INTERNATIONAL
COOPERATION

Co-operation with the NMIs regarding PTs in calibration area and accreditation of PTs schemes

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April 2021

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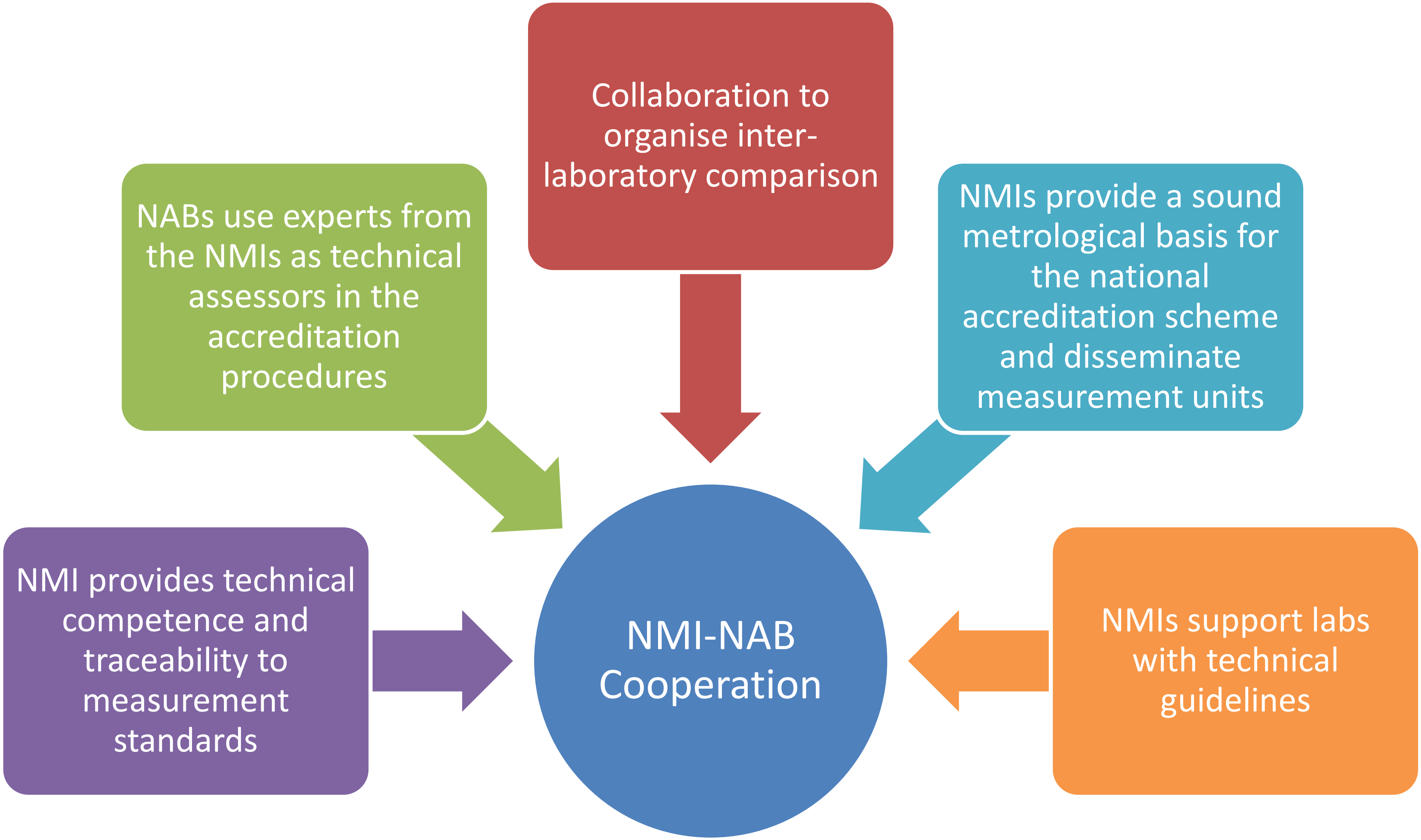
- Key challenges in a calibration PT
  - ✓ Homogeneity
  - ✓ Stability
  - ✓ Reference Values
  - ✓ Evaluation of results

