



**National Accreditation Board for Testing  
and Calibration Laboratories (NABL)**



## **Specific Criteria for Accreditation of Calibration Laboratories**

**(Mechanical, Fluid Flow, Radiological, Electro-Technical & Thermal  
Calibration)**

### Amendment Sheet

Sl.	Page No.	Clause No.	Date of Amendment	Amendment	Reasons	Signature QM	Signature CEO
1							
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8							
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<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129		Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)		
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 1 of 133

## Contents

Sl.	Title	Page No.
	Amendment sheet	1
	Contents	2
	Introduction	3
<b>1.</b>	<b>Mechanical Calibration</b>	<b>4</b>
	Chapter 1(A): Dimensional Metrology	5
	Chapter 1(B): Calibration of Mass (Weights)	28
	Chapter 1(C): Calibration of Mass (Weighing Scale & Balance)	35
	Chapter 1(D): Volume	41
	Chapter 1(E): Density and Viscosity Measurement	47
	Chapter 1(F): Calibration of Force Proving Instruments	58
	Chapter 1(G): Calibration of UTM, Tension Creep and Torsion Testing Machine	67
	Chapter 1(H): Calibration of Mobile Force Measuring System (Push Pull Gauge)	70
	Chapter 1(I): Calibration of Verification of Hardness Testing Machine	71
	Chapter 1(J): Calibration of Torque Measuring Devices	76
	Chapter 1(K): Calibration of Torque Generating Devices	85
	Chapter 1(L): Calibration of Pressure Balance/Dead Weight Tester	89
	Chapter 1(M): Calibration of Pressure Indicating Devices	97
	Chapter 1(N): Verification of Impact Testing Machine	103
	Chapter 1(O): Calibration of Duro Meter (Rubber Hardness Testers)	107
<b>2.</b>	<b>Fluid Flow Calibration</b>	<b>111</b>
<b>3.</b>	<b>Radiological Calibration</b>	<b>117</b>
<b>4.</b>	<b>Electro-Technical Calibration</b>	<b>121</b>
<b>5.</b>	<b>Thermal Calibration</b>	<b>127</b>

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 2 of 133

## Introduction

The purpose of this document is to facilitate the calibration laboratories to carry out calibration activities in accordance with ISO/IEC 17025 adhering to the following NABL Policy documents -

1. NABL 165- NABL's Policies for Accreditation (as per ISO/IEC 17025: 2017)
2. NABL 133 –NABL Policy for use of NABL symbol/ claim of accreditation by accredited conformity Assessment Bodies & NABL Accredited CAB combined ILAC MRA Mark
3. NABL 142- Policy on Traceability of Measurement Results
4. NABL 143- Policy on Calibration and Measurement Capability (CMC) & Uncertainty in Calibration
5. NABL 163- Policy for participation in Proficiency Testing Activities
6. NABL 164-Guideline for Inter Laboratory Comparison in Calibration Laboratories where Formal PT programs are not available.
7. NABL 130- Specific Criteria for Site Testing & Site Calibration

In addition, calibration laboratories shall also follow the other relevant NABL documents.

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Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 3 of 133

# 1. Mechanical Calibration

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 4 of 133

## Chapter 1(A): Dimensional Metrology

### 1. Recommended General Requirements

- 1.1 **Acoustic Noise:** Noise level shall be maintained less than 60 dBA, wherever it adversely affects the required accuracy of measurement.
- 1.2 **Illumination:** The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.
- 1.6 The ambient temperature shall be  $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$  with a maximum variation of  $2^{\circ}\text{C}$  per day and  $1^{\circ}\text{C}$  per hour.
- 1.7 In case of calibration of reference masters demanding finer uncertainties such as calibration of Gauge Blocks by comparison with standards or with interferometer, the ambient temperature shall be  $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$  with a maximum variation of  $0.5^{\circ}\text{C}$  during measurement.
- 1.8 Recommended Environmental monitoring equipment:
  - Temperature with a resolution of  $0.1^{\circ}\text{C}$
  - Humidity with a resolution of 1% RH
 However, laboratory shall evaluate the requirement of accuracy, resolution and Uncertainty depending on the CMC aimed at.

### 2. Recommended Requirements – Calibration - Linear Measurements

	Sl. No.	Equipment	Permanent Facility	On Site	Mobile
2.1 Linear - Measuring Instruments	1	Vernier & Digital Calipers	✓	✓ (Above 1m length)	✓
	2	External Micrometers, Depth Micrometers, Micrometer Head, Micrometer Setting Pieces	✓	X	✓
	3	Bench Micrometer & Micrometer Head	✓	X	✓
	4	Bore Gauges	✓	X	✓
	5	Stick Micrometers	✓	X	✓
	6	Tripoint Internal Micrometers	✓	X	✓
	7	Depth Gauges	✓	X	✓
	8	Dial Gauge	✓	X	✓
	9	Steel Scales & Line Standards,	✓	X	✓
	10	Measuring Tape, Pie-Tape	✓	X	✓
	11	Extensometer	✓	✓	X

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 5 of 133

2.2 Linear – Reference (Measuring) Equipments	SI.No.	Equipment	Permanent Facility	On Site	Mobile
	1	Gauge Block Comparators	✓ <sup>\$</sup>	✓	X
	2	Metroscope/ Universal Length Measuring Machine (ULM)	✓ <sup>\$</sup>	✓	X
	3	Length Measuring Machine	✓ <sup>\$</sup>	✓	X
	4	Universal Measuring Microscope (UMM)	✓	✓	X
	5	Profile Projector	✓ <sup>\$</sup>	✓	X
	6	Bore Comparator	✓	✓	X
	7	Dial Gauge Calibrator	✓	✓	✓
	8	Floating carriage Micrometer	✓	✓	X
	9	Coordinate Measuring Machines	✓ <sup>\$</sup>	✓	X
	10	Laser Interferometer	✓	X	X
	11	Flatness Interferometer	✓	X	X

⌘ - Permanent facility for their internal use only and not meant for external customer.

2.3 Linear - Measuring Aids	SI.No.	Equipment	Permanent Facility	On Site	Mobile
	1	Straight Edges	✓	✓	✓*
	2	Engineers Parallels	✓	✓	✓*
	3	Surface Plate (Cast Iron)	✓	✓	✓*
	4	Surface Plate (Granite)	✓	✓	✓*
	5	Angle Plates, Right Angle & Box Angle Plates	✓	✓	✓*
	6	Engineers Square	✓	✓	✓*
	7	Granite Squares	✓	✓	✓*
	8	Cylindrical Square	✓	✓	✓*
	9	V-Block, Welded V- Blocks, Universal And Elongated V-Blocks,	✓	✓	✓*
	10	Test Mandrels (Straight)	✓	X	X
	11	Test Mandrels (Taper)	✓	X	X
	12	Bench Centre	✓	✓	✓*
13	Vernier Height Gauge	✓	✓	✓	

\*Size of DUC is subject to facility available at Mobile Facility

2.4 Linear & Form – Reference Equipment	SI.No.	Equipment	Permanent Facility	On Site	Mobile
	1	Gauge Blocks	✓	X	X <sup>1</sup>
	2	Gauge Block Accessories	✓	X	X
	3	Long Gauge Blocks, Length Bar	✓	X	X
	4	Step Gauges	✓	X	X
	5	Caliper Checker, Check Master	✓	X	X
	6	Height Setting Micrometers	✓	✓	✓
	7	Electronic Height Gauges	✓	✓	✓
	8	Height Master	✓	✓	✓
9	Raiser Blocks	✓	✓	✓	

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Doc. No: NABL 129

Specific Criteria for Accreditation of Calibration Laboratories  
(Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)

Issue No: 01

Issue Date: 02-Apr-2019

Amend No: --

Amend Date: --

Page 6 of 133

	10	Cylindrical Setting Standards	✓	X	X
	11	Roundness Master S (Hemi-Sphere, Magnification Standard, Master Cylinder, Optical Flat)	✓	X	X
	12	Surface Roughness Masters (Surface Masters, Depth Standard)	✓	X	X
	13	Radius Standard/ Contour Master	✓	X	X
	14	Glass Scale, Graticule And Glass Grid	✓	X	X

X1: Gauge Block calibration needs stringent environmental control, leveled surface and vibration free environment. The Gauge Block calibrator should not be subjected to shocks during transit. Hence, recommending for mobile calibration facility may be difficult.

2.5 Angle Measuring Devices	Sl.No.	Equipment	Permanent Facility	On Site	Mobile
	1	Angle Gauges	✓	X	X
	2	Bevel Protractors	✓	X	X
	3	Combination Set	✓	X	X
	4	Sine Centre, Sine Bar & Sine Table	✓	X	X
	5	Spirit Levels, Frame Level	✓	X	X
	6	Electronic Level	✓	X	X
	7	Inclinometer	✓	X	X
	8	Clinometers	✓	X	X
	9	Auto Collimator,	✓	X	X
	10	Polygon Mirrors, Prisms	✓	X	X
	11	Rotary Table, Index Table	✓	X	X
	12	Rotary Calibrator	✓	X	X

2.6 Surface Topography Measuring Devices	Sl.No.	Equipment	Permanent Facility	On Site	Mobile
	1	Surface Roughness Masters, Depth Masters	✓	X	X
	2	Radius Standards, Master Sphere For CMM	✓	X	X
	3	Optical Flats, Optical Parallels	✓	X	X
	4	Hemi-Sphere (Roundness Master)	✓	X	X
	5	Magnification Master (Flick Standard)	✓	X	X
	6	Master Cylinder, Cylindrical Square	✓	X	X
	7	Roughness Tester-Portable	✓	✓	X
	8	Roughness Tester-Stand Alone	✓	✓	X
	9	Contour Profiler	✓	✓	X
	10	Roundness Tester	✓	✓	X
11	Form Tester	✓	✓	X	

2.7 Gauges	Sl.No.	Equipment	Permanent Facility	On Site	Mobile
	1	Setting Master Plug Gauges	✓	X	X
	2	Plug Gauges	✓	X	X
	3	Setting Master Ring Gauges	✓	X	X
	4	Ring Gauges	✓	X	X

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Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 7 of 133



	5	Snap Gauges	✓	X	X
	6	Taper Gauges	✓	X	X
	7	Thread Plug Gauges	✓	X	X
	8	Thread Ring Gauges	✓	X	X
	9	Screw Pitch Gauges	✓	X	X
	10	Form Gauges	✓	X	X
	11	Radius Gauges	✓	X	X
	12	Feeler Gauges	✓	X	X
	13	Plated Wire Gauges	✓	X	X
	14	Indicating Gauges	✓	X	X
	15	Air Gauges	✓	X	X

2.8 Miscellaneous	Sl.No.	Equipment	Permanent Facility	On Site	Mobile
	1	Test Sieves	✓	X	X
	2	Gears, Master Gears	✓	X	X
	3	Gear Testing Equipments	✓	✓	X
	4	Involute & Lead Masters	✓	X	X
	5	CNC Machine Tools	✓\$	✓	X

\$ - Permanent facility for their internal use only and not meant for external customer

2.9 Industrial Gauge and Template	Sl.No.	Equipment	Permanent Facility	On Site	Mobile
	1	Welding Fillet Gauge, Templates, Vickers/Knoop/Rockwell Diamond Cone Indenter/ Weld/ Hi-Lo Gauge, Bridge Cam Gauge /Traverse Of Cupping Machine /Limit Gauges/CD Gauge/PCD Gauge / Cube Mould/Welding Gauge/Width Gauge/ Paddle Gauge/Weld Fillet Gauge/ Flakiness Gauge/ Elongation Gauge/ Receiver Gauge/ Plain Work Piece/ Lever Arm/ Master Connecting Rod/ Inspection JIG And Fixture/Moulds	✓	X	X

### 3. Metrological Requirements

Laboratories performing calibration in Dimensional metrology needs to ensure the followings:

- In dimension, scope to be recommended only for the better resolution for the same equipment (master) and range. Coarser resolution for same equipment and range need not be to be specified in the scope. However, uncertainty to be reported taking in to account of Resolution of the UUC , Type of the scale (analogue, digital, vernier) and the method adopted for the scale reading.
- Height Gauge (Analog and Electronic) and Vernier caliper & micrometer (above 1m length) can be calibrated both at lab and site, in case of site calibration; temperature condition is to be maintained as 20±2°C.
- Calibration of steel scale can be done by using profile projector. However length of steel scale being calibrated depends on the available plate size of the profile projector. Comparison with Vernier caliper is not recommended.

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Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 8 of 133

- Calibration of measuring tape can be done by using tape measuring machine or any other equipment with magnifying facility and tension adjustment unit. Range for tape to be specified in the scope instead of any length.
- Calibration of surface plate can be allowed with master equipment having Sensitivity 10  $\mu\text{m}/\text{m}$  or better and the CMC to be demonstrated for a particular grade and size of surface plate. Size to be specified for surface plate instead of any length.
- Calibration of master / reference ring gauges, setting master using bore gauge is not acceptable. However, for limit gauges (for class 8 of IT grade and above) bore gauges can also be used subject to satisfactory TUR.
- Calibration of surface roughness specimen (with specified value) using surface roughness tester and vice versa shall not be allowed.
- Calibration of Viscosity Flow cup cannot be considered under Dimensional metrology.

#### 4. Recommended Reference Masters and Standards for Dimension

##### Linear Measuring Devices

##### 4.1 Measuring Instruments

Sl. No.	Equipment/ DUC	Relevant Standards/ Guidelines	Recommended Parameters to measure	Recommended Master equipment used for calibration
1	Calipers Vernier Up to 1000mm	IS: 3651 Part1	Error-External Jaws Error-Internal Jaws Error-Depth Parallelism of Ext Jaws Parallelism of Int. Jaws Repeatability of dial if present	Gauge Blocks / Length bars/ LMM/ Caliper Checker Gauge Blocks Micrometer, Slip gauge accessories
	Calipers Vernier Up to 500mm	IS: 3651 Part2	Error-External Jaws Error-Internal Jaws Error-Depth Parallelism of Ext Jaws Parallelism of Int. Jaws Repeatability of dial if present	Gauge Blocks / Length bars/ LMM/ Caliper Checker Gauge Blocks Micrometer, Slip gauge accessories
	Calipers Vernier Above 1000mm Up to 4000mm	IS: 3651 Part3	Error-External Jaws Error-Internal Jaws Parallelism of Ext Jaws Parallelism of Int. Jaws Repeatability of dial if present	Gauge Blocks / Length bars/ LMM Gauge Blocks/ Length bars/ LMM Micrometer, Slip gauge accessories
2	Dial indicators-plunger	IS: 2092	Hysteresis Repeatability Accuracy (total error band) over any 1/10 rev 1 rev, 2 rev. Up to 10 rev	Dial Calibration Tester/ Micrometer Head/ Gauge Blocks / ULM
3	Dial indicators-Lever	IS: 11498	Hysteresis Repeatability Accuracy (total error band) over any 0.1mm	Dial Calibration Tester/ Micrometer Head, surface plate & Gauge Blocks / ULM

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 9 of 133

			half rev Total	
4	Internal micrometers Stick Micrometers	IS:2966	Micrometer screw error Error in length of each extension rod When thimble reads zero	ULM/ Inside micrometer checker/ V block, Dial gauge, Gauge Block, Vertical Comparator
5	External Micrometers-	IS: 2967	Micrometer Screw Error (Error of Measurement) Flatness of anvils Parallelism of anvils	Gauge Block set, Micrometer Checker Optical Flat Set of 4 Optical Parallels
6	Depth Micrometer	BS:6468	Micrometer screw error Flatness of Reference face Error in length of each extension rod when thimble reads zero	Depth gauge Master/ Gauge Blocks V-Block & Surface Plate
7	Micrometer Head,	IS: 9483	Deviation of Traverse over 25mm Flatness of Anvil	ULM/ Gauge Blocks & Electronic Comparator / Optical Flat
8	Micrometer setting Rods A. Flat ended	-	Parallelism Gauge Length	Gauge Blocks Surface Plate & Electronic Comparator
	Micrometer setting Rods B. Round ended	-	Gauge Length	Gauge Blocks, Surface Plate & Electronic Comparator/LMM
9	Metric Steel Scales Upto 2000m/ Shrinkage Scale	IS: 1481	Deviation from nominal length between – end face to first graduation line – any two adjacent mm scale marks (Excluding 1st mm scale mark) – any two adjacent centimeter scale marks (Excluding 1st cm scale mark) – any two adjacent decimeter scale marks (Excluding 1st dm scale mark) – end face to 1000mm graduation line – end face to 2000mm graduation line	Scale Measuring Equipment with Optical Viewing/ sensing with magnifying facility  Profile Projector (Upto Plate length) / UMM  <i>Note: comparison with vernier caliper is not recommended</i>
10	Metric Steel Tapes 1,2,10,15,20,30 & 50m	IS: 1269	Deviation from nominal length between – outer edge of ring/ latch to first graduation line in push condition – inner edge of ring/ latch to first graduation line in pull condition – end face to first graduation line – any two adjacent	Tape Measuring Equipment with Optical Viewing/ sensing with magnifying & tension adjustment facility.

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Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 10 of 133

			<ul style="list-style-type: none"> <li>millimeter graduation lines</li> <li>– any two adjacent centimeter graduation lines</li> <li>– any two adjacent decimeter graduation lines</li> <li>– any two adjacent meter graduation lines</li> <li>– end face to any metre graduation line</li> </ul>	
11	Woven metallic & glass fiber tape Measuring Tape (steel)	IS:1269 Part I	<ul style="list-style-type: none"> <li>-Deviation from nominal at suitable intervals</li> <li>-Chord length at catenary correction</li> </ul>	Profile Projector Or Length Measuring Machine Or Tape Measuring Equipment with Optical Viewing/sensing With tension attachment
12	Pie-Tape	----	<ul style="list-style-type: none"> <li>-Calibration of Vernier Scale (Diameter Scale)</li> <li>-Calibration of Main Scale (Diameter Scale)</li> <li>-Calibration of Vernier Scale (Circumferential Scale)</li> <li>-Calibration of Main Scale (Circumferential Scale)</li> </ul>	Profile Projector Or Length Measuring Machine Or Tape Measuring Equipment with Optical Viewing/sensing
13	Extensometer, Class 0.5, 1, & 2	IS:12872	<ul style="list-style-type: none"> <li>-Relative Bias Error before Gain Correction</li> <li>-Relative Bias Error after Gain Correction for increase in length</li> <li>-Gain Correction for decrease in length</li> <li>-Relative gauge length error</li> <li>Drift of DRO</li> </ul>	Digital Height Gauge Profile Projector
14	Bore Gauges (2-point)	JIS B 7515	Transmission	ULM / Dial Calibration Tester
15	Bore Comparator/ 3 Anvil Micrometer, 3 Anvil Intramike	----	Transmission/ Micrometer Error	Set of Ring Gauges (min 3 rings to cover the range)
16	Vernier Height Gauge Upto 1000mm  Electronic Height Gauge	IS: 2921	<ul style="list-style-type: none"> <li>Measuring Error at 5 to 8 places along working length</li> <li>Parallelism of scribe to base</li> <li>Squareness of movement to the base (only for 2D Electronic Height Gauge)</li> </ul>	Gauge Blocks / Step Gauge / Elec. comparator/ Caliper Checker Elec. Comparator & Surface Plate Squareness tester/ Granite/ Cylindrical Square
17	Vernier Depth Gauge Upto 300 mm	IS:4213	<ul style="list-style-type: none"> <li>Error at 5 to 8 places along working length</li> <li>Flatness of reference face</li> <li>Parallelism between the</li> </ul>	Gauge Blocks  V-Block & Dial Indicator

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129		Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)		
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 11 of 133

			beam measuring face and reference face	
18	Dial Thickness Gauge	-	Length	Gauge Block (0 Grade)

Laboratory shall follow the requirements of NABL 141 for the selection of reference master and equipments for "Test Uncertainty Ratio (TUR)".

