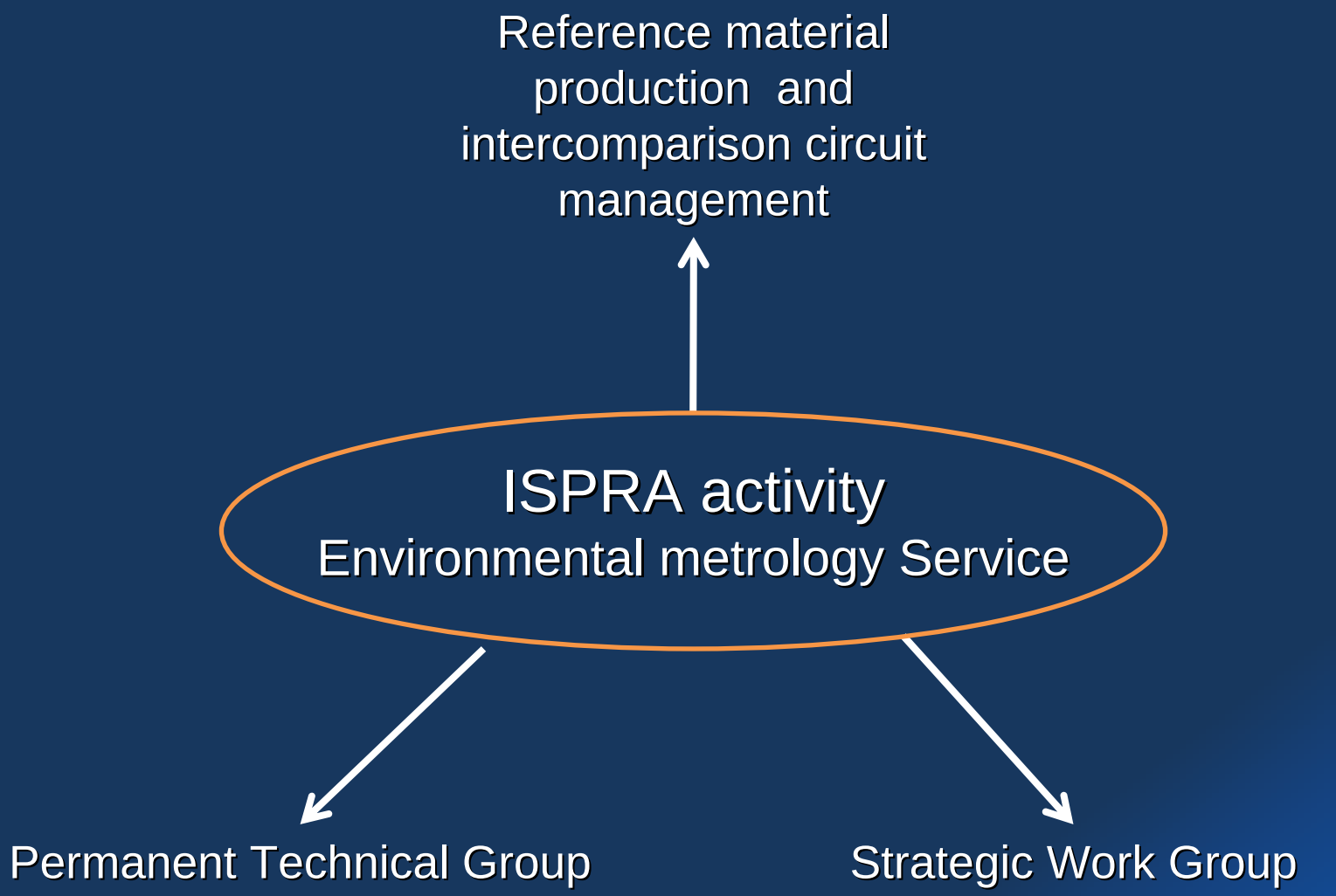
Testing sample
Result



Analytical process


Testing sample
Analytical method
Instruments
Standard / Reference material
Result
Traceability
ISO 17025 p.to 5.6
Analytical process


Analytical method
Testing sample
Instruments
Result
Standard / Reference material
Traceability
ISO 17025 p.to 5.6
Analytical process
Reference material
Quality assurance
ISO 17025 p.to 5.9


Reference material production and intercomparison circuit management
ISPRA activity
Environmental metrology Service
Permanent Technical Group
Strategic Work Group

ISPRA's activity - Environmental metrology Service

Permanent Technical Group: ongoing monitoring of Environmental Agency laboratory data quality

- Procedures for intercomparison circuit management
- Uncertainty decisional rules
- …..

