Verification and calibration of measuring equipment for use in a testing laboratory

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Presentation outline

• Introduction
• Definitions
• The “requirement”
• Metrological traceability
• Calibration
• Calibration versus appropriate accuracy
• Examples of inappropriate calibration
• Direct versus indirect traceability
• Calibration intervals
• Concluding remarks
Introduction

• ISO/IEC 17025 accredited Testing Laboratories are required to have their measuring equipment calibrated.
• Why – to ensure required accuracy can be achieved!
• Assessors are now raising non-conformances for inappropriate calibrations and inadequate metrological traceability
• This presentation is intended to inform and empower Testing Laboratories regarding this requirement.
• The objective is to ensure that the requirement for imported metrological traceability can be met adequately.
Definitions

• “Accuracy” – closeness to the true value (SI value)
• “Uncertainty of measurement” – range of values within which the true value is likely to be with a specified level of confidence

Note:
• One can have a very accurate measurement but with a large uncertainty
• One can have a very inaccurate measurement with a very small uncertainty

“Verification” – demonstration by objective evidence that an instrument is fit for its intended measurement purpose
The requirement....

• ISO/IEC 17025 para 6.5.1 states that, “The laboratory shall establish and maintain metrological traceability of its measurement results by means of a documented unbroken chain of calibrations, each contributing to the measurement uncertainty, linking them to an appropriate reference”

• This remains a technical requirement whether a lab is accredited or not, to ensure valid measurement results
The requirement…. (2)

• It all starts with the required test specification limits…..
  • The pH of the water was between 7.2 and 8.4…
  • The residual chlorine in the water was > 20 ppm…
  • The sodium content in the viennas was < 100 mg/100 g…
  • The cadmium content in the pineapples was < 1 mg/kg…
  • The fat content in the sample was < 3 g/100 g
  • The tensile strength of the mild steel bar was between 700 MPa and 900 MPa
  • The hardness of the material was between 48 and 52 HRC
  • The resistance of the wire was between 0.5 Ω and 1 Ω at 20°C
  • The number of colony forming units (CFUs) in effluent after incubation at 37°C for 24 hours was ≤ 100.
The requirement....(3)

• These will then dictate certain influence parameter tolerance limits......
  • To test for pH the temperature of the solution must be between 18°C and 23°C...
  • The volume of the sample for residual chlorine test must be 250 mL ± 5 mL...
  • The mass of the pineapple sample must be 0,5 g ± 10 µg...
  • The temperature of the incubator must be maintained at 37°C ± 0,3°C...
  • The diameter of the tensile test sample must be 25 mm ± 0,25 mm...
  • Etc.

• This implies that we need to be able to perform these measurements to better accuracies than the test tolerance limits
Metrological Traceability

SI

NMIs

Cal Labs

Testing Labs

Industry
Importing metrological traceability

• SANAS policy allows for the following options (PM01 and TR25):
  • Calibration by SANAS accredited calibration laboratories;
  • Calibration by laboratories accredited by full members of the ILAC arrangement;
  • Calibration by the National Metrology Institute of South Africa
  • Calibration by members of the International Committee of Weights and Measures (CIPM) arrangement, whose Calibration and Measurement Capabilities (CMC’s) are accepted into the International Bureau of Weights and Measures (BIPM) Key Comparison Database (KCDB).
Establishing metrological traceability in-house

• SANAS policy allows for Self-calibration in-house provided that (TR25):
  • Calibrated Reference Standards are available;
  • A documented and validated calibration procedure or method is followed;
  • A formal means exists to record the results of the calibration – normally a calibration certificate;
  • Evidence of the demonstrated competence of the personnel performing the calibration;
Establishing metrological traceability in-house (2)

• SANAS policy allows for Self-calibration in-house provided that (TR25) cont.:
  • A calibration schedule is available indicating technically valid calibration intervals for Reference Standards;
  • A procedure is available and applied for the estimation of measurement uncertainty;
  • Records are available to demonstrate the ongoing assurance that the results produced by the laboratory remain technically valid;
  • Documented requirements for environmental conditions, and records are available where applicable; and
  • Evidence of internal audits of all activities that could influence the quality of calibration results is available.
Calibration

• Is required to ensure the measuring instrument can perform the **required** measurement with a defined (appropriate) accuracy
• “Accuracy” is defined as the “**closeness to the true value**”
• What is the “true value”?
• The value of a higher order Reference Standard all the way to the SI.
Calibration (2)

• Two main approaches
  • Calibration at discrete cardinal points each with an error (correction) and associated measurement uncertainty (Direct Traceability)
  • Conformance testing against appropriate specification limits (usually the Manufacturer’s Accuracy Specifications)
    Refer to ISO/IEC 17025:2017 Annex A Para A.2.3 (Indirect Traceability)
## Calibration - Direct Traceability