#### 4.2 Measuring Equipment

Sl. No.	Equipment/ DUC	Relevant Standards/ Guidelines	Parameters to be measured	Master equipment used for calibration
1	a. Profile Projector  b. Video Measuring Machine	Relevant standards & guidelines, JIS B 7184	Mandatory Parameters: Magnification Position (Linear) Angle  Linear and Angular	<i>Linear</i> - Glass scale/Gauge Block, Laser Interferometer.  <i>Magnification</i> : Gauge Block and Digital Caliper  <i>Angle</i> : Angular Graticule (Protractor)/ Angle Gauges
2	Microscope	ISO 10936-1	Axis movement Magnification	Glass Scale/Gauge Block
3	Co-Ordinate Measuring Machines(CMM)	IS15635/ ISO10360	Probing Error (MPEP) Scanning Probing Error (Tij) Length Measuring Error (MPEE)	Test Sphere Step gauge, Gauge Blocks/ Ball bar
4	Gauge Block Comparators	Euramet Cg-2	Error in Length Measurement -Differences of central length $l_c$ -Difference of central length of pair -Deviations $f_o$ and $f_u$ from the central length. Sensitivity / Hysteresis	Reference Gauge Blocks
5	Laser Interferometer	Relevant standards & guidelines	-Wave length -Air Temperature - Pressure - Humidity OR Length Measuring error of Basic laser, with Environmental Compensation unit and with Environmental Compensation unit and material temperature sensor.	Iodine Stabilized Laser & Equipment suitable for measurement of Air temperature, pressure and humidity OR Laser Interferometer with related sensors and Environmental Compensation unit
6	Dial Gauge Calibrator		Drum Accuracy	Gauge Blocks & Electronic Comparator
7	Length Measuring Machine		Length Measuring error over entire range	Gauge Blocks/ Setting Rods/ Laser Interferometer

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 12 of 133

8	ULM		Length Measuring error over entire range	Gauge Blocks / Laser Interferometer
9	Floating Carriage Micrometer/ Diameter Measuring Machine	MOY/SCMI/9	Overall Accuracy, Micrometer Head Error, Alignment of centres to base, Parallelism of micrometer face to line of centres	Gauge Blocks & Electronic Comparator/ ULM, Surface Plate, Test Mandrel, Electronic Comparator
10	Flatness Interferometer	Manufacturer's Test Protocol	Flatness	Reference (Transmission) Flat

#### 4.3 Measuring Aids

Sl. No.	Equipment/ DUC	Relevant Standards/ Guidelines	Parameters to be measured	Master equipment used for calibration
1	Surface Plates – Cast Iron Surface Plates – Granite	IS: 2285 IS:7327 IS 12937	Flatness deviation of the working surface overall  Flatness deviation of any local area of 250x250 mm of working surface	-Dial indicator with Robust stand and distance piece for small plates -Rigid Bridge with a flat surface having relief in the centre along with Precision level (sensitivity 10 µm/m or better) / Autocollimator -Comparator (Datum Gauge) for local area/ Straight Edge (0 Grade) and Gauge Block (0 Grade)
2	Straight Edges Grade 00, 0, 1,&2	IS: 2220 IS: 12937	Straightness of working faces Parallelism of working face	Surface Plate & Dial Indicator/ Precision Level/ Autocollimator/ Laser Interferometer/ CMM
3	Engineer's Parallels Grade 1 & 2	IS:4241	Thickness and width Variation in Thickness Parallelism Equality of pairs	Surface Plate, Gauge Block & Dial Indicator/ Precision Level/ Autocollimator/ Laser Interferometer/ CMM
4	Engineer's Square/ Tri Square, Upto 1100 mm Grade A, B & C Type 1, 2 & 3	IS:2103 IS 12937	Straightness of blade edge &  Parallelism of blade edge & stock/ base  Squareness of external square Squareness of internal square	Surface Plate & Dial Indicator/ CMM  Surface Plate & Dial Indicator/CMM  Squareness tester/ Granite Square/ Cylindrical square, Gauge Block & Dial Indicator/ CMM
5	Granite Square	Based on IS:2103	Flatness of working faces Squareness of working faces	Surface Plate & Dial Indicator/ CMM Squareness tester/ Granite Square/ Cylindrical square & Dial Indicator/ CMM
6	Cylindrical squares	IS:6952	Squareness of cylindrical	Squareness tester, Surface

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Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 13 of 133

	Up to 750mm length Type A & B		surface to end faces Straightness of cylinder side Flatness of end faces	Plate & Dial Indicator Or Form tester Or CMM
7	Angle Plate, Size: 1 to 10 Grades: 1 & 2  Précision Angle Plate, Size: 1 to 6	IS:2554  IS:6973	Flatness of working faces  Parallelism of opposite faces & edges  Squareness of exterior faces over width (H) Squareness of end faces with respect to exterior faces over total length (L)	Precision level/ Surface gauge set up (Surface Plate, Jacks & Dial Indicator )  Surface Plate & Dial Indicator  Squareness Tester/ Digital Height Gauge / Engineers or cylindrical square with Dial Indicator CMM/ Gauge Block
8	Box Angle Plate, Size: 1 to 6 Grades: 1 & 2	IS:6232	Flatness of working (exterior) faces  Parallelism of opposite working faces Squareness of adjacent faces over dimensions Matched Pair	Precision level/ Surface gauge set up (Surface Plate, Jacks & Dial Indicator )/ Straight Edge & Gauge Blocks Surface Plate & Dial Indicator Squareness Tester/ Digital Height Gauge / Engineers or cylindrical square with Dial Indicator CMM/ Gauge Block
9	Block Squares Types: Solid & Hallow  Grades: 1 & 2 Size: 1 to 11	IS:4563	Flatness of working faces Parallelism between working faces  Squareness between working faces	Precision level/ Surface gauge set up (Surface Plate, Jacks & Dial Indicator )  Surface Plate & Dial Indicator Squareness Tester/ Digital Height Gauge / Engineers or cylindrical square with Dial Indicator CMM
10	Plain V-Block Type A1, A2, B1 & B2  Grade 0, 1 & 2	IS:2949	Parallelism of axis of Vee to base surfaces  Parallelism of axis of Vee to side surfaces  Matching tolerance of V axes above the base (for matched pairs) Height over Minimum & Maximum Cylinders (for matched pairs)	Surface Plate, Test Mandrels & Dial Indicator Or CMM

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 14 of 133

			Symmetry of axis of Vee to side surfaces Squarness	
11	Test Mandrels (Straight) φ40x150mm to φ12x1600mm	IS: 2063/ ISO 230-1	Variation in diameter Total Runout	Indicating Snap gauge/ comparator Bench Centre & Dial Indicator
12	Test Mandrels (Taper) φ12x100mm to φ50x500mm straight portion	IS: 2063/ ISO 230-1	Variation in diameter Total Runout Position of Gauge Plane Half Taper Angle Roundness Straightness of taper Datum step(where applicable)	Indicating Snap gauge/ comparator Bench Centre & Dial Indicator Sine Centre with surface plate, Gauge Blocks and Dial Indicator/ CMM Roundness Tester
13	Reference Discs		Diameter at four positions Roundness	ULM/ CMM Roundness Tester/ Form Tester
14	A. Coating Thickness Gauge B. Foil  C. Ultrasonic Thickness Gauge	Nil	Coating Thickness  Thickness  Thickness /Height	Thickness Foil  Electronic Probe with comparator stand/ Dial Gauge with LC. 0.1μm  Gauge Block and Long Gauge Block
15	Bench Centre	IS 5980	Co-axiality Parallelsim of Axis of Centres	Test Mandrel, Lever Dial Gauge Or CMM
16	Ball Bar & Master	--	Linear displacement. Center Distance	ULM/ CMM

#### 4.4 Reference Masters

Sl. No.	Equipment/ DUC	Relevant Standards/ Guidelines	Parameters to be measured	Master equipment used for calibration
1	Gauge Blocks Upto 100mm length  Grades: K, 00, 0,1 & 2	IS: 2984  ISO 3650	Gauge Length Variation in Length Flatness	-Gauge Block Interferometer for Gr K & 00 -Gauge Block Comparator & Bridge type Gauge Blocks for others.  Reference Gauge Block Grade of Gauge Block Calibrated Gr K Gr K Gr K Gr 0 Gr K Gr 1 Gr K Gr 2
2	Gauge Blocks Above 100mm / Length	IS: 2984/ ISO 3650	Gauge Length Variation in Length	Electronic comparator & Gauge Blocks/

#### National Accreditation Board for Testing and Calibration Laboratories

Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 15 of 133



	Bars	IS:7014	Flatness	Length Measuring Machine/ CMM
3	Gauge Block accessories	IS:4440	Flatness Parallelism	Optical Flat/Measuring Pin Surface Plate & Electronic Comparator
4	Caliper Checker		Pitch Block Accuracy (Length)	Step Gauge/ Gauge Blocks & CMM / Surface Plate & Electronic Comparator
5	Height Master	IS : 13907	Error (Micrometer Screw) over its range – Pitch Block Accuracy Error (Height Setting) over entire range Indexing error check on micrometer drum	Gauge Blocks Surface plate Electronic Comparator
6	Check master		Pitch Block Accuracy (Length)	CMM Or Gauge block, Surface Plate & Electronic Comparator with Probe Or Gauge Block, Surface plate, Dial gauge (lever type) & Height gauge for holding.
7	Cylindrical Setting Standards (Master)	IS:4349	Diameter Variation in diameter Runout	ULM/ Electronic comparator & Gr 0 Gauge Blocks/ CMM Bench centre
8	Raiser block	IS : 13907	Mean height of raiser blocks	Electronic comparator & Gr 0 Gauge Blocks
9	Master cylinder		Circularity Cylindricity Straightness	Form Tester
10	a. Glass Scale/Pick Glass Scale  b. Graticule and Glass Grid  c. Angle Graticule	JIS B 7541	Error between graduation lines over entire length.          Angle	Laser Interferometer          Universal Measuring Microscope OR Laser Interferometer   Profile Projector/ Universal Measuring Microscope (L.C. 30" or better)/ Laser Interferometer

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 16 of 133

#### 4.5 Angle Measuring Devices

Sl. No.	Equipment/ DUC	Relevant Standards/ Guidelines	Parameters to be measured	Master equipment used for calibration
1	Angle Gauges		Error in angle	Autocollimator & Precision Rotary table/ Gauge Blocks Gr 1, Sine Bar, Dial Indicator, Surface Plate, combination of Sine Bar & Slip Gauge set
2	Bevel Protractors Type A, B, C & D	IS:4239	Error of indication  Flatness of Blade  Straightness of working edges of blade	UMM with Precision Dividing Head / Angle Gauges/ Profile Projector/ CMM
3	Sine Center	IS: 5979	Centre distance between rollers Parallelism of centre axis to bottom contact plane of rollers Angular measurement at 15°,30°,45°	Surface plate, Gauge Blocks –Gr 1, Cylindrical Mandrel, Taper Plug Gauge & Dial Indicator Or CMM Angle gauge blocks
4	Single angled Sine table, Size: 100 mm to 200 mm	IS: 5939 IS: 5943	Centre distance between rollers Parallelism of work table top surface to base Angular measurement at 15°,30°,45°	Surface plate, Gauge Blocks –Gr 1, Cylindrical Mandrel, Taper Plug Gauge & Dial Indicator Or CMM Angle gauge blocks
5	Sine Bar, Size:100 mm to 500 mm	IS:5359	Centre distance between rollers Parallelism of working surface to contact surface Angular measurement at 15°,30°,45°	Surface plate, Gauge Blocks –Gr 1, Cylindrical Mandrel, Taper Plug Gauge & Dial Indicator Or CMM Angle gauge blocks
6	Combination Sets	Based on IS: 4239	Error in of Protractor readings over entire range Error in Square head (Angles 90° & 45°) Error in Centre head (Angles 90° & 45°)	Angle Gauge Profile Projector Universal Measuring Microscope
7	Inclinometer		Error in of Scale readings over entire range	Rotary Table & Angle gauge Block
8	Clinometers	MOY/SCMI/36	Error in Bubble Accuracy (reading) Error in of Scale	Electronic Level with Sine bar/ Tilt Table & Meter Bridge & Rotary Table

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 17 of 133

			readings over entire range	
9	Auto Collimator		Error in Auto Collimator readings over entire range	Auto Collimator (Master) Double Sided Plane Mirror Metre Bridge Precision Rotary Table
10	Spirit Level Type 1 (Flat) Type 2 (Vee) Type 3 (Frame Level/ Block level)	IS:5706	Bubble Accuracy (Sensitivity) Consistency (Repeatability) Flatness of Base  Parallelism between working surfaces including Vee Perpendicularity between working surfaces including Vee(for Frame Level only)	Robust tilting Table/ Arm & Electronic Level/ Autocollimator  Dial Indicator 0.001 & Surface plate Movable Stage & Dial Indicator 0.001/ Dial Indicator 0.001, Surface plate, & Precision Cylindrical Mandrel Squareness Tester/ Digital Height Gauge / Engineers or cylindrical square with Dial Indicator
11	Electronic level	JIS B 7510	Error in Electronic level reading over the entire range	Robust tilting Table with Arm & Electronic Level/ Autocollimator (Electronic Level shall comply with TUR 1:3)
12	Polygon mirrors		Error between angles of adjacent faces Cumulative Error between Faces	Auto Collimator, Precision Indexing/ Rotary Table
13	Prisms		Error between angles of adjacent faces Cumulative Error between Faces	Auto Collimator, Precision Indexing/ Rotary Table
14	Rotary table		Error in readings over 360°	Auto Collimator & Precision Polygon Mirror
15	Index table		Error in readings over 360°	Auto Collimator & Precision Polygon Mirror
16	Rotary calibrator		Error in readings over 360°	Precision Indexing/ Rotary Table & Precision Polygon Mirror

#### 4.6 Surface Topography Measuring Devices

Sl. No.	Equipment/ DUC	Relevant Standards/ Guidelines	Parameters to be measured	Master equipment used for calibration
1	Roughness masters	IS:3073 IS:10707	Surface Roughness Parameters according to standard available	Surface Roughness Tester (Stand alone Unit)
2	Depth masters		Groove Depth	Surface Roughness Tester (Stand alone Unit) / Optical Profiler

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 18 of 133

3	Surface Roughness Tester (Portable)		Magnification Surface Roughness Parameters	Roughness masters (min 3 Ra values)
4	Surface Roughness Tester (Stand alone Unit)		Magnification Noise in the instrument Straightness of sideways Surface Roughness Parameters	Depth master Optical Flat  Roughness masters (min 3 Ra values)
5	Surface Profiler / Contour Profiler		Radius Error in Master to fit the required polynomial function	Depth master Optical Flat  Roughness masters (min 3 Ra values)/ Radius Standard/ Contour master
6	Optical Flats – Type A	IS: 5440	Flatness	Flatness Interferometer / Master flat & monochromatic light source
7	Optical Parallels (Optical Flats – Type B) Grade I & II	IS: 5440	Flatness of both faces Parallelism between both faces Size	Flatness Interferometer Flatness Interferometer Gauge Blocks Gr 0 & Electronic Comparator
8	Roundness masters – Hemi-Sphere		Roundness	Roundness Tester
9	Roundness masters – Magnification Master/ Flick Standard		Roundness (flick depth)	Roundness Tester
10	Roundness Tester		Spindle Rotational Accuracy Pickup Magnification	Cylindrical standards, Glass Hemisphere, Cylindrical square, Magnification Master
11	Form Tester		Spindle Rotational Accuracy Pickup Magnification Straightness of Horizontal Arm Straightness of Vertical Column Perpendicularity of Vertical Column to Spindle axis	Cylindrical standards, Glass Hemisphere, Cylindrical square, Magnification Master
12	Radius standards/ Reference Sphere for CMM, Steel Ball		Radius Circularity Surface Roughness	ULM/ CMM Roundness Tester Contour Profiler

#### 4.7 Gauges

Sl. No.	Equipment/ DUC	Relevant Standards/ Guidelines	Parameters to be measured	Master equipment used for calibration
1	A. Limit gauges for internal diameter. (Plain Plug gauges) upto 500 mm	IS:3455 IS: 6137 IS: 6244 IS: 6246	Diameter at four positions	ULM/ Gauge Blocks Gr 0 & Bench comparator/ CMM

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 19 of 133

	A-1 Cylindrical plug gauges A-2 Spherical plug or disc gauges A-3 Segmental cylindrical bar gauge A-4 Segmental spherical plug gauge A-5 Segmental cylindrical bar gauge with reduced measuring faces A-6 Rod gauge with spherical ends			
2	B. Limit Gauges for external diameter B-1 Full form cylindrical ring gauge (Plain Ring Gauge) B-2 Gap Gauges	IS:3455	Diameter at four positions  Dimension across faces Parallelism where applicable	ULM/ CMM  Gauge Block/ ULM/ CMM
3	C. To inspect or adjust Limit gauges C-1 Reference Gauges C-2 Block gauges	IS:3455 IS: 6137 IS: 6244 IS: 6246	Diameter at four positions Roundness	ULM/ CMM Roundness Tester/ Form Tester
4	D - Cylindrical Pins 0.1mm upto 20 mm Grades 0,1, 2	IS: 11103	Diameter	ULM/ Electronic Probe with comparator/ Digital Micrometer L.C. 0.1 $\mu$ m.
5	Ring Gauges  Plain & Master setting ring gauges (Same as C1above)	IS:7876 IS:3455  IS:3485	Diameter at four positions  Diameter at four positions Roundness	ULM/ CMM  ULM/ CMM Roundness Tester/ Form Tester
6	A. Plain Snap Gauges  B. Go & No Go Snap gauges, 3 mm to 250 mm C. Single ended progressive type Plate Snap Gauge 3 mm to 160 mm Same as B2 Above	IS:7876  IS: 3477  IS: 8023 IS: 3455	Gap size Parallelism	ULM/ CMM Gauge Blocks
7	Taper gauges  A. taper plug gauge B. taper ring gauge	IS: 9529 IS: 2251 IS: 9475	Position of Gauge Plane (Gauge Length) Half Taper Angle Major Diameter Straightness of taper Datum step(where applicable)	Sine Bar or Sine Centre with surface plate, Gauge Blocks and Dial Indicator/ CMM Roundness Tester/ ULM, Measuring Pin
8	Thread Plug Gauges A. Go & No go Screw plug gauges & Screw check plug gauges	IS:10685 EURAMET-cg10/V.01	Major diameter Simple effective diameter	Thread Measuring Wires & ULM/ Floating Carriage Micrometer/ Vertical Metroscope