Close Cooperation between NAB and NIM



Contribution of NIMs to PTs

Provision of Traceability to participants- **BIAS!**

Technical Assessors for accreditation of PTPs

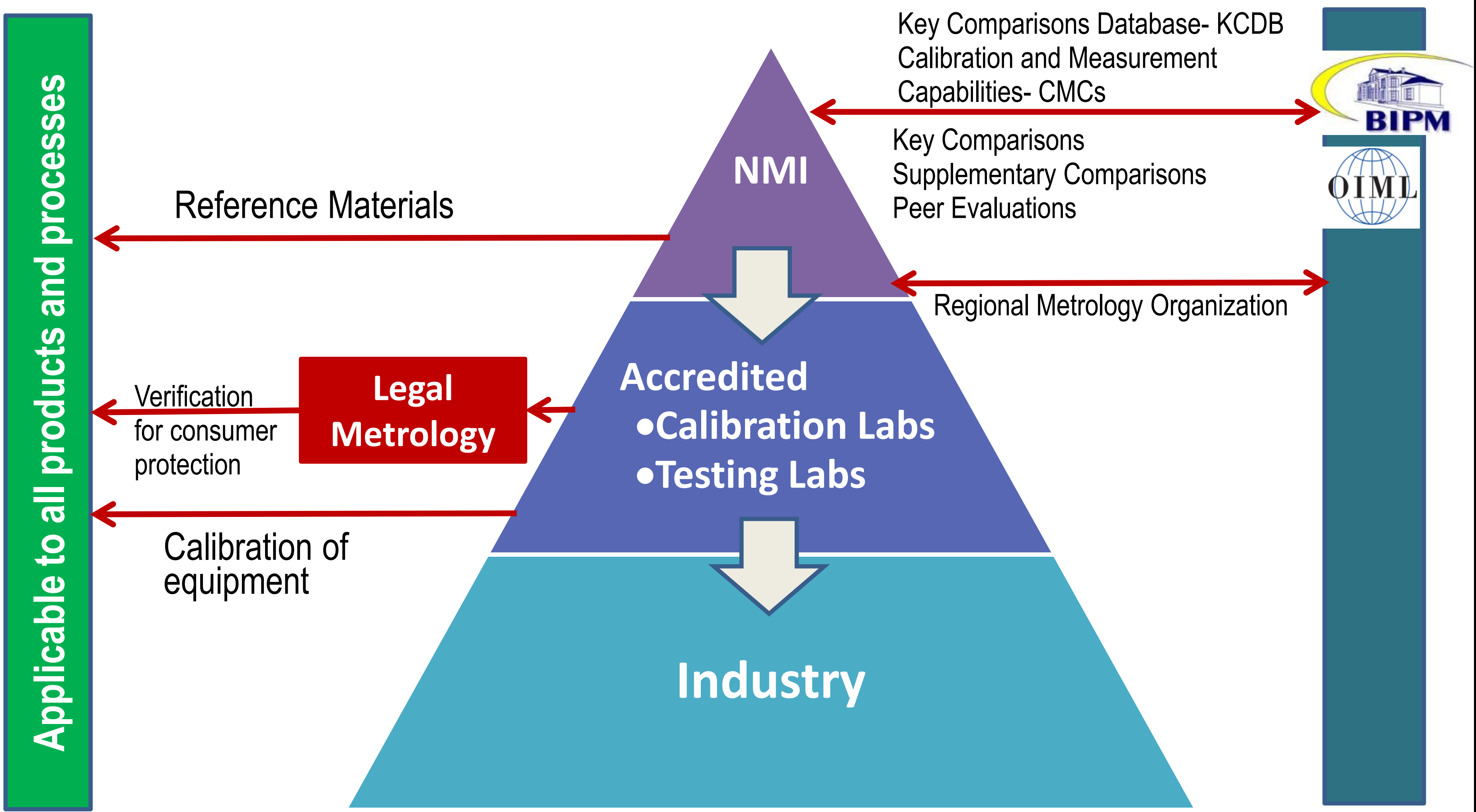
Organization of PTs

Support for Audits during assessments

Provision of Reference Values

Support for technical issues (estimation of reference values, uncertainty, performance statistics

Traceability to Participants and of the Reference Value



Support for Audits during assessments

- Laboratories receive a test item with accurately determined characteristics (calibrated by the NIM), which is to be calibrated in the context of an accreditation process.
- The test item is given either by the assessor or provided by the NMI.