<table>
<thead>
<tr>
<th>Nominal</th>
<th>Applied</th>
<th>UUT Indicated</th>
<th>Correction</th>
<th>Uncertainty of Measurement</th>
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<td>1,003</td>
<td>1,002</td>
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## Calibration - Direct Traceability (2)

<table>
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<th>Correction</th>
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Calibration – Indirect Traceability

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</table>

- This instrument can now be used anywhere in the range from 1 to 1000 with an accuracy of ± 0,5%
Advantages of direct traceability

• Highest accuracy
• Smallest uncertainty
• May be lower cost (can be fewer calibration points)
• Instruments can be used at accuracies better than their manufacturers’ specified accuracies
Disadvantages of direct traceability

• Users cannot simply note the displayed reading on their “calibrated” instrument
• Corrections must be applied before use
• Instrument can usually only be reliably used at the calibration points without significant additional mathematics
• Instruments can usually only be used for one specific application
• Uncertainty budgets often have to be updated after each re-calibration to consider the latest values.
Advantages of indirect traceability

- Instruments can be used reliably at any point within their operating range
- The user can note the displayed reading without having to apply any corrections
- Uncertainty budgets do not have to change after every re-calibration
Disadvantages of indirect traceability

• Slight lower accuracy
• Larger measurement uncertainty
• Requires detailed contract review with calibration service providers as this is not the “default” calibration option (more work for the cal labs)
• May be more expensive (increased cost must be weighed against cost saving due to less effort required by user)
Calibration versus fit-for-purpose accuracy

- Calibration should guarantee traceability to SI
- BUT does this automatically ensure the measuring instrument is fit for purpose?
- NO!!
- “Calibration” does NOT necessarily ensure an instrument can perform the intended measurement with the required accuracy
- Importance of detailed contract review with the calibration service provider
Mass example of unfit calibration

- Test Tolerance is 100 g ± 1 mg (99.999 g to 100.001 g)
- Balance calibrated at 100 g nominal mass
- Reported error is - 0.01 g
- Reported UoM is ± 5 mg
Mass example of unfit calibration (2)

100,001 g

100 g

99,999 g

99,995 g

99,990 g

99,985 g

100,005 g

99,995 g

Corr + 0,01 g
Temp. example of unfit calibration

• Incubator must be controlled at 37°C ± 0,3°C (36,7°C to 37,3°C)
• Thermometer calibrated at 20°C nominal temperature
• Reported error is + 0,1°C
• Reported UoM is ± 0,05°C
Temp example of unfit calibration (2)

37,3°C

37°C

36,7°C

20,15°C
20,1°C
20,05°C

Corr -0,1°C

20,05°C
20,0°C
19,95°C
Calibration Intervals

- Calibration is strictly speaking only valid at the time it is performed
- Statement on most calibration certificates,

“Measurement results recorded in this certificate were correct at the time of calibration. Subsequent accuracy will depend on factors such as care, handling and frequency of use. It is recommended that recalibration be undertaken at an interval that will ensure that the instrument remains within the desired limits.”
Calibration Intervals (2)

• How do we maintain confidence in the calibration of an instrument after it leaves the cal lab?
• We perform verifications/intermediate checks!
• The verification/intermediate check points must be carefully selected to be technically appropriate
  • Verification at range limits
  • Verification of selected range
  • Value of “signature” verifications
  • Etc.
Verification/Intermediate checks

![Graph with verification points and intermediate checks](image-url)
Concluding remarks

• Instruments must be calibrated to maintain an appropriate accuracy
• “Calibration” does not automatically ensure an instrument is fit for intended purpose
• The “accuracy” of an instrument must be known at the point of use – this does not necessarily need to be a calibration point
• A measurement point should under normal circumstances be bounded by calibration points
Concluding remarks (2)

- Re-calibration intervals should be chosen to ensure confidence is maintained in appropriate accuracy
- Re-calibration intervals cannot be increased indefinitely – must be based on “risk”
- Increased confidence can be secured through verifications/intermediate checks
Concluding remarks (3)

• In temperature measurement
  • A thermometer measures its own temperature…..
  • Is the thermometer at the temperature of what it is you are trying to measure? (Liquid, incubator etc.)
  • Larger errors due to method of temperature measurement than due to calibration…. (inhomogeneity of iso-thermal media)

• This topic cannot be given justice in one 25 minute slot. NLA-SA plans to run a special one or two day course/workshop to cover more detail.
Thank You
We measure what matters