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 20 of 133

	Taper Thread Plug Gauge	ANSI B 1.2 IS 2334 IS 10216 ANSI B 1.5 ANSI B 1.8 ANSI B 1.9 DIN 103  IS 8999 ASME B 1.20.5	Simple effective Diameter at Gauge Plane	
9	A. Thread measuring cylinder/Wire	IS:6311	Actual diameter of individual wire Uniformity of diameter	Electronic Probe with Comparator/ ULM/ / Digital Micrometer L.C. 0.1 μm
10	Thread Ring Gauges  Thread Ring Gauge (1mm to 4mm)  Taper Thread Ring Gauge	IS:2334 EURAMET- cg10/V.01  ANSI B 1.2 IS 2334 IS 10216 ANSI B 1.5 ANSI B 1.8 ANSI B 1.9 DIN 103  IS 8999 ASME B 1.20.5	Minor diameter Simple effective diameter     Simple effective Diameter at Gauge Plane	3 Point Bore gauge ULM  Standard GO/ NOGO Check Plug Gauge. ( Only for 1mm to 4mm Thread ring Gauge )
	A3. Thread Pitch Gauges	IS: 4211	Pitch Flank Angle	Profile Projector/ UMM
11	Form gauges		Profile	Profile Projector
12	Radius gauges (concave and convex profiles), 0.6 to 25 mm	IS: 5273	Radius	Profile Projector

#### 4.8 Miscellaneous

Sl. No.	Equipment/ DUC	Relevant Standards/ Guidelines	Parameters to be measured	Master equipment used for calibration
1	Test Sieves	IS:460 Part I, II, III	Aperture size	Profile Projector/ UMM/ Vernier/ digital Caliper
2	Gears		Involute Profile error Tooth Alignment Error Circular Pitch Error (adjacent & cumulative) Radial Runout	Gear Tester/ CMM
3	Master Gears		Involute Profile error Tooth Alignment Error Circular Pitch Error	Gear Tester

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 21 of 133

			(adjacent & cumulative) Radial Runout	
4	Gear Tester		Involute Profile error Tooth Alignment Error Circular Pitch Error (adjacent & cumulative) Radial Runout Pickup Magnification Error Centre Alignment Error	Involute & Lead Masters Master Gear Gauge Blocks Gr 0, Test Mandrel & Dial Indicator
5	Involute & Lead Masters		Involute Profile error Tooth Alignment Error	Gear Tester
6.	CNC Machine Tools	ISO 230-2, ISO 230-1, VDI/DGQ 3441, IS 2063, JIS B 6192, JIS B 6190	Positional Accuracy (Linear, Angular, Pitch, Yaw, Straightness, Squareness, Flatness)	Laser Interferometer with relevant Optics

- Laboratory may use any other appropriate Reference Standards / Master equipment(s) which shall be capable of measuring above parameters depending on the DUC and its grade/accuracy to meet the requirements of relevant IS/ISO/ other standards.
- The reference standard/ master equipment shall be of accuracy 3 times better than the DUC, including uncertainty.
- Individual thermometers for measurement of temperature of reference equipments and DUC.

**Note:** Reference Gauge Block shall be of '0' Grade or better wherever mentioned as master.

#### 4.9 Industrial Gauge and Template

Sl. No.	Equipment/ DUC	Relevant Standards/ Guidelines	Parameters to be measured	Master equipment used for calibration
1	Welding Fillet Gauge, Templates, Vickers/Knoop/ Rockwell Diamond Cone Indenter/ Weld/ Hi-Lo gauge, bridge cam gauge / Traverse of cupping machine / Limit Gauges/CD Gauge/PCD Gauge / Cube mould/Welding Gauge/Width gauge/ paddle gauge/Weld fillet gauge/ Flakiness gauge/ Elongation gauge/ Receiver Gauge/ Plain work piece/ lever arm/ Master connecting rod/ Inspection JIG and Fixture/Moulds	As applicable	As per requirement inline with relevant standard/ specification	Profile Projector/ Video Measuring Machine/ CMM/ 2D Electronic Height Gauge

Laboratory may use any other appropriate Reference Standards / Master equipment(s) which shall be capable of measuring above parameters depending on the DUC and its grade/accuracy to meet the requirements of relevant IS/ISO/ other standards.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 22 of 133

## 5. Recommended Calibration Procedure: (Some important equations)

### 5.1 Equation for Temperature correction for calibration at dissimilar temperature:

All dimensions reported by the laboratory shall be the dimensions of the artifact at 20°C.

Since the gauge being measured may not be at 20°C, and all artifacts change dimension with temperature change, there is an uncertainty in the length due to uncertainty in temperature. This component is calculated using the following equation.

$$\Delta L = \alpha (20^\circ\text{C} - t) L \quad [2.10.1a]$$

Where,

L is the artifact length at Celsius temperature 't'

$\Delta L$  is the length correction

$\alpha$  is the coefficient of thermal expansion (CTE)

t is the artifact temperature in Celsius

This equation leads to 2 sources of uncertainty in the correction  $\Delta L$ , one from the temperature standard uncertainty ( $u_t$ ) and the other from the CTE standard uncertainty ( $u_\alpha$ )

Equation of uncertainty in length

$$U^2(\Delta L) = [\alpha L u_t]^2 + [L(20^\circ\text{C} - t) u_\alpha]^2 \quad [2.10.1b]$$

If the temperature measurements rely on one thermometer near or attached to one of the gauges there may be another source of uncertainty because of temperature changes between the two gauges. This needs to be considered.

### 5.2 Equation for Temperature correction for calibration at dissimilar temperature and dissimilar metals:

Calibration at a temperature other than 20°C

$$\Delta L = (\alpha_t - \alpha_r) (t - 20^\circ\text{C}) L \quad [2.10.2a]$$

where,

L is the artifact length at Celsius temperature 't'

$\Delta L$  is the length correction

$\alpha_t$  is the coefficient of thermal expansion (CTE) for UUC

$\alpha_r$  is the coefficient of thermal expansion (CTE) for Reference

t is the artifact (UUC) temperature in Celsius

Combination of the above (equation re-written as):

$$\Delta L = L [(t - 20^\circ\text{C}) \alpha_t - (t_r - 20^\circ\text{C}) \alpha_r] \quad [2.10.2b]$$

Where,

t temperature of the UUC

t<sub>r</sub> temperature of the reference

### 5.3 Equation for calculation of error in calibration of Vernier calipers:

The deviation e(result of calibration) is given by expression:

$$e = l_i * (1 + \alpha_m \theta_m) - l_e * (1 + \alpha_e \theta_e) + d_f \quad [2.10.3a]$$

where,

e - deviation (result of calibration) at 20°C

$l_i$  - indicated value on the Vernier caliper (reading)

$\alpha_m$  - Thermal expansion coefficient of Vernier caliper

$\theta_m$  - deviation of temperature of the vernier caliper

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 23 of 133



$l_c$  - calibrated length of the gauge block at 20°C or the value shown by the CMM  
 $\alpha_e$  - Thermal expansion coefficient of the gauge block(or the measuring system of the CMM)  
 $\theta_e$  - deviation of temperature of the gauge block(or the measuring system of the CMM)  
 $d_F$  - difference of the deformation caused by measuring force in measurement force and in calibration (assumed to be 0)

If new quantities are defined as :

$$\delta\theta = \theta_m - \theta_e$$

$$\delta\alpha = \alpha_m - \alpha_e$$

Then equation [8.10.3a] gets the following form:

$$e = l_i * (1 + \alpha_m \theta_e + \alpha_m \delta\theta) - l_e * (1 + \alpha_m \theta_e - \delta\alpha \theta_e) + d_F \quad [2.10.3b]$$

**5.4 Same equations given above can be used for calculation of error in case of Micrometers and Dial gauges by substituting accordingly:**

- a) Micrometer or Dial gauge(for  $l_i$ ,  $\alpha_m$ ,  $\theta_m$ ) in place of vernier caliper and
- b) (For  $l_e$ ,  $\alpha_e$ ,  $\theta_e$ ) the calibration device used for calibration of micrometer or dial gauge.

Combination of the above (equation re-written as):

$$\Delta L = L [(t_r - 20^\circ\text{C}) - (t_r - 20^\circ\text{C})] \quad 2.10.4b]$$

Where,

$t_t$  temperature of the UUC

$t_r$  temperature of the reference

**5.5 Equation for calculation of error in calibration of the Optical projector:**

The mathematical model of magnification of optical profile is expressed as

$$M = \frac{\Delta L + L_2(1 + \alpha_2 \Delta T_2)}{L_1(1 + \alpha_1 \Delta T_1)} \quad [2.10.5a]$$

Where,

M –magnification measured

$\Delta L$  – Length of deviation of standard scale image from reading scale (measurands)

$L_1, L_2$  – Length of standard and reading scale at 20°C

$\alpha_1, \alpha_2$  – Thermal expansion coefficient of standard and reading scale

$\Delta T_1$  - Temperature difference between standard scale and reference temperature 20°C

$\Delta T_2$  - Temperature difference between reading scale and reference temperature 20°C

The magnification error of optical projector  $\Delta M$  is defined as below:

$$\Delta M = \frac{M - MN}{MN} \times 100\% \quad [2.10.5b]$$

Where,

MN Nominal magnification (constant)

Equation (2.10.5b) can be modified to (2.10.5c) by substituting the term M in Equation (2.10.5a)

$$\Delta M = \left( \frac{\Delta L + L_2(1 + \alpha_2 \Delta T_2)}{L_1(1 + \alpha_1 \Delta T_1)MN} - 1 \right) \times 100\% \quad [2.10.5c]$$

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 24 of 133

## 5.6 Equation for calculation of error in calibration of a Gauge Block:

The length  $l_x$  of the unknown gauge block at the reference temperature is obtained by the relationship:

$$l_x = [l_s + \delta l_D + \delta l + \delta l_c - L(\bar{\alpha} X \delta t + \delta \alpha X \Delta \bar{t}) - \delta l_v]$$

Where,

$l_s$  - length of the reference gauge block at the reference temperature  $t_0 = 20^\circ\text{C}$  according to its calibration certificate;

$\delta l_D$  - Change of the length of the reference gauge block since its last calibration due to drift

$\delta l$  - observed difference in length between the unknown and the reference gauge block;

$\delta l_c$  - correction for non-linearity and offset of the comparator;

$L$  - nominal length of the Gauge Blocks considered;

$\bar{\alpha} = (\alpha_x + \alpha_s)/2$  - average of the thermal expansion coefficients of the unknown and reference gauge blocks;

$\delta t = (t_x - t_s)$  - temperature difference between the unknown and reference Gauge Blocks

$\delta \alpha = (\alpha_x - \alpha_s)$  - difference in the thermal expansion coefficients between the unknown and the reference Gauge Blocks;

$\Delta \bar{t} = (t_x + t_s)/2 - t_0$  - deviation of the average temperature of the unknown and the reference gauge blocks from the reference temperature;

$\delta l_v$  = correction for non-central contacting of the measuring faces of the unknown gauge block.

## 6. Measurement Uncertainty

6.1 Repeatability (Type A)

6.2 Resolution - 1/4 in case of analog gauges (however, 1/10 can be used when DUC is used for referencing), 1/2 in case of digital instruments

6.3 Uncertainty of master (s) [When error of the Master is not corrected during calibration then it should be added as an additional uncertainty component]

6.4 Geometrical errors like flatness, straightness etc.

6.5 Effect of Temperature:

- Deviation from reference Temperature (ambient) i.e. 20 deg C.
- Difference in temperature between DUC and master (if not measured, 20% deviation from reference temperature has to be taken)
- Difference in Thermal expansion coefficient
- Uncertainty in Thermal expansion coefficient (ref. NABL 141)

6.6 Error & Uncertainty of temperature monitoring equipments

6.7 Error: Half of the error in the masters is to be considered for uncertainty evaluation, wherever these are not compensated.

## 7. Recommended National/ International Standards, References and Guidelines

**Linear Measuring Devices:**

**7.1 Measuring Instruments:**

- IS: 3651 (Part I, II, and III) – Specification for Vernier Calipers.
- IS: 2092 - Specification for Plunger Type Dial Gauges.
- IS: 11498 - Specification for lever type dial gauges.
- IS: 2966 - Specification for internal Micrometers (including Stick Micrometers).
- IS: 2967 - Specification for External Micrometer.
- IS: 13907- height setting micrometer and rising block
- IS: 1481 - Specification for Metric Steel Scales for Engineers.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 25 of 133

- IS:1270 - Metric steel tape (winding type)
- IS:1269 Part I-Material measure of length, Part 1: Woven metallic and glass fibre tape measures
- IS:12872 - Metallic materials- Verification of Extensometers used in uniaxial testing
- IS:15372 - Measurement of Roundness-Terms, definitions and parameters of roundness
- IS: 4213 - Engineering Metrology – Vernier depth gauges up to 300 mm: Specifications.
- IS: 15374 - Engineering Metrology –Methods for the assessment of departure from roundness-measurement by two and three point.

### 7.2 Master Equipments:

- IS15635/ ISO10360-2- Performance assessment of coordinate measuring machines
- Euramet Cg-2- Calibration of gauge block comparators
- ISO 10936-1 Optical and optical instruments –operation microscopes- part-1 requirements and test methods

### 7.3 Measuring Aids:

- IS: 2063/ ISO 230 Part 1- Test Code for Machine Tools – Geometric Accuracy of Machines Operating under no Load or Finishing Conditions
- IS: 2285 - Engineering Metrology – Measuring Equipment – Cast Iron Surface Plates – Specification.
- IS: 7327 - Engineering Metrology – Measuring Equipment – Granite Surface Plates – Specification.
- IS: 2554 - Specification for Cast Iron Angle Plates.
- IS: 6232 - Specification for Cast Iron Box Angle Plates.
- IS: 2103 - Specification for Engineer's Squares.
- IS: 2220 - Engineering Metrology – Steel Straight edges : Specifications.
- IS: 4241 - Engineering Metrology – Engineer's Parallels – Specification.
- IS: 6952 - Engineering Metrology – Cylindrical squares
- IS: 6973 - Engineering Metrology- Precision Angle plates
- IS: 2949 - Engineering Metrology – Plain V-Blocks for inspection purposes – Specification.
- IS: 4492 - Engineering Metrology -Welded V-blocks (Diameter Range 300 to 2000 mm)
- IS: 4960 - Engineering Metrology- Universal and elongated type V-Blocks
- IS: 6232 - Engineering Metrology- Cast Iron Box Angle Plates
- 

### 7.4 Reference Masters:

- IS: 2984 - Specification for Gauge Blocks.
- ISO 3650 - Gauge Blocks
- IS: 2921 - Specification for Vernier Height Gauges.
- IS: 9483 - Measuring instruments-micrometer head
- IS:7014 - Engineering Metrology – Length Bars
- IS:4440 - Engineering Metrology – Precision Equipment - Gauge Block Accessories

### 7.5 Angle Measuring Devices:

- IS:4239 - Engineering Metrology – Mechanical Bevel Protractors
- IS:5939 - Engineering Metrology – Single Angle Sine Tables
- IS:5359 - Engineering Metrology – Sine Bars
- IS: 5979 - Engineering Metrology – Sine centres
- IS:5706 - Optical instruments - Spirit levels for use in precision engineering

### 7.6 Surface Topography Measuring Devices:

- IS:10707 -Instrument for measurement of surface roughness by profile methods
- IS:3510 - Engineering Metrology – Toolmaker's Flats

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 26 of 133

### 7.7 Gauges:

- IS: 3455 - Gauging Practice for Plain Work pieces.
- IS:7876 - Engineering Metrology - Gauge allowances and manufacturing tolerances for plain gauges for outside measurements for ISO fit sizes (nominal size up to 500 mm)
- IS:3485 - Engineering Metrology - Plain and Master Setting Ring Gauges (Size Range from 1 up to and Including 315 mm)
- IS: 3477 - Engineering Metrology – ‘Go’, ‘No Go’ Snap gauges
- IS: 8023 - Gauges – Single ended Progressive type plate snap gauges (up to 160 mm): Specification.
- IS: 9529 - Engineering Metrology - Taper Plug Gauges and Taper Ring Gauges to Check Taper Bore of Gauge Handles and Taper Shanks of Gauging Members
- IS: 5273 - Specification for Radius Gauges.
- IS: 3179 -Engineering Metrology – Feeler Gauges.

### 7.8 Miscellaneous:

- IS 460 (Part I, II and III) – Specification for Test Sieves.

**Note:** This technical requirement is based on the above mentioned guideline. Lab may follow any relevant standard; however care shall be taken to follow the requirements in totality.

## 8. Recommended Calibration interval

Reference Equipment	Recommended interval
Autocollimator, polygon mirror, Hemisphere, Magnification standards, master cylinder, Radius master, Reference Sphere, Gauge Blocks (00/K grade if calibrated by interferometry method), Step Gauge, Laser Interferometer, Involute & Profile Master, Master Gear	3 years
Gauge Blocks (comparison method of calibration), Master setting ring, Optical Flat, Depth Master, Roughness Master, Floating Carriage Diameter Measuring Machine, Cylindrical Setting Standards, Electronic Height Gauge, All Measuring equipments / machines which have moving / rotating parts like, reference micrometers, profile projectors, Length measuring machine, ULM and scale calibrator, CMM, Roundness tester, Surface roughness tester, Caliper Checkers, Glass Scale,	2 years
Standard Foil, Electronic / Spirit Level, Measuring Tape calibrator	1 year

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 27 of 133

## Chapter 1B: Calibration of Mass (Weights)

### 1. Recommended General Requirements

- 1.1 **Acoustic Noise:** Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 **Illumination:** The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.
- 1.6 The calibration of weights shall be performed at suitable conditions under ambient atmospheric pressure at temperatures closer to room temperature (1) Typical recommended values are given below:

Weight Class	Temperature change during Calibration(2)	Range of Relative Humidity of the Air(3)
E1	$\pm 0.3^\circ\text{C}$ per hour with a maximum of $\pm 0.5^\circ\text{C}$ per 12 hours	40% to 60% with a maximum of $\pm 5\%$ per 4 hours
E2	$\pm 0.7^\circ\text{C}$ per hour with a maximum of $\pm 1^\circ\text{C}$ per 12 hours	40% to 60% with a maximum of $\pm 10\%$ per 4 hours
F1	$\pm 1.5^\circ\text{C}$ per hour with a maximum of $\pm 2^\circ\text{C}$ per 12 hours	40% to 60% with a maximum of $\pm 15\%$ per 4 hours
F2	$\pm 2^\circ\text{C}$ per hour with a maximum of $\pm 3.5^\circ\text{C}$ per 12 hours	
M1	$\pm 3^\circ\text{C}$ per hour with a maximum of $\pm 5^\circ\text{C}$ per 12 hours	

**Note (1):** It is also important that the difference in temperature between the weights and the air inside the mass comparator is as small as possible. Keeping the reference weight and the test weight inside the mass comparator before and during the calibration to reduce the temperature difference.