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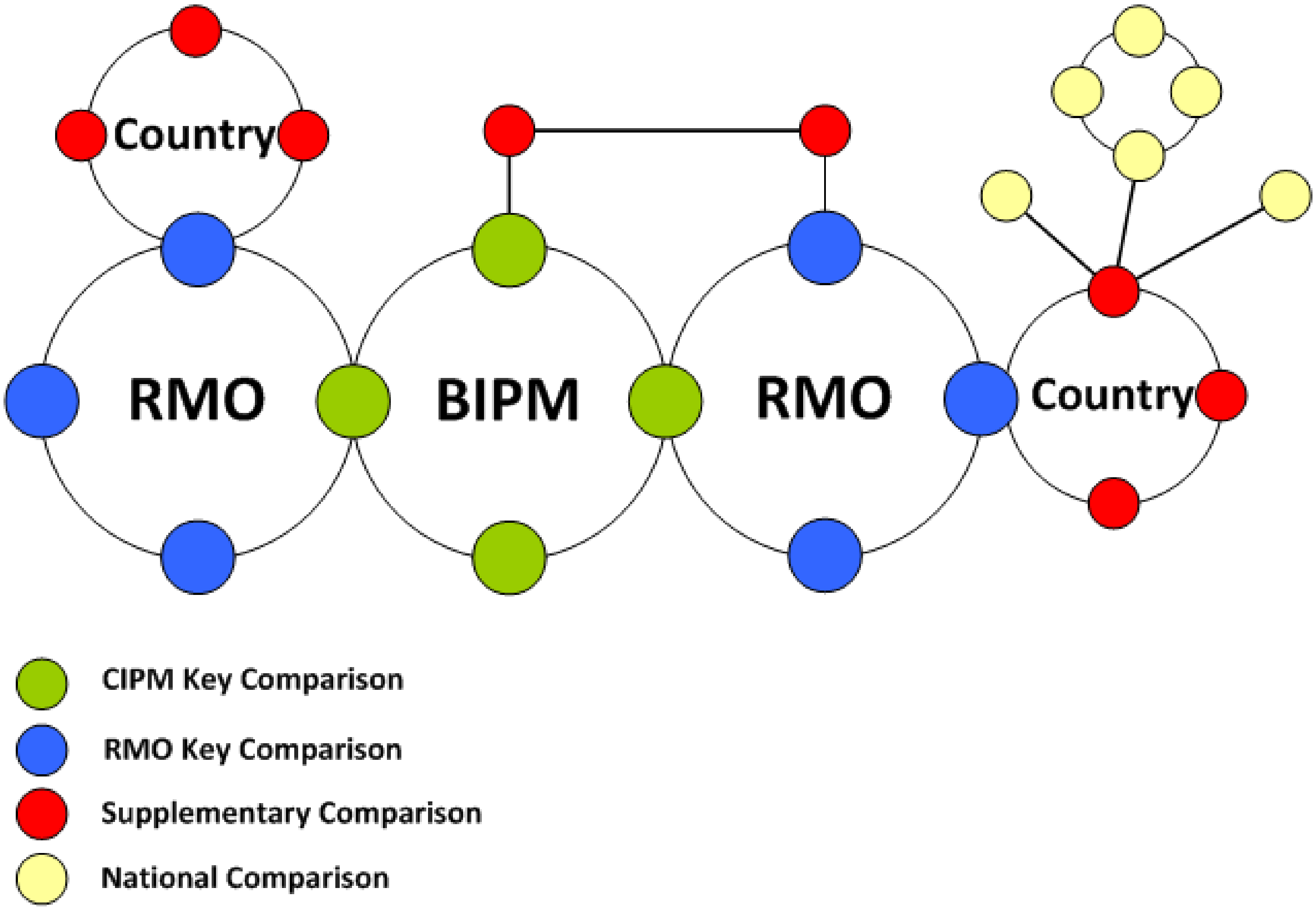
International System of Comparisons



Types of ILCs/PTs (calibration) organized by NIMs

Multilateral or Bilateral:
• The NMI can act as the reference laboratory only if relevant CMCs have been already published in the BIPM-KCDB. In such a case, the reference value is provided by the NMI • In any other case it should act as a participant. In this case, the reference value can be provided either by another NMI with relevant CMCs, or as a consensus of all participants • NMIs can provide reference values, while the PT is organized by a PT provider

Key Challenges in a Calibration PT

- Determine Homogeneity and Stability.
- Determine Assigned Value.
- Calculate Performance Statistics.
- Takes time to complete, stability of transfer standard is an issue, strict monitoring of circulation and allocated time)

Key Challenges in a Calibration PT- Homogeneity & Stability

ISO/IEC 17043-4.4.3.1: Criteria shall be established based on the effect that inhomogeneity and instability will have on the evaluation of participants' performance.

NOTE 1 Requirements in subclause are intended to ensure that participants receive **comparable PT items which remain stable throughout the PT**. Careful planning, selection and shipping are required and testing is needed to confirm it.

NOTE 3 In some cases **PT items that are not sufficiently homogeneous or stable are the best available**. In this case, the uncertainties of assigned values or the evaluation of results should take account of this.

Key Challenges in a Calibration PT- Homogeneity & Stability

ISO/IEC 17043-4.4.3.4: PT items shall be stable to ensure that they will not undergo significant change throughout the PT (including storage, transport, handling). When this is not possible stability shall be quantified and included as an additional component in the measurement uncertainty of the assigned value of the PT item.

Key Challenges in a Calibration PT- Homogeneity & Stability

- The analogous situation in calibration is the distinction usually made between the uncertainty “at calibration” and that “in use”.
- Statements such as: “the reported expanded uncertainty contains no allowance for the long-term drift of the DUT, possible effects of transportation between laboratories nor measurement uncertainties in the user’s laboratory” on the calibration certificate attempt to warn the user that the actual measurement uncertainty when using the DUT in his laboratory will most certainly be underestimated if only the uncertainty on the calibration certificate is used.