Strategic Work Group: Support of an integrated network of analytical activities, within the Environmental Agency System, in order to develop synergies and improve effectiveness and efficiency

- Analytical method harmonization upon several environmental matrix
- In depth analysis of specific themes (sampling, uncertainties)
- Reference laboratories
- …..

ISPRA's activity - Environmental metrology Service

Through reference material production and characterization, carried out within its Environmental Metrology Laboratory, ISPRA has made it possible for the entire Environmental Agency Laboratory System to make comparisons in relation to the analytical results produced by intercomparison circuits.

ISPRA's activity - Environmental metrology Service

Intercomparison Circuit participation – Report L 93/01

	Chemistry	Biology	Physics
Partecipating structures	81	81	81
Partecipating structures with non nil analytical activity	75	65	41
Structures normally partecipating to Intercomparison Circuits	72	59	12
Percentage of laboratories attending to Intercomparison Circuit (of total n° laboratories with non nil normal analytical activity)	96%	91%	29%
Total amount of samples analyzed by laboratories partecipating to intercomparison circuit	1282	1798	160
Analysed sample mean value per partecipating laboratory	17.8	30.5	13.3
Percentage of normal institutional analytical activities dedicated to Intercomparison Circuit	0.3%	0.7%	1%

ISPRA's activity - Environmental metrology Service

Use of Certificated Reference Material – Report L 93/01

	Chemistry	Biology	Physics
Partecipating structures (questionnaries)	81	81	81
Normal and non nil analytical activity	75	65	41
N° of laboratories acquiring CRM in matrix	42	19	1
Percentage of laboratories acquiring/using CRM (of the total n° of laboratories with Normal and non nil analytical activity)	56%	29%	2%
Total amount of CRM acquired by laboratories	510	274	3
Mean value of CRM acquired per laboratory	12.1	14.4	--

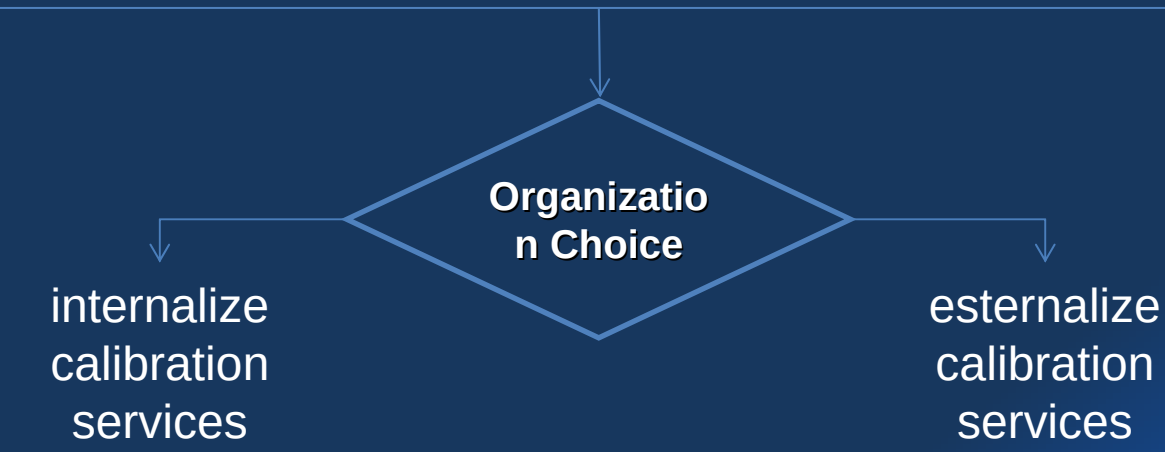
ARPA Sicilia's policy in traceability assurance in mass, temperature and volume measurements :