**Note (2):** This is the change in the temperature of the laboratory. Thermal stabilization of balances and weights also requires an appropriate temperature stability of laboratory for 24 hours before calibration.

- 1.7 Thermal Stabilization Requirement for Test Weights: Prior to performing any calibration, the weights need to be acclimated to the ambient conditions of the laboratory. In particular, weights of classes E1, E2 and F1 shall be close to the temperature in the weighing area.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 28 of 133

Thermal stabilization in hours:

$\Delta T^*$	Nominal value	Class E1	Class E2	Class F1	Class F2
$\pm 20^\circ\text{C}$	1 000, 2 000, 5 000 kg	-	-	79	5
	100, 200, 500 kg	-	70	33	4
	10, 20, 50 kg	45	27	12	3
	1, 2, 5 kg	18	12	6	2
	100, 200, 500 g	8	5	3	1
	10, 20, 50 g	2	2	1	1
	< 10 g	1		0.5	
$\pm 5^\circ\text{C}$	1 000, 2 000, 5 000 kg	-	-	1	1
	100, 200, 500 kg	-	40	2	1
	10, 20, 50 kg	36	18	4	1
	1, 2, 5 kg	15	8	3	1
	100, 200, 500g	6	4	2	0.5
	10, 20, 50 g	2	1	1	0.5
	< 10 g	0.5			
$\pm 2^\circ\text{C}$	1 000, 2 000, 5 000 kg	-	-	1	0.5
	100, 200, 500 kg	-	16	1	0.5
	10, 20, 50 kg	27	10	1	0.5
	1, 2, 5 kg	12	5	1	0.5
	100, 200, 500 g	5	3	1	0.5
	< 100 g	2	1		0.5
	$\pm 0.5^\circ\text{C}$	1 000, 2 000, 5 000 kg	-	-	-
100, 200, 500 kg		-	1	0.5	0.5
10, 20, 50 kg		11	1	0.5	0.5
1, 2, 5 kg		7	1	0.5	0.5
100, 200, 500 g		3	1		0.5
< 100 g		1	0.5		

$\Delta T^*$  = Initial difference between weight temperature and laboratory temperature.

**Note:** Thermal stabilization hours before calibration should be clearly mentioned in the certificate issued to the customer.

## 2. Specific Requirement – Calibration of Weights

### 2.1 Scope: Calibration of Weights

Specific Requirements for calibration of weights with following details:

Sl. No.	Description	Relevant Standard	Permanent facility	Onsite calibration	Mobile facility
1	Weights (E <sub>1</sub> , E <sub>2</sub> , F <sub>1</sub> , F <sub>2</sub> , M <sub>1</sub> , M <sub>2</sub> , M <sub>3</sub> )	OIML-R 111-1	√	X	X

**Note 1:** Newtonian weights, non-metric weights can also be calibrated to accuracy class equivalent to OIML R111 -1. However, the conventional mass values and its uncertainty should be given in SI units.

**Note 2:** This technical requirement is based on the above-mentioned guideline. Lab may follow any relevant standard; however care shall be taken to follow the requirements in totality.

**Note 3:** ASTM standard weights can also be calibrated if the relevant standard is followed in total.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 29 of 133

**Note 4:** Laboratory shall apply for calibration and not for verification of Weights for commercial application. Verification may require approval from Dept. of Legal Metrology, Regulatory Bodies, etc.

### 3. Selection of Reference weights

The reference weight shall generally be of a higher class of accuracy than the weight to be calibrated. In the calibration of weights of class E<sub>1</sub>, the reference weight shall have similar or better metrological characteristics (magnetic properties, surface roughness) than the weights to be calibrated.

Weight that can be used as Reference	Class of Weights that can be calibrated						
	E <sub>1</sub>	E <sub>2</sub>	F <sub>1</sub>	F <sub>2</sub>	M <sub>1</sub>	M <sub>2</sub>	M <sub>3</sub>
E <sub>1</sub>	√	√	√	√	√	√	√
E <sub>2</sub>	-	-	√	√	√	√	√
F <sub>1</sub>	-	-	-	√	√	√	√
F <sub>2</sub>	-	-	-	-	√	√	√

**Note:** M<sub>1</sub>, M<sub>2</sub> and M<sub>3</sub> class weights are not recommended to be used as a reference for the calibration of weights. (Important Technical Points 2.15). However, M<sub>1</sub> weights can be used for above 20 kg with coarser uncertainty.

### 4. Selection of comparator/ balance

On the basis of the accuracy class, a mass comparator is to be selected in such a way that its uncertainty component is balanced in proportion to the overall uncertainty of the weighing result. The most important uncertainty component of a mass comparator is calculated from its standard deviation. The specification of the manufacturer can be selected as a first approximation for the value of a standard deviation. It must be taken into account. However, that this indication is decisive for the smallest nominal value. It should therefore not exceed an amount of 30% of the combined standard uncertainty u<sub>1</sub> (k=2).

#### 4.1 Example:

weight to be Calibrated	Class	Permissible Error as per OIML R 111	Uncertainty required (1/3 of the error) with k=2	Standard deviation of the comparator required =
1 mg	E <sub>2</sub>	0.006 mg	0.002 mg	(0.002)/3 mg S ≤ 0.00067 mg

Note: It is not recommended to calibrate a higher accuracy class weight with a lower accuracy class of reference weight and comparator/balance with coarser resolution without the consent of the customer.

#### 4.2 Selection of comparator balance for calibration of weights depending on class of accuracy

Nominal Value		E <sub>1</sub>	E <sub>2</sub>	F <sub>1</sub>	F <sub>2</sub>	M <sub>1</sub>	M <sub>2</sub>	M <sub>3</sub>
		Standard deviation of repeatability in mg						
5000	kg			2778	8889	27778	88889	277778
2000	kg			1111	3333	11111	33333	111111
1000	kg		178	556	1778	5556	17778	55556
500	kg		89	278	889	2778	8889	27778
200	kg		33	111	333	1111	3333	11111
100	kg		17.8	56	178	556	1778	5556
50	kg	2.78	8.9	28	89	278	889	2778
20	kg	1.11	3.3	11	33	111	333	1111

### National Accreditation Board for Testing and Calibration Laboratories

Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 30 of 133

10	kg	0.56	1.78	6	18	56	178	556
5	kg	0.28	0.89	2.8	8.9	28	89	278
2	kg	0.11	0.33	1.11	3.3	11.1	33	111
1	kg	0.056	0.178	0.556	1.78	5.56	17.8	56
500	g	0.028	0.089	0.278	0.89	2.78	8.89	28
200	g	0.011	0.033	0.111	0.33	1.11	3.33	11.1
100	g	0.006	0.018	0.056	0.18	0.556	1.78	5.56
50	g	0.0033	0.011	0.034	0.11	0.333	1.11	3.33
20	g	0.0028	0.009	0.028	0.089	0.278	0.889	2.78
10	g	0.0022	0.007	0.022	0.067	0.222	0.667	2.22
5	g	0.0018	0.006	0.018	0.056	0.178	0.556	1.78
2	g	0.0013	0.004	0.013	0.044	0.133	0.444	1.33
1	g	0.0011	0.0034	0.011	0.033	0.111	0.333	1.11
500	mg	0.0009	0.0028	0.009	0.028	0.089	0.278	8.89
200	mg	0.0007	0.0022	0.007	0.022	0.067	0.222	
100	mg	0.0006	0.0018	0.006	0.018	0.056	0.178	
50	mg	0.0004	0.0013	0.004	0.013	0.044		
20	mg	0.0003	0.0011	0.0034	0.011	0.034		
10	mg	0.0003	0.0009	0.0028	0.009	0.028		
5	mg	0.0003	0.0007	0.0022	0.006	0.022		
2	mg	0.0003	0.0007	0.0022	0.006	0.022		
1	mg	0.0003	0.0007	0.0022	0.006	0.022		

## 5. Recommended Calibration interval

For the reference Weights and comparators used in calibration of Weights at permanent lab facility.

Reference Equipment	Recommended interval
Weights of E <sub>1</sub> to F <sub>2</sub> class	3 years
Weights of class M1	2 years
Comparator / Balance	1 year

(Based on the historical data validity of reference weights may be extended upto 5 years for E1)

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

## 6. Metrological Requirements

6.1 For Each weight, the expanded uncertainty, U, for k=2, of the conventional mass, shall be less than or equal to one third of the maximum permissible error.

6.2 For each weight, the conventional mass, m<sub>c</sub> (determined with an expanded uncertainty, U, according to 5.2 of OIML R-111-1) shall not differ from the nominal value of the weights, m<sub>0</sub> by more than the maximum permissible error, δm minus the expanded uncertainty.

$$m_0 - (\delta m - U) \leq m_c \leq m_0 + (\delta m - U)$$

6.3 For class E1 and E2 weights, which are always accompanied by certificates giving the appropriate data, the deviation from the nominal value, m<sub>c</sub>-m<sub>0</sub>, shall be taken into account by the user.

6.4 Calibration certificate shall state, as a minimum: the conventional mass of each weight, m<sub>c</sub> an indication of whether a weight has been adjusted prior to calibration, its expanded uncertainty U and the values of the coverage factor k.

6.5 The certificate for class E1 weights shall state, as a minimum, the values of conventional mass, m<sub>c</sub>, the expanded uncertainty, U, and the coverage factor k and density or volume for each weight. In addition, the certificate shall state if the density or volume was measured or estimated.

6.6 The certificate for class E2 weights shall state, as a minimum, the value of conventional mass m<sub>c</sub>, of each weight, the expanded uncertainty, U, and the coverage factor k.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 31 of 133



6.7 Altitude and corresponding changes in air density can affect the measurement error when using the conventional mass of weight; therefore, the buoyancy correction shall be used, which requires the density of the weight to be known. If class E weights are to be used above 330 m, the density of the weights shall be provided along with their associated uncertainty for Class F1 the same is true above 800 m. Otherwise, the manufacturer shall take the lowered buoyancy affect at higher altitude in to consideration in specifying the weights class for the standards of conventional mass.

## 7. Calibration Methods

There are two methods for determination of conventional mass of weights in a weight set.

### 7.1 Direct Comparison Method

Minimum Number of Weighing Cycles (as per OIML R-111-1)					
Class	E1	E2	F1	F2	M1, M2, M3
Min. number of ABBA	3	2	1	1	1
Min. number of ABA	5	3	2	1	1
Min. number of AB1...BnA	5	3	2	1	1

### 7.2 Sub -Division/Sub-Multiplication Method (Ref. C.3.2 of OIML R-111-1)

Reference Weights	Vs	5+2+2*+1
Reference Weights	Vs	5+2+2*+1*
5	Vs	2+2*+1
5	Vs	2+2*+1*
2+1	Vs	2*+1*
2+1	Vs	2+1
2+1*	Vs	2*+1
2+1*	Vs	2*+1
2	Vs	1+1*
2	Vs	1+1*
2*	Vs	1+1*
2*	Vs	1+1*

**Note:** Method used for calibration should be clearly mentioned in the calibration certificate issued to the customer.

### 7.3 Legal Aspects

Calibration of weights done by any accredited laboratories is meant for scientific and industrial purpose only. However, if used for commercial trading, additional recognition/ approval shall be complied as required by Dept. of Legal metrology, Regulatory bodies, etc. This should be clearly mentioned in the calibration certificate issued to the customer.

## 8. Determination of Air Density and its Uncertainty

6.1 In 2008 CIPM recommended that the following equation be used to determine  $\rho_a$  the density of Air

$$\rho_a = [3.483740 + 1.4446 \cdot (x_{CO_2} - 0.0004)] \cdot 10^{-3} \cdot p / ZT \cdot (1 - 0.378 \cdot x_v)$$

Where,  $\rho_a$  : Density of air in kg/m<sup>3</sup>

$x_{CO_2}$  : The mole fraction of carbon dioxide (assumed 400 ppm, if not measured)

p : pressure

Z : compressibility

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 32 of 133

$x_v$  : mole fraction of water vapor  
 T: Thermodynamic temperature using ITS-90

## 9. Calibration of Newton Weights, Pressure Balance Weights and Non-metric Weights

- 9.1 Weights used for realization of Pressure in Dead weight pressure balance or weights used for realization of force in Newton are to be calibrated on true mass basis.
- 9.2 If, weights are calibrated on conventional mass basis equation for conversion from conventional mass to true mass to be mentioned to enable the user to apply appropriate buoyancy correction.
- 9.3 Newton or Force weights are typically of a slotted design or with a centre hole and are typically marked with a nominal Force in Newton. Force is calculated with respect to Local gravity 'g<sub>L</sub>' during calibration using the formula given below:

$$F = m (1 - \rho_a / \rho_m) * g_{LC} \quad [2.19.3a]$$

Where

F = Force in Newton

m = True Mass in Kg

$\rho_a$  = air density in kg/m<sup>3</sup>

$\rho_m$  = density of weights in kg/m<sup>3</sup>

$\gamma_L$  = Local gravity in m/sec<sup>2</sup> (value of 'g' at the customer's site )

The force values can be converted either to the standard 'g' value or to the customer's 'g' value using the formula given below:

To convert Force to standard 'g' value:

$$F = m (1 - \rho_a / \rho_m) * g_s / g_{LC} \quad [2.19.3b]$$

Where,

$g_s$  = Standard gravity in m/sec<sup>2</sup> (9.80665 m/sec<sup>2</sup>)

$g_{LC}$  = Local gravity in m/sec<sup>2</sup> (value of 'g' at the site of calibration).

To convert Force to 'g' value at customer's site:

$$F = m (1 - \rho_a / \rho_m) * g_L / g_{LC} \quad [2.19.3c]$$

Where,

$g_L$  = Local gravity in m/sec<sup>2</sup> (value of 'g' at the customer's site)

This conversion can be done if; the customer provides 'g' value at his site.

9.4 When the customer requires the force weight with respect to his local 'g' value he has to provide the same with uncertainty. Then the force value shall be calculated using the local 'g' value and declare in the certificate in terms mass value along with the calculated value in Newton. 'g' value of the calibration laboratory shall also be known to sufficient accuracy.

9.5 The Laboratory may calibrate weights of non-metric units (e.g. Pound or Ounce etc.) However, the results shall be reported in SI units like kg, g, mg, etc. along with the calculated equivalent value in the non-metric unit or mention the conversion factor to be used.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 33 of 133

Table 1 Maximum permissible errors for weights ( $\pm \delta m$  in mg)

Nominal value*	Class E <sub>1</sub>	Class E <sub>2</sub>	Class F <sub>1</sub>	Class F <sub>2</sub>	Class M <sub>1</sub>	Class M <sub>1-2</sub>	Class M <sub>2</sub>	Class M <sub>2-3</sub>	Class M <sub>3</sub>
5 000 kg			25 000	80 000	250 000	500 000	800 000	1 600 000	2 500 000
2 000 kg			10 000	30 000	100 000	200 000	300 000	600 000	1 000 000
1 000 kg		1 600	5 000	16 000	50 000	100 000	160 000	300 000	500 000
500 kg		800	2 500	8 000	25 000	50 000	80 000	160 000	250 000
200 kg		300	1 000	3 000	10 000	20 000	30 000	60 000	100 000
100 kg		160	500	1 600	5 000	10 000	16 000	30 000	50 000
50 kg	25	80	250	800	2 500	5 000	8 000	16 000	25 000
20 kg	10	30	100	300	1 000		3 000		10 000
10 kg	5.0	16	50	160	500		1 600		5 000
5 kg	2.5	8.0	25	80	250		800		2 500
2 kg	1.0	3.0	10	30	100		300		1 000
1 kg	0.5	1.6	5.0	16	50		160		500
500 g	0.25	0.8	2.5	8.0	25		80		250
200 g	0.10	0.3	1.0	3.0	10		30		100
100 g	0.05	0.16	0.5	1.6	5.0		16		50
50 g	0.03	0.10	0.3	1.0	3.0		10		30
20 g	0.025	0.08	0.25	0.8	2.5		8.0		25
10 g	0.020	0.06	0.20	0.6	2.0		6.0		20
5 g	0.016	0.05	0.16	0.5	1.6		5.0		16
2 g	0.012	0.04	0.12	0.4	1.2		4.0		12
1 g	0.010	0.03	0.10	0.3	1.0		3.0		10
500 mg	0.008	0.025	0.08	0.25	0.8		2.5		
200 mg	0.006	0.020	0.06	0.20	0.6		2.0		
100 mg	0.005	0.016	0.05	0.16	0.5		1.6		
50 mg	0.004	0.012	0.04	0.12	0.4				
20 mg	0.003	0.010	0.03	0.10	0.3				
10 mg	0.003	0.008	0.025	0.08	0.25				
5 mg	0.003	0.006	0.020	0.06	0.20				
2 mg	0.003	0.006	0.020	0.06	0.20				
1 mg	0.003	0.006	0.020	0.06	0.20				

## 10. National/ International Standards, References and Guidelines

- OIML R111-1-2004 Metrological and technical requirement of weights Classes E<sub>1</sub>, E<sub>2</sub>, F<sub>1</sub>, F<sub>2</sub>, M<sub>1</sub>, M<sub>2</sub>, M<sub>3</sub>.
- OIML D28 2004: Conventional value of the result of weighing in air.
- ASTM E617 - 13 Standard Specifications for Laboratory Weights and Precision Mass Standards.
- OIML R 47 Edition 1079(E) - Standard weights for testing of high capacity weighing machines

**Note: - Latest version of the relevant standard(s) should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 34 of 133

# Chapter 1(C): Calibration of Mass (Weighing Scale & Balance)

## 1 Recommended General Requirements

- 1.1 Acoustic Noise: Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 Illumination: The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.

### 1.6 Thermal Stabilization Requirements

The effect of Convection:

- When the reference weights used are transported for calibration site, they may not have the same temperature as that of the balance and its environment, then there will be heat exchange between the weights and their environment. Due to this there will be apparent change in mass in relation to the temperature difference.
- An initial temperature may be reduced to a smaller value by acclimatization (habituation to a new climate) over time. This occurs faster for smaller weights than for larger weights.
- When a weight is put on the load receptor of a balance, the actual difference in temperature will produce an air flow about the weight leading to parasitic forces which results in conventional mass of the weight. This value will be greater for large weights than for small ones.

Hence, proper thermal stabilization time may be required before proceeding for calibration of the weighing balance.

### 1.7 Effect of Gravity 'g' on Calibration of Balance

- i. The weighing values are different when the weighing height changes.
- ii. The further a weight is from the centre of earth, the smaller the gravitational force acting on it. It decreases with the square of the distance.  
Example: The weight display changes when the weighing is performed at 10 m higher (moving from the first floor to fourth floor of a building). To determine the weight of a body, the balance measures the weight force i.e., the force of attraction (Gravitation force) between the earth & the weighing sample. The force depends essentially on the latitude of the location & its height above sea level (distance from the centre of earth).
- iii. The nearer a location is to the equator, the greater the centrifugal acceleration due to the rotation of the earth. The centrifugal acceleration counteracts the force of attraction (Gravitation Force).
- iv. The poles are the greatest distance from the equator & closest to the earth centre. The force acting on a mass is therefore greatest at the poles.