Key Challenges in a Calibration PT- Homogeneity

- **Definition**: condition of being of uniform structure or composition with respect to one or more specified properties.
- This means that if a property results in a variance in the measurement then, homogeneity has not been completely realized.
- When an artifact (transfer standard) is used in a PT, its design must be such that the measurand is defined carefully, addressing all the properties of the artifact that can cause variability in the measurement results.

So HOMOGENEITY effects the precise DEFINITION OF THE MEASURAND

Key Challenges in a Calibration PT- Homogeneity

All Participants Measure the Same Artifact Under the Same Conditions

The majority of PTs for calibration laboratories involve the measurement of one or more artifacts under appropriately defined measurement conditions.

One such example is when the PT artifacts are two standard resistors.

Since all participants are measuring the same artifacts, additional measurements or statistical tests of the homogeneity of the artifacts are not required.

Key Challenges in a Calibration PT- Homogeneity

All Participants Measure the Same Artifact Under the Same Conditions

In order to ensure that:

All participants are measuring the same property (i.e. resistance) under the same conditions

+

Inhomogeneity is effectively eliminated in the measurement result

The measurand must be appropriately and explicitly defined for environmental effects:

The measurand of the PT is the electrical resistance of the artifacts ***at a temperature of 25 ºC and a pressure of 101325 Pa. The test current is not to exceed 10mA for the 100Ω resistor and 150µA for the 20Ω resistor respectively.***

Key Challenges in a Calibration PT- Homogeneity

All Participants Measure the Same Artifact Under the Same Conditions

There will still be some uncertainty associated with realizing the measurement at the defined conditions

+

The uncertainty in the measurement of T, P and I must be considered and included in the PT uncertainty analysis if they are significant

+

In some cases, the participants may not be able to perform measurements at the defined parameters

PT provider or participant: have to apply a correction to the measured value in order to correct for the known offset. Thus the uncertainty associated with the measurement of environmental parameters, and uncertainty of any correction, must be estimated and included in the overall uncertainty analysis if they are significant

Key Challenges in a Calibration PT- Homogeneity

All Participants Measure an Artifact at Different Locations

- Destructive test. Artifact is permanently indented, making it necessary to take measurements on different locations of the artifact.

- The reference laboratory makes two series of measurements at random locations across the artifact before and after circulation. The standard deviation of all measurements can act as an estimate of std. uncertainty of homogeneity for the artifact.

Key Challenges in a Calibration PT- Homogeneity

All Participants Measure an Artifact at Different Locations

The PTP should also evaluate each participant's data to determine if there is any detectible trend identification that could be related to the homogeneity of the artifacts.

Any review of participant data must result in a very clear trend and must show strong correspondence with the reference laboratory's opening and closing data before any conclusions about the homogeneity of the artifact can be made.

Key Challenges in a Calibration PT- Homogeneity

All Participants Measure an Artifact at Different Locations

The participants may not possess equipment capable of the same accuracy or resolution and therefore their assigned uncertainties will vary significantly

The participating laboratory staff may not have the same level of technical expertise as the reference laboratory staff.

All participant data should be considered suspect unless an obvious trend is observed

Key Challenges in a Calibration PT- Stability

- VIM defines stability as **the property of a measuring instrument, whereby its metrological properties remain constant in time**.
- ISO Guide 30: "**ability of a reference material, when stored under specified conditions to maintain a stated property value within specified limits for a specified period of time**".
- Both definitions are incomplete for the purpose of PT's for calibration laboratories.
- **DOES SUCH A SYSTEM EXIST?**
- **DOES IT HOLD FOR METROLOGICAL ARTIFACTS?**

Key Challenges in a Calibration PT- Stability

Alternative: since it is not always possible to accurately estimate the uncertainty due to a particular condition of stability, measure and evaluate the artifacts' stability during the PT round

Why? Because from experience it is known that despite efforts of PT scheme developers, effects of transportation can substantially increase the uncertainty associated with the reference laboratory measurement

Key Challenges in a Calibration PT- Stability

During the PT design phase (providers responsibility):

Artifact stability should be considered and its magnitude estimated and compared with other uncertainty components of reference lab

It should be judged if the stability uncertainty is suitable for validating participants' CMC's.