METROLOGICAL LABORATORY

UNI CEI EN ISO/IEC 17025:2005 p.to 5.6

For calibration laboratories, [……] calibrations and measurements made by the laboratory are traceable to the International System of Units (SI)

For testing laboratories, *all equipment used for tests ad/or calibrations, …. having a significant effect on accuracy or validity of the results of the test, calibration or sampling shall be calibrated before being put into service.*



ARPA Sicilia's METROLOGICAL LABORATORY tasks

- Electronic balance calibration
- Conventional mass value calibration
- Temperature indicator calibration
- Thermostatic equipment characterization
- Micropipette calibration

Electronic balance calibration

Through mass standards calibrated by I.N.Ri.M. and work standards calibrated in situ, referring to the former, a periodical calibration is carried out of analytical and technical balances used within the 10 ARPA Sicilia's laboratoeries, in the range of 1mg÷2kg

TRACEABILITY

- Mass national standard
- Working standard I.N.Ri.M.
- First line standard ARPA Sicilia
- Working standard ARPA Sicilia
- travelling standard ARPA Sicilia

Conventional mass value calibration

Our lab carries out conventional mass value calibration in the range of 1mg-2kg class E2 and below. Mass samples, traceable to National Mass Standard, are used by our 10 laboratories for periodical balance verifications undertaken between each calibration.

max 5g d=10^{-7}g
50.000.000 divisions

max 220g d=10^{-6}g
200.000.000 divisions

max 2000g d=10^{-4}g
20.000.000 divisions

Thermometric indicator calibration

Fundamental for:

- Proper preservation of samples subjected to analysis, reference material etc..
- Carrying out eco-toxicological or microbiological tests
- Controlling processes where temperature is the subject of measurement (purification and extraction steps during chemical tests, emission tests, in situ measurements, etc)

Thermometric indicator calibration

Thermometric indicators (RTD Pt100 4w), used as work and traveling standards, are calibrated in situ via first line temperature standard - calibrated by I.N.Ri.M.,- using highly stable and uniform thermostatic baths (range of -40 ÷ +250 °C)

TRACEABILITY

temperature national standard

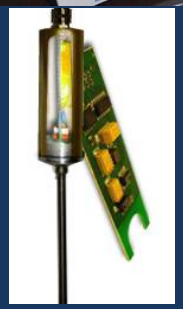
Working standard I.N.Ri.M.