**Example:** In the case of 200 g weight that shows exactly 200.00000 g on the first floor, the following weight result on the fourth floor (10m height).

$$200g \times r^2 / (r + \Delta)^2$$

Where, r is the radius of earth at that point of measurement,  $\Delta$  is change in height

$$200 \times 6370000^2 / 6370010^2 = 199.99937g$$

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 35 of 133

## 2 General Requirements - Calibration of Weighing Scale/Balance

### 2.1 Scope: Calibration of Weighing Balance and Mass Comparator

#### 2.1.1. Specific Requirements

Sl. No	Parameter	Relevant Standard	Permanent Facility	Onsite Calibration	Mobile Facility
1	Non-automatic Weighing Balances	EURAMET cg -18/v.3 or OIML-R-76-1 and OIML-R76-2	√*	√	x
2	Electronic Balances		√*	√	x
3	Comparators		√*	√	x
4	High Capacity Weighing Machines (above 50 kg)	OIML R 47	√*	√	x

**\*Note – 1** Permanent facility for their internal use only and not meant for external customer.

**Note – 2** This technical requirement is based on the above-mentioned guideline. Lab may follow any relevant standard; however, care shall be taken to follow the requirements in totality.

## 3 Metrological Requirements

3.1 Reference and Standard weights used for calibration of weighing balance and comparator shall follow the requirement of OIML R-111-1.

3.2 Place of Calibration: Calibration shall be performed at the place where the balance is being used. If the balance is moved to another location after calibration, possible effects are due to:

- difference in local 'g' acceleration due to gravity (refer 2.10)
- variation in environmental conditions.
- mechanical and thermal conditions during transportation and are likely to alter the performance of the balance and may invalidate the status of calibration. Moving the balance after calibration shall therefore be avoided and the calibration certificate shall not be accepted as evidence of traceability.

3.3 Lab cannot calibrate balances using lower class of accuracy weights than required (Table 2.5.2)

### Classification of Weighing Scale/Balances

Weighing Scale/balances are classified into different accuracy class based on their minimum readability

3.4 Following types of balances are considered in this guideline: Table-1

Sl.	Type of Balance	Minimum Readability (d)	No. of Digits after Decimal Place (g)	Accuracy Class
1	Ultra-micro balance	d = 0.1 μg = 0.0000001 g	7	I
2	Micro balance	d = 1 μg = 0.000001 g	6	I
3	Semi-micro balance	d = 0.01 mg = 0.00001 g	5	I
4	Analytical balance	d = 0.1 mg = 0.0001 g	4	I
5	Precision balance	d = 50 mg to 1 mg = 0.05 g to 0.001 g	2 to 3	II
6	Medium balance	d = 1 g to 2 g	0	III
7	Ordinary balance	d > 5 g	0	IV

### National Accreditation Board for Testing and Calibration Laboratories

Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 36 of 133

**Classifications are based on OIML R-76-1: Table-2**

Accuracy Class		Verification of Scale Interval, e	Number of Verification Scale Intervals, n = Max/e		Minimum Capacity, Min (Lower limit)
			Minimum	Minimum	
Special	I	$0.001\text{ g} \leq e^*$	50 000**	-	100 e
High	II	$0.001\text{g} \leq e \leq 0.05\text{ g}$ $0.1\text{ g} \leq e$	100 5 000	100 000 100 000	20 e 50 e
Medium	III	$0.1\text{g} \leq e \leq 2\text{ g}$ $5\text{ g} \leq e$	100 500	10 000 10 000	20 e 20 e
Ordinary	IIII	$5\text{ g} \leq e$	100	1 000	10 e

\* It is not normally feasible to test and verify an instrument  $e < 1\text{ mg}$ , due to uncertainty of the test loads.

\*\* Refer see exception in 3.4.4 of OIML R 76-1.

**Selection of Reference Weights for Balance/ Comparator Calibration**

3.4.1. The reference weight used for calibration shall have traceability and satisfy the following: 3.4.1.1. Determination of the known conventional value of mass  $m_c$  and/or the correction  $\delta m_c$  to its nominal value  $m_N$  :  $\delta m_c = m_c - m_N$ , together with the expanded uncertainty of the calibration  $U_{95}$ , or

3.4.1.2. Confirmation that  $m_c$  is within specified maximum permissible errors.  $mpe$ :  $m_N - (mpe - U_{95}) \leq m_c \leq m_N + (mpe - U_{95})$

3.4.1.3. The standards shall further satisfy the following requirements to the extent as appropriate in view of their accuracy: a) density  $\rho_s$  sufficiently close to  $\rho_C = 8000\text{ kg/m}^3$ , b) surface finish suitable to prevent a change in mass through contamination by dirt or adhesion layers, c) magnetic properties such that interaction with the instrument to be calibrated is minimized.

3.4.1.4. Weights that comply with the relevant specifications of the International Recommendation OIML R 111 [4] should satisfy all these requirements.

3.4.1.5. The maximum permissible errors or the uncertainties of calibration of the standard weights shall be compatible with the scale interval of the instrument and/or the needs of the customer with regard to the uncertainty of the calibration of his instrument.

3.4.2. A probable selection of weights for calibration of weighing machines depending on capacity and scale division (resolution):

Class of weights to be selected	n = Capacity divided by scale division(resolution)				
	n>300000 or above	n> 100000 to 300000	n> 30000 to 100000	n>10000 to 30000	n = 10000 or smaller
E1	√	√	√	√	√
E2		√	√	√	√
F1			√	√	√
F2				√	√
M1					√

The design and accuracy of weights used for in-house calibrations shall be appropriate to the weighing machine being calibrated, and where possible should have a 95% confidence level uncertainty of calibration less than half the smallest digit size or recorded scale interval of the weighing machine to be calibrated. Where groups of weights are to be used to make up a single load, this criterion shall be applied to the arithmetic sum of the weight's individual calibration uncertainties.

3.4.3. Weighing machines as described in Table 1 can usually be calibrated using calibrated weights in the pattern of the designated OIML class. The table assumes that the uncertainty of calibration of the weights used

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 37 of 133

will be 1/3 of its specified maximum permissible error. In most cases it will be possible to obtain smaller calibration uncertainties than this, and it may therefore be possible to use a weight of a lower class. However, when selecting suitable weights, attention shall still be given to properties of the weights other than accuracy, such as magnetism, corrosion and wear resistance. In most laboratory applications, it would not be appropriate to select a class lower than M1.

### 3.4.4 Example for Selection of Weights for Balance Calibration:

Capacity of the balance	Calibration weight required	Readability	Required accuracy of weight (Readability/3)	Error as per E2 Class	Error as per F1 class	Selectable class as per class requirement
1000 g	1 kg	0.01g = 10 mg	10/3 = 3 mg	1.6 mg	5mg	1.6 mg

**Note:** Based on the historical data validity of reference weights may be extended upto 5 years for E1.

## 4 Recommended Calibration interval

For the reference weights used in calibration of balance/ comparator at permanent lab or at site facility.

Reference Weights	Recommended Interval (Permanent)	Recommended Interval *(For on-Site)
Weights of E1 class	3 years	2 years
Weights of class E2 to F2	2 years	1 years
Weights of class M1 to M2	1 Year	Year

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

## 4 Calibration Procedure

Performance Check of the balance:

### 4.1 Repeatability Test

This test is carried out at max load capacity and half load capacity of the balance under calibration. Atleast 10 readings for balance upto 10 kg and 5 readings for balance above 10 kg shall be taken and the standard deviation gives the repeatability values. Maximum of the two should be considered for uncertainty calculation.

### 4.2 Linearity Test or Departure of Indication from the Nominal Value

The departure of indication from nominal value or the linearity of the scale is measured at sufficiently equally spaced points over the ranges of the balance to ensure safe interpolation, if needed between these points. Usually minimum 10 such readings are taken including no load and the maximum capacity load.

### 4.3 Eccentricity Test

This test is carried out at a load recommended by the manufacturer of the balance or if it is not known a load between one-third (1/3) and half (1/2) of the maximum capacity of the balance may be used. A single weight should be used for this test.

### 4.4 Hysteresis Test (if the Balance is calibrated first time or after a major repair)

This test is required to be carried out only if the balance is calibrated for the first time or after a major repair. It is carried out with a weight equal to half the capacity of the balance and the reading P1 is noted. Then weight(s) are added to nearer to the maximum capacity of the balance and reading Q1 is noted. Remove all the weights. Repeat these steps 3 more times and note down the values of P2 Q2, P3 Q3 and P4 Q4. Then the average difference between the P values and Q values gives the hysteresis error.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 38 of 133

## 5 Measurement Uncertainty

### 5.1 Uncertainty Components in Balance Calibration ( $u_{ba}$ )

The components of measurement uncertainty to be considered but are not limited to the following:

- Repeatability
- Linearity or departure of indication
- Resolution
- Reference standard weights
- Drift in mass or instability of the mass of weights used
- Eccentricity (whenever the test is carried out)

### 5.2 Uncertainty due to Repeatability

At least six repeated readings shall be taken with a weight near to the maximum capacity of the balance or the range to be calibrated. Estimated standard deviation will be the repeatability contribution.

### 5.3 Uncertainty due to Eccentric Loading

If this contribution is known to be significant, the magnitude must be estimated and if necessary the contribution must be included in the uncertainty budget.

Acceptable solution for the uncertainty due to eccentricity:

$$u_E = [(d_1/d_2) * D] / (2 * \sqrt{3})$$

Where: D is the difference between maximum and minimum values from the eccentricity test performed according to OIML R 76-2;  $d_1$  is the estimated distance between the centers of the weights; and  $d_2$  is the distance from the center of the load receptor to one of the corners.

**Note:** When 10 readings are taken for estimation of repeatability error, the contribution of eccentricity need not be added to the uncertainty.

### 5.4 Uncertainties associated with the Balance Correction Factor

#### a) Uncertainty due to the display resolution of a digital balance

For a digital balance with the scale interval, d, the uncertainty due to resolution is:

$$u_d = (d/2/\sqrt{3})$$

#### b) Uncertainty due to Reference Mass

$$u_s = (U_s/k)$$

Where  $U_s$  is the standard uncertainty of the reference standard weight and k is the coverage factor from its calibration certificate.

#### c) Uncertainty due to Drift in Mass

$$\text{Drift in Mass } u_D = 10\% \text{ of } u_s / \sqrt{3}$$

#### d) Uncertainty due to Repeatability

Standard uncertainty due to Repeatability

$$u_A = s/\sqrt{n-1}$$

### 5.5 Combined Standard Uncertainty of the Weighing Balance

$$u_c = \sqrt{(u_s^2 + u_d^2 + U_D + u_A^2)}$$

Expanded uncertainty  $U = k * u_c$

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 39 of 133



## 5.6 Overall Uncertainty of the Balance

The overall uncertainty of the balance to be reported is the limit of the performance (F) of the balance given by:

$$F = k * SD(\max) + |C_{\max}| + U(C_{\max})$$

Where,

SD(max) = the maximum standard deviation of repeatability at half load and full load

|C<sub>max</sub>| = magnitude of the maximum correction for the balance reading

U(C<sub>max</sub>) = the expanded uncertainty associated with C<sub>max</sub> the correction of the balance

## 5.7 Verification of Comparator

To verify the performance of the comparator same procedure shall be followed to ascertain its performance as per manufacturer specification. Only uncertainty due to standard deviation (from repeatability) is considered during calibration of weights. No other components like eccentricity, error of indication etc are taken into account for a comparator.

## 6. National/ International Standards, References and Guidelines

- OIML R76-1 Metrological and technical requirements – Non-automatic weighing instruments.
- OIML R76-2 Non-automatic weighing instruments – Test report format.
- EURAMET cg – 18 V.03 guidelines on the calibration of Non-automatic weighing instruments.
- OIML-R-111-1 Weights of classes E1, E2, F1, F2, M1, M1-2, M2, M2-3 and M3 metrological and Technical requirements.
- OIML R 47- Standard weights for testing of high capacity weighing machine.
- OIML D28 Conventional value of the result of weighing in air.
- UKAS Guide Lab - 14: Calibration of weighing balance.

**Note: - Latest version of the relevant standard(s) should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 40 of 133

# Chapter 1(D): Volume

## 1 Recommended General Requirements

- 1.1 **Acoustic Noise:** Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 **Illumination:** The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.

## 2 General Requirements - Calibration of Volumetric Apparatus

### 2.1 Scope: Calibration of Volumetric Apparatus

#### 2.1.1 Specific Requirements for Calibration of Volumetric Apparatus

Sl.	Description	Relevant Standard	Permanent Facility	Onsite Calibration	Mobile Facility
1	Burettes (As per IS 1997, ISO 385)	IS/ISO 4787:2010	√	X	X*
2	Single-volume (One mark) pipettes (As per IS 1117, ISO 648)	IS/ISO 4787:2010	√	X	X*
3	Graduated pipettes (As per IS 4162, ISO 835)	IS/ISO 4787:2010	√	X	X*
4	One-mark volumetric flasks (As per IS 915, ISO 1042)	IS/ISO 4787:2010	√	X	X*
5	Graduated measuring cylinders (As per IS 878, ISO 4788)	IS/ISO 4787:2010	√	X	X*
6	Piston Operated Volumetric Apparatus like; a) Single –channel piston pipettes with air interface (as per ISO 8655-2) b) Multi-channel piston pipettes (as per ISO 8655-2) c) Positive –displacement pipettes (as per ISO 8655-2) d) piston burettes (as per ISO 8655-3) e) Diluters (as per ISO 8655-4) f) Dispensers (as per ISO8655-5)	ISO 8655 – 6 & ISO/TR 20461	√	X	X*

**Note 1:** Since the Validity of calibration of a weighing balance will be no more valid if its place is disturbed, because of change in 'g' value, environmental changes and effect of transportation, onsite calibration of volumetric apparatus is not recommended.

**\*Note 2:** In case of mobile calibration, laboratory has to demonstrate competency in respect of weighing balance calibration every time, control of change of temperature and vibration at the place of calibration.

**Note 3:** The above standards are not applicable for medical syringes of the type used for giving injections.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 41 of 133

**Note 4:** This technical requirement is based on the above-mentioned guideline. Lab may follow any relevant standard; however, care shall be taken to follow the requirements in totality.

### 3 Metrological Requirements

#### Volume Measurement by Gravimetric Method:

- Calibration of volumetric instruments recommends the gravimetric method in which mass volume of the distilled water dispensed from a volumetric instrument is measured with a balance and then corrected to a dispensed quantity (volumetric value). No method can measure directly the physical quantity of a minute volume. Therefore, the most common and precise method is to measure the mass value of distilled water, whose physical properties are known using a balance and then convert the mass to a volumetric value.
- Conversion from a mass value to a volumetric value involves the temperature of the distilled water and the barometric pressure as parameters. However, the variation in measured results due to barometric fluctuation is negligible and in practice it will be sufficient to set and use a representative value (fixed value) of the location of measurement. Consequently, the equipment at the time of volume calibration will be balance and the thermometer.
- Uncertainty components in volume calibration by gravimetric method:
  - a) Mass Measurement using Balance:
    - i. Performance of the balance,
    - ii. Error due to measurement method
    - iii. Evaporation of the distilled water during weighing
  - b) Mass to Volume Conversion: The density of the distilled water is approximately 1g/ ml but varies with change in temperature of water.
- Meanwhile, in order to measure the mass of an object, it is necessary to correct for buoyancy, since the balance is calibrated using a weight (density 8000 kg/m<sup>3</sup>). Therefore, these factors (density variation of distilled water due to temperature change and correction for buoyancy) shall be considered when converting the measured mass value of the distilled water to a volumetric value.
- Uncertainty concerning the mass to volume conversion values relates to the uncertainty of water temperature and the barometric pressure measurements.
- Proficiency of Operator: Dispensed volume of volumetric apparatus is known to be influenced by operator skill. Therefore dispensed volume depends on both on the performance of the volumetric apparatus itself and the level of operator skill. Operator skill is a significant uncertainty component in volume calibration
- **Uncertainty based on the Performance of the Balance:**  
Repeatability, linearity, resolution and sensitivity drift are performance factors of the balance that affect the volume measurement. It is presumed that, the balance is properly calibrated at the time of volume measurement.
- **Uncertainty based on Evaporation:**  
Once distilled water is dispensed from a volumetric apparatus such as a pipette into the small cup et on the balance, a certain amount of evaporation takes place before the mass of the water is determined by the balance. This evaporation amount is a component of uncertainty
- **Uncertainty based on the Temperature (Water Temperature) and Barometric Pressure:**  
The density variation of distilled water is approximately 0.02% per °C between 15°C and 30°C. Therefore, when the error of temperature measurement of distilled water is 1°C, it will be an error of 0.02% after conversion into volume.

The influence of barometric pressure on the conversion to volume will be as minute as 0.01hPa per pressure change of 100 hPa between 850 hPa to 1050 hPa. The pressure change at a location is normally  $\pm 15$  hPa. Even though an average (fixed value) of pressure  $\pm 30$  hPa is used, whose influence

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 42 of 133

on the conversion to volume is within 0.003%. The rate of influence of water temperature and barometric pressure on the mass to volume conversion can be readily in the Z factor table.

#### 4 Selection of Reference Apparatus and Reference Liquid for Volumetric Calibration

##### 4.1 Balance

The balance used as reference for calibration shall have traceability certificate and meet the requirements mentioned below:

4.1.1 It is recommended that balance used for calibration of volumetric apparatus shall have a readability / resolution of the order of 1/3<sup>rd</sup> of accuracy specified for volumetric apparatus.

4.1.2 Recommended weighing balances for calibration of volumetric apparatus as per IS/ISO 4787:2010.

Selected volume under test <sup>a</sup> V	Resolution mg	Standard deviation (repeatability) mg	Linearity mg
100 µl < V ≤ 10 ml	0.1	0.2	0.2
10 ml < V < 1 000 ml	1	1	2
1 000 ml < V < 2000 ml	10	10	20
V > 2000 ml	100	100	200

<sup>a</sup> - For practical purposes, the nominal volume may be used to choose the balance

4.1.3 Minimum requirements of balances for micro pipette calibration as per ISO 8655-6.

Selected volume <sup>a</sup> of apparatus under test V	Resolution mg	Repeatability and linearity mg	Standard uncertainty of measurement mg
1 µl ≤ V ≤ 10 µl	0.001	0.002	0.002
10 µl < V ≤ 100 µl	0.01	0.02	0.02
100 µl < V ≤ 1000 µl	0.1	0.2	0.2
1 ml < V ≤ 10 ml	0.1	0.2	0.2
10 ml < V ≤ 200 ml	1	2	2

<sup>a</sup> For practical purposes, the nominal volume may be used to choose the balance.