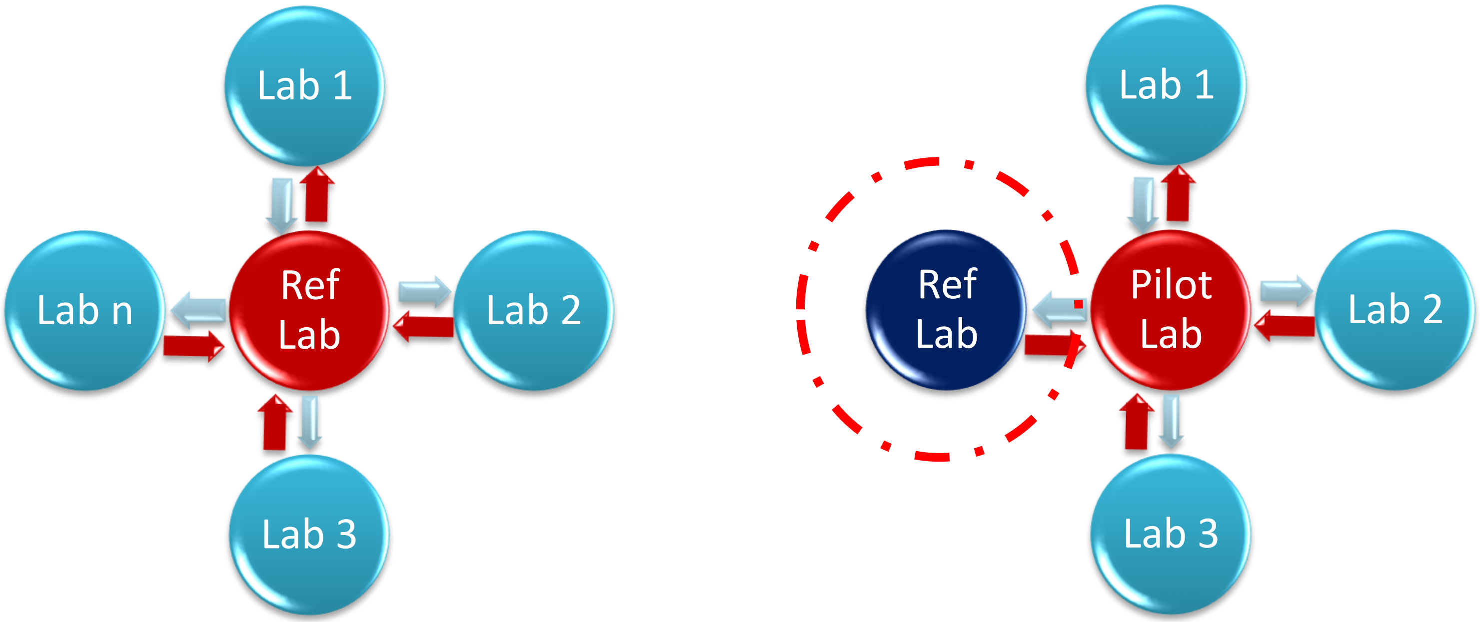
Consideration of appropriate measurement model describing artifact's stability

Measurements after PT completion should validate initial estimates and established limits. If indications of a changing artifact are present a non-conformance investigation might be needed

Key Challenges in a Calibration PT- Stability

Short Term Stability – Petal or Modified Petal Design

- Most conservative design but allows stability to be determined with greatest confidence and smallest uncertainty.



Key Challenges in a Calibration PT- Stability

Short Term Stability – Petal or Modified Petal Design

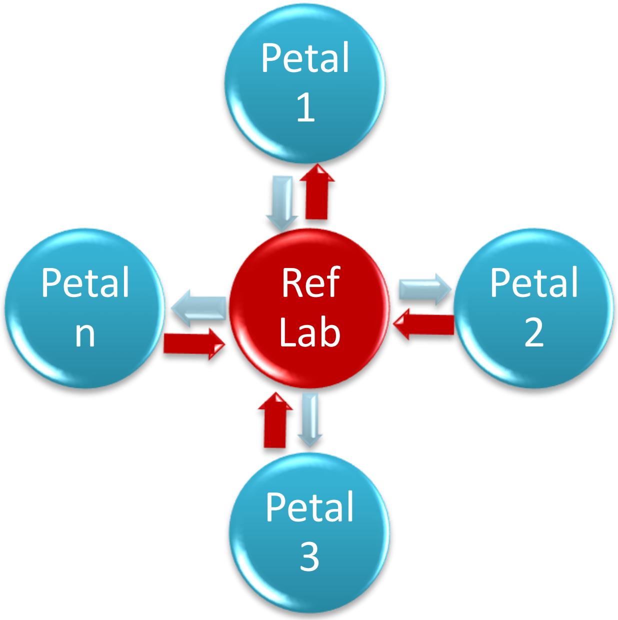
- **Most applicable:** when participating laboratories exhibit small and comparable measurement uncertainties or when there are concerns about the artifact (transfer standard) stability.

+	−
isolation of measurement data of each participant	high operational costs and duration of scheme (partially compensated if a modified petal design is used).

Key Challenges in a Calibration PT- Stability

Short Term Stability – Petal or Modified Petal Design

- Another design for cases where there are many participants: **multiple petal design**
- Disadvantage: linking of different petals to provide common assigned value (if required) and increase in its uncertainty due to variability between different petal assigned values.