First line standard ARPA Sicilia

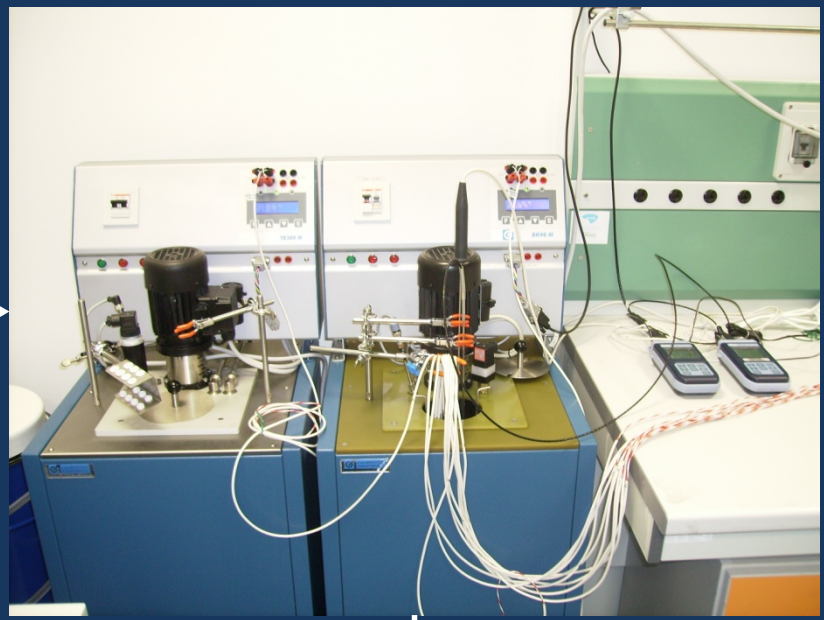
Working standard ARPA Sicilia

travelling standard ARPA Sicilia

Thermometric indicator calibration



first line standard



Monitoring thermostatic equipments

lab temperature indicator calibration

data logger calibration

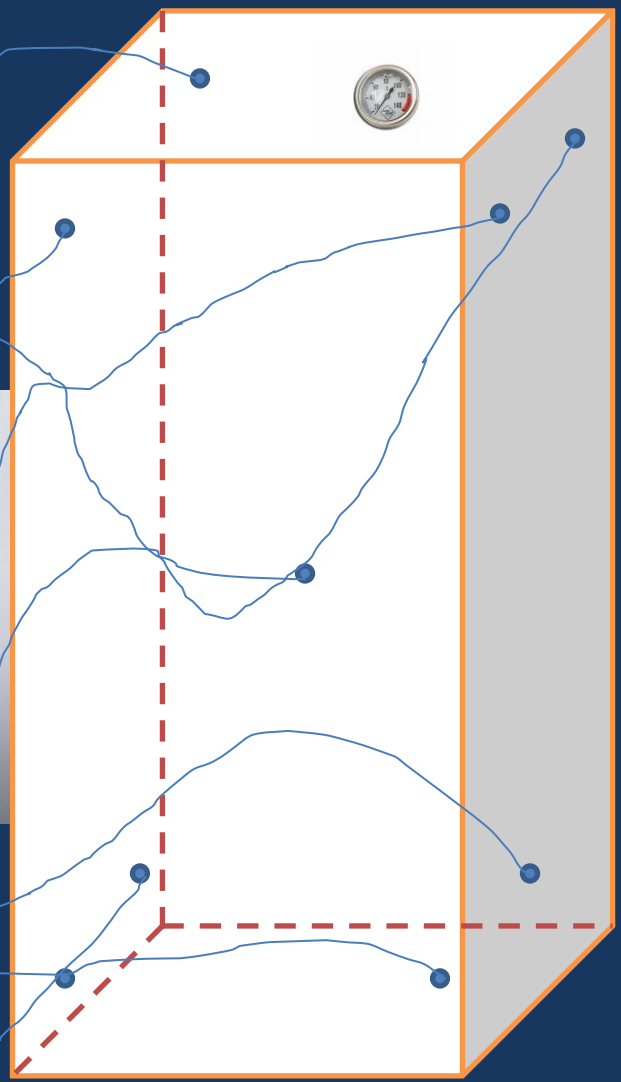
Temperature measurement instrument control

Characterization thermostatic equipment

STABILITY ?

Time variation

Afnor NF X 15-140



UNIFORMITY ?

Internal volume variation

At least 9 thermo-resistences 4w simultaneously record the temperature within the instrument working volume. Gathered data allow evaluation of thermostat stability and uniformity, together with other useful parametres for a more adequate usage of the instrument

Micropipette calibration

As they are fundamental in lab activities, micropipettes need to be calibrated in the same accurate manner as more complex instruments

Calibration is carried out by mass determination according to ISO 8655 prescriptions, in order to calculate the volume using weighed mass and density of the used liquid

Accessories such as evaporation traps, are used to minimize influence factors, especially when small volumes are considered (v<10ul)

FUTURE STEPS

After Calibration procedures have been tested and applied inside ARPA Sicilia's laboratories, the following steps will be taken:

1) Quality System documents are under review prior to application for accreditation to the Competent Body

2) Accreditation of balance, mass, thermometric indicator, thermostatic equipment, micropipette calibration

3) Calibration services will be fully available within the ISPRA / ARPA / APPA laboratory system