#### 5 Recommended Calibration Interval

Reference Weights	Recommended Interval	Reference Balance	Recommended Interval
Weights of E1 class	3 years	Electronic weighing balance	1 Year
Weights of class E2 to F2	2 years		
Weights of class M1	1 Year		

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

#### 6 Calibration Methods

Calibration of volumetric apparatus can be done either of the following two methods:

- Gravimetric Method
- Volumetric Method

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 43 of 133

## 5.1 Calibration Procedure (Based on Gravimetric Method)

5.1.1 Based on IS/ISO 4787 for Laboratory glass ware - volumetric instruments. This is applicable for calibration of volumetric instruments made from glass for the range above 0.1ml to 10000 ml, in order to obtain the best accuracy in use.

5.1.2 Based on ISO 8655-6 for piston operated volumetric apparatus:

This is applicable for  $\leq 1\mu\text{l}$  to 200 ml volumetric apparatus

For calibration procedure of different types of apparatus, follow the clause references of the above Standard as given below:

- a) Single –channel piston pipettes with air interface (as per ISO 8655-2) follow clause 7.2 of the above standard.
- b) Multi-channel piston pipettes (as per ISO 8655-2) follow clause 7.3 of the above standard.
- c) Positive –displacement pipettes (as per ISO 8655-2) follow clause 7.4 of the above standard.
- d) Piston burettes (as per ISO 8655-3) follow clause 7.5 of the above standard.
- e) Diluters (as per ISO 8655-4) follow clause 7.6 of the above standard.
- f) Dispensers (as per ISO8655-5) follow clause 7.6 of the above standard.

5.1.3 The standard reference temperature is the temperature at which the volumetric instrument is intended to contain or deliver its volume (capacity). Shall be 27°C (tropical countries like India).

When the volumetric instrument is required for use in a country which has adopted a standard reference temperature of 20°C, this figure shall be substituted for 27°C.

## 6 Equation for Calculation of Volume

### 6.1 Method -I

#### Calculation of Volume as per IS/ISO 4787:2010

The general equation for calculation of the volume at the reference temperature of 27°C,  $V_{27}$ , from the apparent mass of the water, contained or delivered, is as follows:

$$V_{27} = (I_L - I_E) \times (\rho_w - \rho_A)^{-1} \times (1 - \rho_A/\rho_B) \times [1 - \gamma(t-27)]$$

where

$I_L$  - is the balance reading of vessel with water, in grams;

$I_E$  - is the balance reading of empty vessel, in grams (zero in case the balance was tared with the volumetric instrument or receiving vessel);

$\rho_A$  - is the density of air, in g/ml, obtained from Table at the temperature and atmospheric pressure of the test;

$\rho_B$  - is either the actual density of the balance weights when these are adjusted to their nominal mass, or the reference density for which the weights have been adjusted (see the note below), in g/ml, or, when using an electronic balance without weights, the (reference) density of the weights with which it has been adjusted;

$\rho_w$  - is the density of water at  $t$  °C, in grams per milliliter calculated with the "Tanaka" formula (See table B-4 of IS/ISO 4787:2010).

$\gamma$  - is the coefficient of cubical thermal expansion of the material of which the volumetric instrument tested is made, in per °C (see Table below);

$t$  - is the temperature of the water used in testing, in degrees Celsius.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 44 of 133

### 6.1.1 Coefficient of Cubical Thermal Expansion '□'

Material	Coefficient of cubical thermal expansion, □ <sup>a</sup> (x10-6/ °C)
Borosilicate glass 3.3	9.9
Borosilicate glass 5.0	15
Soda-lime G lass	27

**Note:** Weights conforming to International Document OIML D 28 of the International Organization of Legal Calibration have been adjusted to give correct results when weighing in air as though the density of the weights were 8.0 g/ml. Electronic balances are usually adjusted by means of these weights.

6.1.2 In order to give an impression of the extent to which the various parameters influence the result, some parametric tolerances, with the corresponding error in the volume determined, are given in Table below. It is evident from these figures that the measurement of the water temperature is the most critical factor.

Parameter	Parametric Tolerance	Volumetric Error relative to the Volume <sup>a</sup>
Water temperature	± 0.5 °C	± 10 <sup>-4</sup>
Air pressure	± 8 mbar (0.8 kPa)	± 10 <sup>-5</sup>
Air temperature	± 2.5 °C	± 10 <sup>-5</sup>
Relative humidity	± 10 %	± 10 <sup>-6</sup>
Density of weights	± 0.6 g/ml	± 10 <sup>-5</sup>

<sup>a</sup> example : a relative volumetric error of 10<sup>-4</sup> to the measured volume of 100 ml would be 0.01 ml

6.1.3 When the temperature at which the volumetric instrument is used (t<sub>2</sub>) differs from the reference temperature (t<sub>1</sub>), the volume of the volumetric instrument at (t<sub>2</sub>) can be calculated from the following equation:

$$V_2 = V_{t1} [1 + \gamma (t_2 - t_1)]$$

Where  $\gamma$  is the coefficient of cubical thermal expansion (see 4.11.2). For information on the effect of temperature differences.

### 6.2 Method –II

[This can be used for both calibration procedures either IS/ISO 4787:2010 or ISO 8655-6 for calculation of volume]

### 6.3 Method-III

[Applicable for calibration as per IS/ISO 4787:2010 using calibrated standard weight as reference]

## 7 Measurement Uncertainty

The components of uncertainty

Uncertainty in Measurement repeatability (type A)

Uncertainty of the balance including linearity\*

Uncertainty in water temperature measurement

Uncertainty Water Density

Uncertainty Air density

Uncertainty Coefficient of expansion of the material of the volumetric apparatus

Uncertainty due to reference mass and its drift\*

Uncertainty of density of the reference mass\*

Uncertainty due to meniscus

Uncertainty due to evaporation (below 50 μl)

**Note 1:** Linearity of the balance is excluded when reference standard weights are used in calibration.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 45 of 133

**Note 2\*:** If calibrated weights are used during calibration as reference equivalent to the volume of the test apparatus along with the balance, and only the difference is measured - linearity of the balance can be ignored and uncertainty components of reference weight, its density and drift are to be taken into account.

**Note 3:** Uncertainty due to meniscus may be omitted if 10 readings are taken as it reflects in repeatability.

## 8 Recommended References: National/ International Standards, References and Guidelines

- OIML R111-1 Metrological and technical requirement of weights Classes E1, E2, F1, F2, M1, M2, M3.
- OIML D28: Conventional value of the result of weighing in air.
- OIML R76-1 Metrological and technical requirements –Non-automatic weighing instruments.
- OIML R76-2 Non-automatic weighing instruments -Test Report format.
- EURAMET cg-18 V.03 Non-automatic weighing instruments.
- IS/ISO 4787, Laboratory glassware - Volumetric instruments - Methods for testing of capacity and for use.
- IS 1997 / ISO 385, Laboratory glassware — Burettes.
- ISO 648, Laboratory glassware — Single-volume pipettes.
- ISO 835, Laboratory glassware — Graduated pipettes.
- ISO 1042, Laboratory glassware — One-mark volumetric flasks.
- ISO 3696, Water for analytical laboratory use — Specification and test methods.
- IS 878 / ISO 4788, Laboratory glassware — Graduated measuring cylinders.
- ISO/IEC Guide 99, International vocabulary of Calibration — Basic and general concepts and associated terms (new edition) (to be deleted).
- IS 1070 Reagent Grade water – Specification.
- ISO 3696, Water for analytical laboratory use – Specification and test methods.
- ISO/TR 20461: Determination of uncertainty for volume measurements made using the gravimetric method.
- IS 1117: One-mark pipettes.
- IS 915: One-Mark Volumetric Flasks – Specification.
- IS 4162: Specification for Graduated Pipettes.
- ISO 8655-6: Piston-operated volumetric apparatus - Part 6: Gravimetric methods for the determination of measurement error.

**Note:** - Latest version of the relevant standard(s) should be followed

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 46 of 133

# Chapter 1(E): Density and Viscosity Measurement

## I) Density of Solids and Viscosity Measurement

### 1 Recommended General Requirements

- 1.1 Acoustic Noise: Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 Illumination: The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.
- 1.6 Viscometers are calibrated under normal stable room temperature conditions. It is desirable to have a room temperature within 20 °C to and 30 °C with variation of  $\pm 4$  °C.
- 1.7 The ambient temperature shall be (18 °C to 27 °C)  $\pm 2$  °C.

### 2 Recommended Requirements

#### 2.1 Scope: Measurement of Density of Solids

##### Specific Requirements for Calibration

Sl.	Description	Relevant Standard	Permanent facility	Onsite calibration	Mobile facility
1	Density of Solids	OIML G14	√	X	X
2	Density of standard weights	OIML R111-1	√	X	X

**Important Note:** This technical requirement is based on above referred standard taking into account only the salient features required during calibration. Lab may follow any relevant standard, however care shall be taken to follow the requirements in totality.

### 3 Metrological Requirements

For Density measurement of Standard weights follow the requirements of OIML R-111-1 and density of solids.

### 4 Selection of Reference for Calibration Density of Solids

Analytical Balance with density kit, Tripled distilled water, Beaker.

### 5 Legal Aspects

Calibration of density of solids done by any accredited laboratories is meant for scientific and industrial purpose only. However, if used for commercial trading, additional recognition/ approval shall be complied as required by Dept. of Legal metrology, Regulatory bodies, etc.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 47 of 133



## 6 Calibration Procedures

### 6.1 Determination of Density by Hydrostatic Weighing Method:

6.1.1 The density of a solid is determined with the aid of a liquid whose density  $\rho_L$  is known (water or ethanol are usually used as auxiliary liquids). The solid is weighed in air (A) and then in auxiliary liquid (B), the density  $\rho$  can be calculated from the two weighings as follows:

6.1.2 To find the density of the solid

$$\rho = \frac{A}{(A-B)} * (\rho_L - \rho_a) + \rho_a$$

6.1.3 To find the volume of the solid

$$V = (1 - \rho_a / \rho_m) * \frac{(A-B)}{(\rho_L - \rho_a)}$$

Where,  $\rho$  = Density of the sample  
A = Weight of the sample in air  
B = Weight of the sample in liquid  
V = Volume of the Sample  
 $\rho_L$  = Density of the auxiliary liquid  
 $\rho_a$  = Density of air  
 $\rho_m$  = Density of mass of the solid

### 6.2 Determination of Density of Solid matter using Pycnometer:

Pycnometer can also be used to determine the density of homogeneous solid object that does not dissolve in working liquid (distilled water). Pycnometer is a glass measure having a fixed volume V.

First, the solid matter =  $m_s$

Now, weigh the pycnometer with the solid matter inserted =  $m_1$

Then, add distilled water and weigh the pycnometer along with the solid matter and the water =  $m_2$

Now, weight of the water,  $m_w = m_2 - m_1$

Volume of the water  $V_w = m_w / \rho_w$

Volume of the solid matter  $V_s$  is the difference between the water that fills the empty pycnometer and the volume  $V_w$  calculated as above,  $V_s = V - V_w$

Density of the solid matter can be calculated as,

$$\rho_s = \frac{m_s}{V_s}$$

### 6.3 Measurement Uncertainty

Uncertainty contributions for weighing process

- Balance – Repeatability, resolution, linearity
- Reference standard weight
- Density of reference liquid- density, surface tension and temperature
- Air density

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 48 of 133

## II) Density and Viscosity Measurement of Liquids

### 1 Scope: Determination of Density of Liquids and Calibration of Hydrometers

#### 1.1 Recommended Requirements for Calibration

Sl. No	Types of Hydrometers	Relevant Standard	Permanent facility	Onsite calibration	Mobile facility
1	Density of Liquids	OIML G14 2011	√	X	X
2	Density Hydrometers	IS 3104-1982 (part 2) (RA – 2008)	√	X	X
3	Brix Hydrometers	IS 7324-1983 RA 2006	√	X	X
4	Baume Hydrometers	IS 1255-1988 RA1998	√	X	X
5	Twaddle Hydrometer	Comparison method or any relevant standard or guidelines	√	X	X
6	Specific gravity Hydrometer (for liquid petroleum)	IS 1448(P:76) 1968 RA 2003	√	X	X
7	Lactometer	IS 9585 – 1980 (RA -2003)	√	X	X
8	Alcoholometer	IS 3608 -1987 Part -1 without thermometer & Part- 2 with thermometer	√	X	X

**Note 1:** This technical requirement is based on above referred standard taking into account only the salient features required during calibration. Lab may follow any relevant standard; however care shall be taken to follow the requirements in totality.

### 2 Metrological Requirements

i. Requirement stipulated in the relevant standards needs to be followed by the laboratory.

ii. The general classifications of Density Hydrometer are as follows:

**L20:** long scale length, range 20 kg/m<sup>3</sup>, the density interval 0.2 kg/ m<sup>3</sup> with 100 graduations

**L50:** long scale length, range 50 kg/m<sup>3</sup>, the density interval 0.5 kg/ m<sup>3</sup> with 100 graduations

**M50:** Medium scale length, range 50 kg/m<sup>3</sup>, the density interval 1 kg/ m<sup>3</sup> with 50 graduations

**M100:** Medium scale length, range 100 kg/m<sup>3</sup>, the density interval 2 kg/ m<sup>3</sup> with 50 graduations

**S50:** Short scale length, range 50 kg/m<sup>3</sup>, the density interval 2 kg/ m<sup>3</sup> with 25 graduations

**S50SP:** Short scale length, range 50 kg/m<sup>3</sup>, the density interval 1 kg/ m<sup>3</sup> with 50 graduations

### 3 Calibration Methods

#### 3.1.1 There are two methods for determination of density of liquids

- Using pycnometer (specific gravity bottle)
- Using a hydrometer by comparison

#### 3.1.2 There are two methods for calibration of hydrometers

- Hydrostatic weighing (Cuckow's) method
- Comparison method based on Archimedes principle

Hydrometer is calibrated using the method of comparison, in which it is compared with a standard hydrometer whose scale is precisely known.

#### 3.1.3 Density of Liquids using Sinker of known volume

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 49 of 133

The density of a liquid is determined using a sinker of known volume. The sinker is weighed in air and then in the liquid whose density is to be determined. The density  $\rho$  can be determined from the two weighing as follows:

$$\rho = \alpha * \frac{(A-B)}{V} + \rho_a$$

Where,  $\rho$  = Density of the liquid

A = Weight of the sinker in air

B = Weight of the sample in liquid

V = Volume of the Sinker

$\rho_L$  = Density of the auxiliary liquid

$\rho_a$  = Density of air

$\alpha$  = Air buoyancy correction

### 3.1.4 Density of Liquids using a Pycnometer

Pycnometer is a glass measure having a fixed volume. It is closed by a stopper in which there is a small hole which enables, air and excess liquid to eliminate. The capacity of the pycnometer is normally 50 ml or 100 ml.

Density at temperature  $t$  can be calculated as,

$$\rho_{tL} = \frac{(m_L + C) * \rho_{tw}}{(m_w + C)}$$

Where,

$m_L$  = mass of the liquid sample at temperature  $t$  °C

$m_w$  = mass of the distilled water at temperature  $t$  °C

$\rho_{tw}$  = Density of water at temperature  $t$  °C (from the table)

C = Buoyancy correction =  $\rho_{ta} \times m_w$

$\rho_{ta}$  = Air density

### 3.1.5 Calibration of Hydrometers

#### By Cuckow's Method

The Cuckow's method is based on the three equilibrium equations obtained in different conditions and situations:

In the case the hydrometer floats free in a liquid of  $\rho_x$  density at reference temperature of  $T_0$ , the equilibrium equation is:

$$m * g + \pi D \gamma_x = g * V * \rho_x + g * v * \rho_{a1} \quad (1)$$

Where,

m - mass of the hydrometer,

g - local acceleration due to gravity

D - diameter of stem of hydrometer

$\rho_{a1}$  – density of air at the moment of reading hydrometer's indications

$\gamma_x$  – Surface tension of the liquid

V – volume of the hydrometer ( the part immersed in liquid)

v – volume of the stem of hydrometer above the surface of the liquid

- i) When a hydrometer is weighed in air using standard mass, two forces act upon it: the weight of hydrometer in air and the Archimedes' force - due to the air replaced by the volume of the hydrometer. The equilibrium equation is:

$$M_a \times g ( 1 - \rho_{a2}/ \rho_w ) = m \times g - g*(V+v)* [1+\beta(T_2-T_0)] \rho_{a2} \quad (2)$$

Where,  $M_a$  - mass of the hydrometer in air

$\rho_w$  - Density of standard weight

$\beta$  - coefficient of cubic expansion of the glass used to construct the hydrometer

$T_2$  - air temperature during the weighing

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 50 of 133

$\rho_{a2}$  - density of air during weighing

Neglecting  $v\beta(T_2-T_0)$  from equation (2) can be written as

$$M_a \times (1 - \rho_{a2} / \rho_w) \cong m - V \rho_{a2} [1 + \beta(T_2 - T_0)] - v \rho_{a2} \quad (3)$$

- ii) The Hydrometer is weighed being partially immersed in a reference liquid of density  $\rho_L$ , at the same mark as in the case (i) at the temperature  $T_3$  (thus the volume remains unchanged). Two types of forces against it: the weight of the hydrometer and the force due to surface tension  $\gamma_L$  of the liquid (climbing on the stem of the hydrometer); the two forces are equilibrated by the Archimedes' force (or buoyancy) of the liquid (displaced by the volume  $V$  of hydrometer under the surface of liquid) and of the air (displaced by the volume  $v$  of the stem of hydrometer above the surface of liquid) The equilibrium equation is:

$$M_L \times g (1 - \rho_{a3} / \rho_w) = m g + \pi D \gamma_L - g^* V \rho_L [1 + \beta(T_3 - T_0)] - g^* v \rho_{a3} [1 + \beta(T_3 - T_0)] \quad (4)$$

Where,

$M_L$  – is the mass of hydrometer in the reference liquid

$\rho_{a3}$  - is density of air during the hydrostatic weighing

$g^*$  - gravity acceleration at the liquid level

Since the contribution of gravity acceleration gradient is negligible,  $g^* \cong g$ , the equation (4) becomes.

$$M_L (1 - \rho_{a3} / \rho_w) = m + \pi D \gamma_L / g - V \rho_L [1 + \beta(T_3 - T_0)] - v \rho_{a3} \quad (5)$$

### 3.1.6 Calibration of Hydrometer by Comparison Method

- 3.1.6.1** The hydrometer is calibrated using comparison method in which the hydrometer under calibration is compared with reference to a standard hydrometer whose scale is precisely known.