In addition to initial and final measurements, interim measurements on artifacts can be performed by reference laboratory during circulation within each petal

Key Challenges in a Calibration PT- Stability

Short Term Stability – Petal or Modified Petal Design

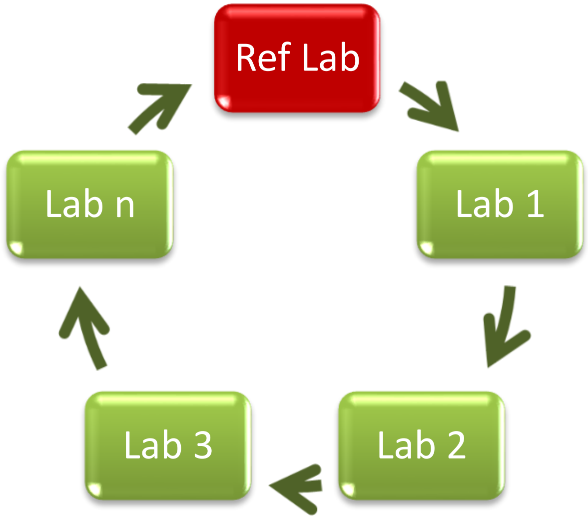
Some artifacts are **inherently stable by design**, construction or materials (dimensional standards, gauge blocks, ring gauges, mass standards etc.).

In these cases short term stability measurements could be minimized. Even in this case though, changes in the artifact due to use or transport still need to be considered

Key Challenges in a Calibration PT- Stability

Short Term Stability – Ring Design

- **Most applicable:** when artifacts are expected to exhibit good short term stability (this means uncertainty due to stability is insignificant as compared to that of the reference laboratory)



In addition to initial and final measurements, artifacts should be measured by reference laboratory periodically to ensure that the actual stability is in agreement with initial expectations

Key Challenges in a Calibration PT- Stability

Short Term Stability – Ring Design

+	−
labour, costs and duration kept minimum	high risk of losing data if the artifact becomes unstable at some point during circulation

Key Challenges in a Calibration PT- Stability

Stability (long-term) and linear regression

- Certain types of artifacts exhibit a uniform change in their value with time.
- Common examples include: standard resistors and Zener voltage reference standards.
- If a **sufficient** history exists it is possible to use a linear regression model to **predict** the reference value realized by the artifact on any given day.

Caution: interim measurements are necessary to ensure that performance is as expected

Enough data to make a statistically sound inference

Will be valid if the uncertainty of the regression is included in the budget for the PT.

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Key Challenges in a Calibration PT- Stability

Stability (long-term) and linear regression

Model: $R_s(t) = R_s(0) + Bt$

FIT COEFFICIENTS	
$R_s(0)$ =	1,0000E+02
B=	5,680E-07

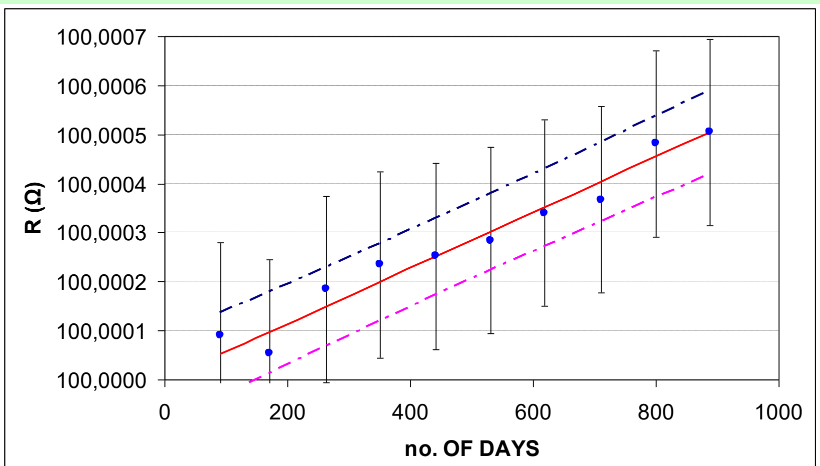
fit

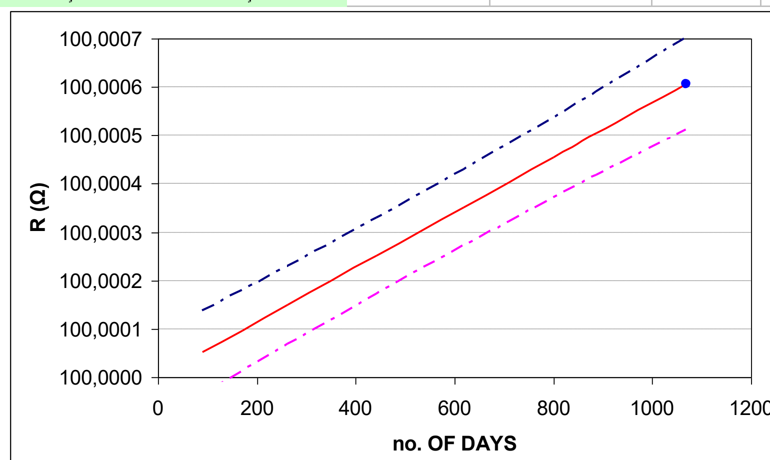
CURVE FIT STATISTICS	
SumSquared Residuals	8,322E-09
Rave	1,000E+02
SumSquared Average	2,013E-07
Coeff of Determination	0,95867