Suppose  $D$  is the diameter of the stem of a hydrometer,  $V$  the volume of its bulb upto the mark to which the hydrometer is floating in a liquid of density  $\rho_L$ ,  $v$  the volume of the stem exposed to air and  $T$  is the surface tension of the liquid, then forces acting on the freely floating hydrometer will be:

Downward forces

- Gravitational force due to its mass =  $m * g$
- Force due to the surface tension of the liquid  $\pi DT$

Upward forces

- Buoyant force due to the liquid displaced by the volume  $V$  of the hydrometer below the liquid surface =  $g * V * \rho_L$
- The buoyant force of the air displaced by the volume  $v$  of the hydrometer stem above the liquid surface =  $g * v * \rho_a$
- Up thrust on the volume of the liquid raised by the surface tension =  $\pi D \rho_a T / \rho_L$

Under equilibrium, Downward Force = Upward force

$$m * g + \pi DT = g * V * \rho_L + g * v * \rho_a + \pi D \rho_a T / \rho_L$$

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 51 of 133

As the volume, air density, liquid density and surface tension are temperature dependant quantities, the hydrometer reading will be correct only at a particular temperature. Therefore, every hydrometer should bear its reference temperature and its surface tension categories.

The hydrometers are calibrated under normal room temperature conditions. Appropriate corrections are applied for hydrometers graduated for density or relative density at reference temperatures. Generally these reference temperatures are 20°C, 15°C, 15.5°C or that desired by the user. Hence correction is applied

### 3.1.7 Classification of Density Hydrometers:

Generally the range of hydrometers is (20 to 50) kg/m<sup>3</sup>, but commercial density hydrometers are available in the ranges of (100, 200 or 1000) kg/m<sup>3</sup>.

- Density hydrometers are classified according to:
  1. Width of the scale i.e. value of density between two graduations
  2. Range
  3. Maximum permissible errors as per the standard IS 3104
- For more details of procedure for calibration refer IS 3104.
- For calibration of Alcoholometer follow IS 3608 -1987 Part -1 without thermometer & Part- 2 with thermometer.

## 3.2 Measurement Uncertainty

### 3.2.1 Cuckow`s method: contributions of uncertainty are due to:

- Process and procedure contributions(Type A)
- Balance – Repeatability, resolution, linearity
- Reference standard weight
- Density of reference liquid- density, surface tension and temperature
- Hydrometer – reference temperature, reference surface tension, width of scale interval, Thermal expansion coefficient, Diameter of the stem
- Environment -Air density, Local gravity

### 3.2.2 Comparison method: Contributions of Uncertainty are due to:

- Calibration process – Type A
- Reference standard hydrometer
- Temperature correction
- Surface tension correction
- Meniscus correction

### 3.2.3 Cuckow`s Method: contributions of uncertainty for CMC are due to:

- Process and procedure contributions (Type A - 10 repeated readings at minimum and maximum).
- Balance – Repeatability, resolution, linearity.
- Reference standard weight.
- Density of reference liquid- density, surface tension and temperature.
- Environment -Air density, Local gravity.

### 3.2.4 Comparison Method: Contributions of uncertainty are due to:

- Calibration process – (Type A - 10 repeated readings at minimum and maximum)
- Reference standard hydrometer

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 52 of 133

- Temperature correction
- Surface tension correction
- Meniscus correction

#### 4 Recommended Calibration Interval

Reference Equipment	Recommended Interval
Hydrometer	1 Year
known density liquids (Standard Reference Material)	As per Manufacturer specification
Analytical balance	1 year
Standard weights	3 years

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

#### 5 Key Points

- 5.1 Demonstration of any CMC values doesn't automatically qualify for granting accreditation until the lab satisfies the stipulated requirement given above.
- 5.2 Laboratory shall demonstrate at least the lower grade of hydrometer.

#### 6 Measurement of Viscosity and Calibration of Viscometers

**Scope: Determination of Viscosity and Calibration of Viscometers**

**Specific Requirements for Calibration**

Sl. No	Description	Relevant Standard	Permanent facility	Onsite calibration	Mobile facility
1	Calibration of Master Viscometer	ASTM D2162-13,	√	X	X
2	Determination of kinematic and Dynamic Viscosity of liquids	ASTM D1480, ASTM D1250	√	X	X
3	Calibration of Glass capillary Viscometers (Direct flow & Reverse flow)	ASTM D446 – 12 alternate Standard ISO 3105:1994	√	X	X
4	Calibration of Rotational Viscometer (Brookfield)	ISO 2555 :1989 ISO1652:2011	√	X	X
5	Calibration of flow Cups for determining efflux time –Paints, varnishes ,lacquers and other viscous fluids	IS:3944 1982,RA 2005 Appendix B	√	X	X

**Note 1:** This technical requirement is based on above referred standard taking into account only the salient features required during calibration. *Lab may follow any relevant standard; however care shall be taken to follow the requirements in totality.*

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 53 of 133

## 7 Metrological Requirements

- The most important factor affecting the quality of a viscosity measurement is temperature.
- Temperature control is the most important parameter for obtaining accurate and precise viscosity measurement. A slight variation in temperature can have a large effect on the viscosity of fluid.
- One of the most obvious factors that can have an effect on the rheological behavior of a material is temperature. Some materials are quite sensitive to temperature and a relatively small variation will result in a significant change in viscosity.
- As the temperature of a fluid increases its viscosity decreases. In the liquids the cohesive forces between the molecules predominates the molecular momentum transfer between the molecules.
- 'g' value shall be known with sufficient accuracy either by Geological Survey of India or any other relevant source for reporting the g value along with viscosity constant in the calibration certificate. This helps the user to apply required correction due to change in g for viscosity constant.
- Laboratory may also calculate 'g' value knowing latitude and height as per the formula. However, same shall be validated. (as given at 4.3.8.2.1 & 4.3.8.2.2 below).

## 8 Recommended Selection of Reference Equipment

### 8.1 For Calibration of Glass Capillary Viscometers

- Standard or reference viscometers of the Ubbelohde, Cannon (NPL), U-tube (NRLM) etc., types, the constants of which is known with the error not exceeding  $\pm 0.01\%$ .
- Reference materials of viscosity: transparent Newtonian liquids, of stable viscosity (silicone liquids are not recommended).
- Thermostatic baths, with temperature control devices ensuring a constant temperature during measurement, the variations not exceeding  $\pm 0.01^\circ\text{C}$ .
- Devices for measurement of flow time of the liquid in a standard or reference viscometer with an error not exceeding  $\pm 0.01\text{s}$  and in a viscometer to be verified with an error not exceeding  $\pm 0.2\text{s}$ .
- Thermometers for the measurement of the temperature in the thermostatic bath with an error not exceeding  $\pm 0.01^\circ\text{C}$ .
- Water jet pump, or other type of suction pump.
- Desiccator.
- Laboratory Glassware (beakers, flasks, funnels, stirrers, etc).
- Liquids for washing the viscometers: distilled water, chromic acid, white spirit, rectified ethyl alcohol, acetone and other solvents.
- Small diameter rubber or plastic tubes.
- Lighting fixtures with negligible thermal radiation.

### 8.2 Effect of Gravity "g" on Calibration

- It is very important to establish the gravitational value of the laboratory, as the same has to be mentioned in the calibration certificate of the Master viscometer to enable the calibration laboratory to apply correction due to 'g' at his laboratory.
- Validation of local 'g' and its Uncertainty.

#### 8.2.1 Formula for calculation of acceleration due to gravity

An approximate value for g, at given latitude and height above sea level, may be calculated from the formula:

$$g = 9.780 \quad 7 (1 + A \sin^2 L - B \sin^2 2L) - 3.086 \times 10^{-6} H m \cdot s^{-2}$$

Where,  $A = 0.005 \ 302 \ 4$ ,  $B = 0.000 \ 005 \ 8$ ,  $L$  = latitude,  $H$  = height in meter above sea level.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 54 of 133

8.2.2 To validate this calculated 'g' value the simple steps given below can be followed:

- Find out the actual 'g' value of NMI from the certificate issued by them or by any other source.
- From the google maps click on the location of NMI, find out latitude and height above sea level. (You can know the 'g' value).
- Calculate the 'g' value using the above formula with these latitude and height. The difference between the calculated value of 'g' and the actual value of the NMI should be within 20 to 30 ppm.
- Now, go to the google maps and click on location of the lab and find out the latitude and height of the place as per google (you can know the 'g' value also).
- Calculate the 'g' value for this latitude and height. The value obtained should be within 20 to 30 ppm.
- Then this value can be taken as 'g' value of the lab and uncertainty of 'g' can be assumed to be within  $\pm 50$  ppm.

## 9 Recommended Calibration Interval

Reference Equipment	Recommended Interval
Analytical Balance	1 Year
Thermometer	1 year

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations

## 10 Calibration Procedures

10.1 Please refer Annexure A-1 of ASTM D446-12 Or Annexure A of ISO 3105 for details

### C.1 Reverse Flow Viscometer

For more details, construction, operation and calibration set up of above type of viscometers refer Annexure A-3 of ASTM D446-12 Or Annexure C of ISO 3105 Or ISO 3104.

### D.1 Flow cups- as per IS 3944, RA 2005 Or ASTM D 1200

For calibration follow IS 3944 "Method for Determination of Flow Time for Flow Cups" Or ASTM D1200.

### E.1 Rotational Viscometers –as per ISO 2555 1989 & ISO 1652

#### Reference Viscometers

- Please refer annexes of the standards ASTM D446 or ISO 3104 or ISO3105.for more details.

## 11 Master Viscometer Calibration

### 11.1 Selection of Reference Equipment

11.1.1 Two or more master viscometers (Cannon or Ubbelohde type) having calibrating constants in the 0.001 to 0.003 mm<sup>2</sup>/s duly calibrated with water at 20°C.

- Kinematic viscosities of 2 or more oil standards are measured at 40°C in this master Viscometers (Cannon or Ubbelohde type).
- A third master viscometer(Cannon or Ubbelohde type), with a calibration constant of 0.003 to 0.009 mm<sup>2</sup>/s<sup>2</sup> duly calibrated at 40°C with two standard oils and its calibration factor calculated at standard conditions for water at 20°C.

11.1.2 Digital Contact Thermometer to meet or exceed following requirements:

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 55 of 133



- Only acceptable sensors are Resistance Temperature Devices (RTD) or high precision thermistors.
- Standard Platinum Resistance Thermometer (SPRT) is also preferred.
- Resolution of 0.001°C with a combined (Display + probe) accuracy of 0.007°C at k=2, response time < 25 s.
- Linearity of < 0.007°C over the temperature to be measured.
- The reference standard viscometer should be so chosen that its flow time for the reference liquid should not be less than 200 second.

11.1.3 **Liquid-in-thermometer:** Kinetic viscosity thermometers having a range 18.5°C to 21.5°C and 38.5 to 41.5°C calibrated to 0.005°C accuracy.

11.1.4 **Thermostat Bath:** A thermostat bath containing water or other transparent liquid deep enough to immerse the master viscometers, so that the upper fiducial mark is at least 50mm below the surface. The efficiency of stirring and the balance between heat loss and input must be such that, the temperature of water does not vary more than ± 0.01°C over the length of the viscometer or from one viscometer position to another. A standard platinum resistance thermometer, approximately 450 mm in length may be used to ensure that the variation in temperature does not exceed ± 0.01°C. Firm support should be provided to hold the master viscometer in a rigid and reproducible within ± 15 min from vertical 0 degree position.

11.1.5 **Timer:** Digital timer capable of measuring 300 to 10000 s with an accuracy of ± 0.03s. A spring wound stop watch may also be used.

## 12 Calibration Procedure

Please refer ASTM D 2162-13

### 12.1 Calibration of Viscosity Oil standards

Please refer ISO 3104

- Calibration of viscosity oil standards at 40°C.
- Follow Clause 9.1 to 9.7 to measure efflux time.
- Correction and calculation of kinematic viscosity at 40°C.
- Buoyancy correction needs to be applied (refer 10.1 of ASTM D2162).
- Temperature correction may be needed due to thermal expansion of glass capillary tube viscometers (refer 10.2.1 of ASTM D2162).
- Determine the density of oil standard at 40°C in accordance with test method ASTM D1480.
- Determine the relative density of the oil at some convenient temperature and obtain the relative density at 40°C in accordance with Guide ASTM D1250.
- Surface tension correction may be necessary for cannon type viscometer. If required, determine the surface tension of the oil standard at 40°C in accordance with test method ASTM D1590.

## 13 Recommended References: National/ International Standards, References And Guidelines

- OIML G 14 Density measurement.
- OIML R111-1 Metrological and technical requirement of weights classes E1, E2, F1, F2, M1, M2, M3.
- OIML R111-2 Weights Classes E1, E2, F1, F2, M1, M2, M3- Test report format.
- OIML R76-1 Metrological and technical requirements – Non automatic weighing instruments.
- OIML R76-2 Non automatic weighing instruments – Test report format.
- ASTM D 2162-13 “Standard practice for basic calibration of Master Viscometers and Viscosity Oil Standards”

## 14 Referenced Documents for ASTM D 2162-13

- ASTM D445 “Test Method for Kinematic Viscosity of Transparent and Opaque Liquids” (and calculation of Dynamic Viscosity)
- ASTM D 446 “Specifications and Operating instructions of Glass Kinematic Viscometers”
- ASTM D1193 “Specification for Reagent Water”

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 56 of 133

- ASTM D1250 “Guide for use of the Petroleum Measurement Tables”
- ASTM D1480 “Test Method for Density and Relative Density (Specific gravity) of Viscous Materials by Birmingham Pycnometer”
- ASTM D1590 “Test Method for Surface Tension of Water”
- ASTM E1 “Specification for ASTM Liquid-in Glass Thermometers”
- ASTM E563 “Practice for Preparation and Use of an Ice-point Bath as a Reference Temperature”
- ASTM E644 “Test Methods for Testing Industrial Resistance Thermometers”
- ASTM 1750 “Guide to use of Water Triple Point Cells”
- ASTM E2593 “Guide for Accuracy Verification of Industrial Platinum Resistance Thermometers”
- ASTM 2877 “Guide for Digital Contact Thermometers”
- ISO 3666 “Viscosity of Water”

#### 15 Referenced Documents for ASTM D446-12

- ASTM D2162-13 “Standard Practice for basic Calibration of Master Viscometers and Viscosity Oil Standards”
- ISO 3104 “Petroleum Products – Transparent and Opaque Liquids- Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity”
- ISO 3105 “Glass Capillary Kinematic Viscometers –Specifications and Operating Instructions”
- ISO 5725 “Basic Methods for the Determination of Repeatability and Reproducibility of a Standard Measurement Method”
- IS 3944 “Method for Determination of Flow Time for Flow Cups”
- ISO 2555 “Plastics –Resins in the Liquid State or as Emulsions or Dispersions -Determination of Apparent Viscosity by the Brookfield Test Method”
- ISO1652 “Rubber Latex- Determination of Apparent Viscosity by Brookfield Test Method”
- OIML R 69 “Glass Capillary Viscometers for the Measurement of Kinematic Viscosity-Verification Method”
- OIML D 17 Hierarchy Scheme for Instruments Measuring the Viscosity of Liquids.

**Note: - Latest version of the relevant standard(s) should be followed**

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129		Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)		
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 57 of 133

## Chapter 1(F): Calibration of Force Proving Instruments

### 1 Recommended General Requirement

- 1.1 **Acoustic Noise:** Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 **Illumination:** The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.
- 1.6 The temperature shall be maintained in the range of 18°C to 28°C. The temperature shall not vary more than  $\pm 1^\circ\text{C}$  throughout a measurement series.

### 2 Recommended Specific Requirements- Calibration of Force Proving Instruments

**Scope:** Calibration of Force proving Instruments used for verification of Uniaxial Testing Machines

Sl. No.	Description	Relevant Standard/ Guidelines	Permanent facility	On-site calibration
1	Load Cells	IS 4169 /	√	X
2	Proving Rings	ISO 376	√	X

Specific requirements for the calibration of Force proving Instruments

**Note 1:** Force proving instruments can also be calibrated as per ASTM E74-06 which is used for calibration of Uniaxial Testing Machines as per ASTM E4-10.

**Note-2:** This technical requirement is based on IS 4169 / ISO 376. Lab may follow any relevant standard; however, care shall be taken to follow the requirements in totality.

### 3 Metrological Requirement for the Calibration Force Proving Instruments

- a) For Each weight, the expanded uncertainty, U, for k=2, of the true mass.
- b) All Newtonian weights preferably of F2 or better will be used as a standard as per OIML R-111.
- c) 'g' value shall be known with sufficient accuracy either by Geological Survey of India or any other relevant source for finer CMC.
- d) Laboratory may also calculate 'g' value knowing latitude and height as per the formula in annexure A. However, same shall be validated as per attached method.
- e) Since mass has to be calibrated in true mass basis; the air buoyancy correction shall be applied.
- f) Knowing the true mass and 'g' value, Newtonian value will be determined after applying buoyancy correction.

### 4 Recommended Selection of Reference Standard Force Calibration Machine

Reference standard calibration machine is required for application of Force to calibrate Force proving instruments. Calibration of force proving instruments will generally be carried out in accordance with IS 4169:2014 /ISO 376:2011 and the uncertainty of the calibration results will be dependent on the applied force, as well as on the performance of the instrument during calibration.

The machine should be capable of calibrating the instruments in compression and tension mode, application of force in both increasing and decreasing steps, performing creep test and axial application of force.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 58 of 133

Force calibration machines should be sturdy enough to receive reaction forces without any deformation, and should have ability to hold the force transducer at the correct alignment- i.e. with its measuring axis vertical and concentric to the applied force-at each applied force, will have an effect on the magnitude of the force vector applied to the transducer measuring axis. The machine should have stiff compression platen without any side force generation which otherwise may affect force transducer output.

The uncertainty of applied force shall be of order  $1 \times 10^{-4}$  (100 ppm or 0.01%) for verification of proving instruments of class 00, 0.02% for class 0.5(refer 8.10.4 of this document). This enables the calibration laboratory to calibrate force proving instruments to the best classification specified within IS 4169 / ISO 376.

## 5 Requirement of Traceability

All definitive measurements that are used for realization of force shall have traceability certificates will include but not limited to the parameters given in the table below.

Sl. No.	Type of Force Calibration Machine	Traceability Certificates required but not limited to
1	Dead Weight	True mass value, 'g' value upto 500kN, Temperature, Humidity and Atmospheric pressure. In case of small machines below 100 kg Traceability certificate obtained by calibrating the machine against calibration grade Force transducer (loadcell), where mass calibration is not feasible.
2	Hydraulic Amplification	True mass value, 'g' value upto 500kN, Temperature, Humidity and Atmospheric pressure and Certificate of Area of piston cylinder unit to interpret Multiplication ratio k.
3	Lever amplification	True mass value, 'g' value upto 500kN, temperature, humidity and atmospheric pressure, Length and ratio of the lever system.
4	Built up system force standard machine	Reference force transducers.

### 5.1 Lever amplification force standard machines

In a lever amplification machine, a deadweight force is amplified by the use of one or more mechanical lever systems, increasing the force by a factor approximately equal to the ratio of the lever arm lengths. Where the traceability of this larger force is directly derived from SI units, the uncertainty contributions that need to be considered will include, but are not limited to, the following:

- ✓ Uncertainty of the deadweight force.
- ✓ Uncertainty of the lever arm length measurement.
- ✓ Uncertainty due to friction within the lever systems.
- ✓ Uncertainty due to effect of temperature on lever arm ratio (thermal expansion, at possibly different rates, of lever systems).
- ✓ Uncertainty due to effect of applied force magnitude on lever arm ratio (elastic distortion of lever systems).
- ✓ Uncertainty due to instability of control system.
- ✓ Uncertainty due to alignment of generated force with transducer's measuring axis.
- ✓ Uncertainty due to positional reproducibility of moveable parts.
- ✓ Uncertainty due to wear/stability of knife-edges, if used.