u(Rs(0))=	3,98E-08
u(B)=	6,92E-05
Syx=	3,23E-05
Sxx=	655855

no.	time (days)	R (Ω)	<R>	RESIDUALS^2	[R-Rave]^2	UL	LL	U(<ρ>) (Ω)
1	90	100,0001	100,0001	1,54E-09	3,57E-08	1,0000014E+02	9,9999965E+01	3,73E-05
2	170	100,0001	100,0001	1,79E-09	5,07E-08	1,0000018E+02	1,0000001E+02	3,61E-05
3	263	100,0002	100,0001	1,24E-09	9,00E-09	1,0000023E+02	1,0000007E+02	3,50E-05
4	350	100,0002	100,0002	1,29E-09	2,00E-09	1,0000028E+02	1,0000012E+02	3,43E-05
5	442	100,0003	100,0003	8,84E-13	7,53E-10	1,0000033E+02	1,0000017E+02	3,39E-05
6	531	100,0003	100,0003	2,96E-10	2,47E-11	1,0000038E+02	1,0000022E+02	3,39E-05
7	618	100,0003	100,0004	1,18E-10	3,69E-09	1,0000043E+02	1,0000027E+02	3,42E-05
8	710	100,0004	100,0004	1,29E-09	7,73E-09	1,0000048E+02	1,0000032E+02	3,50E-05
9	800	100,0005	100,0005	7,60E-10	4,10E-08	1,0000054E+02	1,0000037E+02	3,61E-05
10	887	100,0005	100,0005	6,32E-13	5,07E-08	1,0000059E+02	1,0000042E+02	3,74E-05
11	1068		100,0006			1,0000070E+02	1,0000051E+02	4,10E-05

$$U = t(\alpha,v)\,S_{yx}\sqrt{1+1/n+\frac{(x-\bar{x})^2}{S_{xx}}}$$





PTB

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Key Challenges in a Calibration PT- Stability

Stability (long-term) and linear regression

Model: $R_s(t) = R_s(0) + Bt$

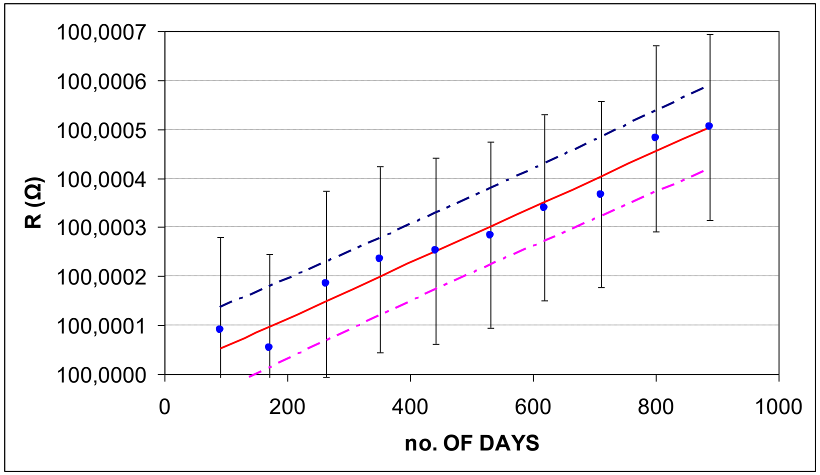
FIT COEFFICIENTS	
$R_s(0)$ =	1,0000E+02
B=	5,680E-07

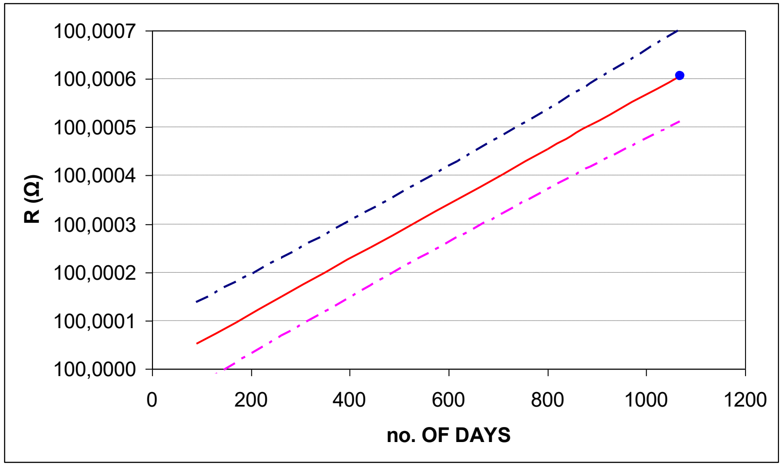
CURVE FIT STATISTICS	
SumSquared Residuals	8,322E-09
Rave	1,000E+02

no.	time (days)	R (Ω)	<R>	RESIDUALS^2	[R-Rave]^2	UL		
1	90	100,0001	100,0001	1,54E-09	3,57E-08	1,0000014E		
2	170	100,0001	100,0001	1,79E-09	5,07E-08	1,0000018E		
3	263	100,0002	100,0001	1,24E-09	9,00E-09	1,0000023E		
4	350	100,0002	100,0002	1,29E-09	2,00E-09	1,0000028E+02	1,0000012E+02	3,43E-05
5	442	100,0003	100,0003	8,84E-13	7,53E-10	1,0000033E+02	1,0000017E+02	3,39E-05
6	531	100,0003	100,0003	2,96E-10	2,47E-11	1,0000038E+02	1,0000022E+02	3,39E-05
7	618	100,0003	100,0004	1,18E-10	3,69E-09	1,0000043E+02	1,0000027E+02	3,42E-05
8	710	100,0004	100,0004	1,29E-09	7,73E-09	1,0000048E+02	1,0000032E+02	3,50E-05
9	800	100,0005	100,0005	7,60E-10	4,10E-08	1,0000054E+02	1,0000037E+02	3,61E-05
10	887	100,0005	100,0005	6,32E-13	5,07E-08	1,0000059E+02	1,0000042E+02	3,74E-05
11	1068		100,0006			1,0000070E+02	1,0000051E+02	4,10E-05

The uncertainty of the regression must be included in the overall uncertainty budget

$$U = t(\alpha,\nu) S_{yx} \sqrt{1 + 1/n + \frac{(x-\bar{x})^2}{S_{xx}}}$$





Key Challenges in a Calibration PT- Reference Value

- ISO/IEC 17043, 4.4.5.2 : Proficiency testing schemes in the **area of calibration** shall have assigned values with **metrological traceability**, including **measurement uncertainty.**
- Methods available for calibration PTs:
  - ✓ reference values;
  - ✓ consensus values from expert participants;
  - ✓ consensus values from participants.

Key Challenges in a Calibration PT- Reference Value

- Procedures available for calibration PTs:
  - ✓ reference values;
  - ✓ consensus values from expert participants;
  - ✓ consensus values from participants.

Key Challenges in a Calibration PT- Reference Value

- Procedures available fo
  - ✓ reference values;
  - ✓ consensus values fr
  - ✓ consensus values fro

- The reference value must be reliable (a reference laboratory with the necessary competence)
- It must have a low uncertainty (much smaller than any of the participants) so as not to affect the participant evaluation

- When a reference laboratory capable of providing a reliable reference value cannot be found.
- When the uncertainty required is smaller than any of the participating laboratories (including reference)
- When PT item's technical characteristics, e.g., resolution, can affect the performance evaluation

Key Challenges in a Calibration PT- Reference Value

Consensus values from participants: Many options:

- ✓ Simple mean
- ✓ Weighted mean
- ✓ Least squares

Robust methods:

- ✓ Median and Median of the Absolute Deviations
- ✓ The cumulative probability algorithm
- ✓ “Value voted most likely to be correct” algorithm

Evaluation of Results

- In most of the cases, E_n is used for evaluation of results in calibration PTs.

$$E_{ni} = \frac{x_i - x_{ref}}{\sqrt{U_i^2 \pm U_{ref}^2}}$$

- with U_i the expanded uncertainty of i^{th} participant result and U_{ref}, of the reference laboratory's assigned value.

WHY?

Evaluation of Results

- If significant inhomogeneity or instability is detected by the reference laboratory then the corresponding uncertainty components have to be considered in order to obtain a realistic evaluation of participants results

$$E_{ni} = \frac{x_i - x_{PT}}{\sqrt{U_i^2 \pm U_{PT}^2}}$$

- with U_{PT} given by:

$$U_{PT} = \sqrt{U_{ref}^2 + U_{stability}^2 + U_{homogen}^2}$$

Conclusions

An NMI:

a) Together with the national accreditation body it organizes national intercomparisons measurements for calibration laboratories in the country and provides a sound metrological basis to the national accreditation scheme.

b) It provides traceability to the national system and through it to the international system.

c) It may offer technical support for the evaluation of PTPs.

d) It may provide reference values, in case that it has published relevant CMCs in the BIPM-KCDB.

e) Is an important source of expertise to the PTPs for the characterization of the transfer standards and statistical analysis of results.

Thank you for your attention!