Where possible, corrections should be made for the estimated effect of any of these components on the magnitude of the generated force. The standard uncertainties associated with these corrections, together with the standard uncertainties due to any effects that cannot be corrected for, should be combined in quadrature (if it can be demonstrated that the effects are not correlated) and then multiplied by a coverage factor to derive an expanded uncertainty for the generated force.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 59 of 133

## 5.2 Build-up System with multiple transducer (1 or 3) system- comparator type force standard machine

These machines are based on a number of force transducers, individually calibrated in a force standard machine and then loaded in parallel. The generated force is calculated as the sum of the forces being measured by the individual transducers. For this type of machine, the uncertainty contributions that need to be considered will include, but are not limited to, the following:

- Uncertainty of the calibrations of the individual transducers.
- Uncertainty due to use of transducers subsequent to their calibration.
- Uncertainty due to alignment of transducers with the measuring axis of the transducer under calibration.
- Uncertainty due to stability/performance of control system and data acquisition methodology.

Where possible, corrections should be made for the estimated effect of any of these components on the magnitude of the generated force. The standard uncertainties associated with these corrections, together with the standard uncertainties due to any effects that cannot be corrected for, should be combined in quadrature (if it can be demonstrated that the effects are not correlated) and then multiplied by a coverage factor to derive an expanded uncertainty for the generated force.

## 5.3 Effect of weight, gravity, buoyancy, and temperature on generated Force

### 5.3.1 Weight Consideration

Weights should be calibrated in terms of Newton. If the weight is in terms of kgs and converted in terms of force using the formula  $[F= m*g]$

For example: for  $m = 1 \text{ kg}$  the generated force  $=1*9.80665= 9.80665 \text{ N}$ .

For Force of 10N, we require denomination of weights 1 kg, 10 g, 5 g, 2 g, 2 g, 500 mg, 200 mg, 10 mg & 1 mg to get 1.019716 kg.

The shape of the weights used in the calibration machine should be such that, it doesn't affect verticality of the measuring axis and concentric to the applied force. Otherwise, the magnitude of error of applied force will be more and hence the uncertainty since, force is a vector quantity.

### 5.3.2 Gravitational Effects consideration

It is very important to establish the gravitational value of the laboratory since it is one of the major quantity during realization of force. The effect of not doing this could be a variation in force produced by the weight perhaps 0.5% of the force. It is therefore recommended that, the Force calibration laboratory establishes local value of gravity (g) and use weights that have been calibrated at that acceleration due to gravity. .

To achieve applied Force uncertainty better than 0.01%, lab to have the local acceleration due to gravity measured at the site of calibration.

### 5.3.3 Buoyancy effect consideration:

The weights are used to generate a downward force in air during force calibration (not in Vacuum). This means that, Archimedes' principle applies i.e. air pressure under the weights causes an upward force. This reduces the effective force generated by the weights and therefore the mass must be increased to allow for this. If the weights are calibrated on conventional mass basis under standard conditions of air density of  $1.2 \text{ kg/m}^3$  at  $20^\circ\text{C}$  and density of weight  $8000 \text{ kg/m}^3$ . The increase is required by a factor of 0.015%. Because, realization of force is a product of true mass local g and local air density.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 60 of 133

### 5.3.4 Effect of change in temperature on calibration results

The weights and the hanger used are of steel material. The variation of temperature changes the volume of the material due thermal expansion and affects the value of mass as it is a function of density and volume.

The variation of temperature affects the air density, which is used in buoyancy correction. Hence, change in temperature affects in realization of force values.

Apart from the above, it affects change in length also in a lever multiplication system and there by uncertainty due to thermal expansion depending on the material used.

In a hydraulic multiplication system, the piston and cylinder area is affected due to change in temperature and it is very sensitive to temperature and changes the generated pressure significantly.

The change in temperature also affects the electronic systems used during calibration either as a control system, reference system or UUT and will contribute to uncertainty.

Hence, Calibration of force proving instruments should be carried out in controlled environmental condition.

## 6 Traceability Paths

There are two distinct traceability paths for the forces generated by the force calibration machine and the method of assessment of the associated uncertainty and CMC depend on the chosen method. Re

### 6.1 Traceability Path A: The force calibration machine directly from transfer standards calibrated in national force standard machine.

Refers to the machines where independent traceability of Mass, length and time are not feasible due to:

- The weights cannot be dismantled by the machine.
- Non-availability of calibration facility for large weights.
- High cost involved.
- Difficult to characterize and estimate the uncertainty components in hydraulic amplification or lever amplification machines.
- If path A is followed for evaluation of CMC, that becomes the measurement capability of the laboratory. The evaluation document should be made available to the assessor during audit for verification.

### 6.2 Traceability Path B: The force calibration machine has independent traceability to the base SI units of Mass, length and time. This traceability is derived from measurements of mass, gravity, lever length, piston areas etc. and the uncertainty associated with the generated force (and the laboratory's claimed CMC) is calculated, as for national force standard machines, from the uncertainties associated with these measurements, together with the other contributions detailed in section 4 of Euramet cg-4. It is necessary also to perform comparisons between the force calibration machine and an appropriate national force standard machine using high quality transfer standards – the procedure for this work may be as described in section 5.2 of Euramet cg-4 but the results need to be analysed in a different way, as it is a comparison exercise rather than a calibration. The analysis needs to demonstrate whether or not the results from the two machines are metrologically compatible – one method for assessing this is described in section 4 of Euramet cg-4 and involves determining whether or not the En values calculated across the range of applied force exceed unity. If these values do exceed unity, it is not sufficient simply to increase the CMC to reduce the En value to an acceptable level, but the whole uncertainty budget associated with the force calibration machine (and with the comparison procedure) should be reviewed to the satisfaction of the national accreditation body.

1. Applicable where weights can be dismantled easily, calibration facility is available and economically feasible.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 61 of 133

2. If path B is followed, estimation of expanded uncertainty is done with following contributions:
  - Type A uncertainty of repeated measurements of nearly ideal artifact.
  - Type B contributions given below.
  - Uncertainty of true Mass\*
  - Uncertainty due to drift in mass.
  - Uncertainty of 'g' acceleration due to gravity.
  - Uncertainty of 'g' due to change in length of mass stack.
  - Uncertainty due to Density of Mass.
  - Uncertainty due to Density of Air.
  - Uncertainty of conventional mass has to be considered where conventional mass value is taken

### 6.3 Procedure for evaluation of CMC of the machines:

1. Determination of the uncertainty of the force generated by the national force standard machine.
2. Determination of the calibration uncertainty of the transfer standard in the national force standard machine.
3. Determination of the uncertainty of the transfer standard's reference value.
4. Determination of the uncertainty of force generation in the calibration machine.
5. Determination of the calibration machine CMC.

**Note 1:** For more details refer EURAMET/cg-04/v.01 calibration guide "Uncertainty of force measurements"

**Note 2:** This may require several force transducers to cover the full range. As they have to be used from 50% to 100 % to avoid interaction effect.

## 7 Legal Aspects

Calibration of Load cell done by any accredited laboratories is meant for scientific and industrial purpose only. However, if load cell is used in equipment for commercial trading, additional recognition/ approval shall be complied as required by Dept. of Legal metrology, Regulatory bodies, etc.

## 8 Recommended Environmental Conditions

8.1 Lab is advised to follow Manufacturer's recommendation for environmental conditions, operations and maintenance. Otherwise, Laboratory using dead weight calibration machine shall fulfill the following conditions for realization of applied force.

8.2 The temperature shall be maintained in the range of 18°C to 28°C. The temperature shall not vary more than ±1°C throughout a measurement series. The relative humidity shall be maintained at 50% RH ± 15% RH.

8.3 For measurement uncertainty associated with the applied force, 'g' value shall be known. for realization of applied force more than 0.01%, 'g' value shall be calculated using the formula given in below. For applied force < 0.01%, 'g' value shall be measured by appropriate authority.

8.4 Validation of local 'g' and its uncertainty

Formula for calculation of Acceleration due to gravity

An approximate value for  $g$ , at given latitude and height above sea level, may be calculated from the formula:

$$g(1 + A \sin^2 L - B \sin^2 2L) - 3.086 \times 10^{-6} H \text{ m}\cdot\text{s}^{-2}$$

Where,  $A = 0.005\ 302\ 4$ ,  $B = 0.000\ 005\ 8$ ,  $L$  = latitude,  $H$  = height in meter above sea level.

8.5 To validate this calculated 'g' value the simple steps given below can be followed:

- Find out the actual 'g' value of NMI from the certificate issued by them or by any other source.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 62 of 133

- Find out the actual 'g' value of NMI from the certificate issued by them or by any other source.
- From the web search engine maps click on the location of NMI, find out latitude and height above sea level. (you can know the 'g' value).
- Calculate the 'g' value using the above formula with these latitude and height. The difference between the calculated value of 'g' and the actual value of the NMI should be within 20 to 30 ppm.
- Now, go to the web search engine maps and click on location of the lab and find out the latitude and height of the place as per web search engine (you can know the 'g' value also).
- Calculate the 'g' value for this latitude and height. The value obtained should be within 20 to 30 ppm.
- Then this value can be taken as 'g' value of the lab and uncertainty of 'g' can be assumed to be within  $\pm 50$  ppm.

8.6 Air density should be known to sufficient accuracy depending on the required uncertainty of the applied force by measuring temperature, RH & barometric pressure.

Approximation formula as per OIML R-111-1: 2004 (pg. No. 76)

$$(E-3.1 \text{ OIML}) \quad \rho_a = \frac{0.34848p - 0.009 \cdot h \cdot e^{(0.061 \cdot t)}}{273.15 + t}$$

Where, Pressure (P) in mbar, temperature (t) in ° C and humidity (h) in % Equation(E-3.1) has a relative uncertainty of  $2 \times 10^{-4}$  in the range  $900 \text{ hPa} < p < 1100 \text{ hPa}$ ,  $10^\circ \text{ C} < t < 30^\circ \text{ C}$  and  $rh < 80\%$ .

8.7 The Magnitude of vibration, Shock or other disturbing conditions shall be such that, they will have a negligible effect on the reading of standard equipment.

8.8 Recommended resolution for environmental monitoring equipment:

- Temperature with a resolution of  $0.1^\circ \text{ C}$
- Humidity with a resolution of 1% RH
- Barometer with a resolution of 1 mbar

However, laboratory may evaluate the requirement of accuracy, resolution and uncertainty of monitoring equipment depending on the CMC claimed.

## 9 Recommended Calibration Interval

Reference Equipment	Recommended Interval
Dead weight calibration machine with stainless steel weights	5 years
Dead weight calibration machine with alloy steel weights	4 years
Lever type and hydraulic amplification machine with stainless steel weights	4 years
Lever type and hydraulic amplification machine with alloy steel weights	3 years
Comparator type machine	26 months

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

## 10 Calibration Methods

Force proving instrument can be calibrated either in tension, compression or both modes.

The range for which Force Proving Instruments is classified is determined by considering each calibration Force, one after the other, starting with the maximum Force & decreasing to the lowest calibration Force. The classification range ceases at the last Force for which the classification requirement is satisfied.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 63 of 133



The Force proving instruments can be classified either for specific Forces or for interpolation, and for either incremental only or incremental / decremental loading directions.

### 10.1 Classification Criteria:

10.1.1 The range of classification of a Force proving instrument shall at least cover the range 50% to 100% of  $F_N$ .

10.1.2 CASE A: For instruments classified only for specific force and incremental only loading, the criteria which shall be considered are:

- The relative reproducibility, repeatability and zero errors.
- The relative creep errors

10.1.3 CASE B: For instruments classified only for specific force and incremental / decremental loading, the criteria which shall be considered are:

- The relative reproducibility, repeatability and zero errors.
- The relative reversibility errors

10.1.4 CASE C: For instruments classified only for interpolation and incremental only loading, the criteria which shall be considered are:

- The relative reproducibility, repeatability and zero errors
- The relative interpolation errors
- The relative creep errors

10.1.5 CASE D: For instruments classified for interpolation and incremental / decremental loading, the criteria which shall be considered are:

- The relative reproducibility, repeatability and zero errors
- The relative interpolation errors
- The relative reversibility errors

### 10.2 Calibration Procedures

Only the salient features are given below.

- Load Fittings
- Force proving instruments used for calibration of Uniaxial testing machines as per IS:1828 (part-1) and Tension creep testing machines as per IS:1828(part-2) are to be calibrated along with its complete accessories. If they are calibrated without their fittings, it should be clearly mentioned in the calibration report.

#### 10.2.1 Minimum Force (lower limit of calibration)

Taking into consideration the accuracy with which the deflection of the instrument can be read during the calibration or during its subsequent use for verifying machines the minimum force applied to a force proving instrument shall comply with the following conditions.

- a) The minimum force shall be greater than or equal to:

Class	Minimum force applied shall be
00	$\geq 4000 \times r$
0.5	$\geq 2000 \times r$
1	$\geq 1000 \times r$
2	$\geq 500 \times r$
Where r is the resolution of Indicator	

Where  $F_f$  = maximum capacity of the force proving device

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 64 of 133

b) The minimum force shall be greater than or equal to 0.02 Ff.

## 11 Measurement Uncertainty

10.1 The components of uncertainty to be considered but are not limited to the following

Combined Uncertainty,  $u_c = \sqrt{(u_1^2 + u_2^2 + u_3^2 + u_4^2 + u_5^2 + u_6^2 + u_7^2 + u_8^2 + u_9^2)}$

Expanded relative uncertainty,  $W = k \times u_c$

Where,

$u_1$  = Standard uncertainty associated with applied calibration force

$u_2$  = Standard uncertainty associated with reproducibility of calibration results

$u_3$  = Standard uncertainty associated with repeatability of calibration results

$u_4$  = Standard uncertainty associated with resolution of the indicator

$u_5$  = Standard uncertainty associated with creep of instrument

$u_6$  = Standard uncertainty associated with drift in zero output

$u_7$  = Standard uncertainty associated with temperature of the instrument

$u_8$  = Standard uncertainty associated with interpolation

$u_9$  = Standard uncertainty associated with reversibility (hysteresis)

The components of uncertainty to be considered but are not limited to the following

Combined relative Uncertainty,  $w_c = \sqrt{(w_1^2 + w_2^2 + w_3^2 + w_4^2 + w_5^2 + w_6^2 + w_7^2 + w_8^2 + w_9^2)}$

Expanded relative uncertainty,  $W = k \times w_c$

Where,

$w_1$  = Relative standard uncertainty associated with applied calibration force

$w_2$  = Relative standard uncertainty associated with reproducibility of calibration results

$w_3$  = Relative standard uncertainty associated with repeatability of calibration results

$w_4$  = Relative standard uncertainty associated with resolution of the indicator

$w_5$  = Relative standard uncertainty associated with creep of instrument

$w_6$  = Relative standard uncertainty associated with drift in zero output

$w_7$  = Relative standard uncertainty associated with temperature of the instrument

$w_8$  = Relative standard uncertainty associated with interpolation

$w_9$  = Relative standard uncertainty associated with reversibility (hysteresis)

**Note 1:** The interpolation component ( $u_8$ ,  $w_8$ ) is not taken into account in the calibration uncertainty with instruments classified for specific forces only.

**Note 2:** The relative uncertainty can be expressed as % by multiplying 100.

**Note 3:** For determination of creep error, refer Cl.7.5 of IS 4169:2014 / ISO 376:2011.

**Note 4:** For determination of uncertainty components, refer Annexure C of IS 4169 / ISO 376.

**Note 5:** Accurate determination of the Hysteresis of the device may be performed on the Dead Weight machines. For other type of machines, Hysteresis shall be evaluated and considered otherwise it will be a combined value of Hysteresis of the machine & device under calibration.

### 10.2 Key Points

10.2.1 Laboratory has to demonstrate the expanded uncertainty applied calibration force (at 95% confidence level) as per table below.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 65 of 133

Limit of combined uncertainty in % for Force Proving Instruments as per standard IS 4169/ ISO 376					
Class	Maximum permissible calibration Force uncertainty With k=2	For interpolation		For specific forces	
		Creep Reversibility)	(No Reversibility ( No Creep)	Creep (No Reversibility)	Reversibility ( No Creep)
00	0.01	0.081	0.075	0.063	0.056
0.5	0.02	0.162	0.151	0.127	0.114
1	0.05	0.325	0.304	0.257	0.229
2.0	0.1	0.651	0.608	0.513	0.458

**Note 1:** Laboratory has to demonstrate the applied Force uncertainty required for different class as per the table above.

**Note 2:** The cut off CMC value for the applied Force of 0.1% and Class 2 with relevant uncertainties given in the above table beyond this accreditation cannot be granted.

10.2.2 Demonstration of the uncertainties of mass, g value and other components of a Force calibration machine doesn't automatically qualify for granting accreditation. The coupling effect, misalignment effect, interaction effect, frictional effect, oscillation effect etc. of the Force calibration machine will have the effect on its performance to evaluate CMC.

The lab has to demonstrate the minimum effects of the above during calibration by taking 10 repeated readings at 10% and 100% in 0°, 120° and 240° rotated positions to arrive at worst case type A uncertainty.

10.2.3 Demonstration of any CMC values doesn't automatically qualify for granting accreditation until the lab satisfies the stipulated requirement given in this document.

## 12 National/ International Standards, References and Guideline

- ISO 376: - Metallic materials-calibration of force -proving instruments used for the verification of uniaxial testing machines.
- IS: 4169, Method for calibration of force -proving instruments used for the verification of uniaxial testing machines.
- OIML R 111-1 Metrological and technical requirement of weights.
- ASTM E 74- 10 Standard practices of Calibration of Force-Measuring Instruments for Verifying the Force Indication of Testing Machines.
- OIML- D28- Conventional value of the result of weighing in air.
- IS 1828 (Part 1 (ISO 7500-1)) -Metallic Materials-Verification of static uniaxial testing machines, Part 1: Tension/ Compression Testing Machines-Verification and calibration of the Force measuring system.

**Note: - Latest version of the relevant standard(s) should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 66 of 133

# Chapter 1(G): Calibration of UTM, Tension Creep and Torsion Testing Machine

## 1 Recommended General Requirement

- 1.1 **Acoustic Noise:** Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 **Illumination:** The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.

## 2 Specific Requirements - Calibration of Force Measuring System of Uni-axial Testing Machine

Sl. No.	Description	Relevant Standard	Permanent Facility	Onsite Calibration
1	Universal Testing Machines / Tensile Testing Machines / Compression Testing Machines	IS1828 (Part 1) ISO 7500-1	√*	√

*\*Note 1: Calibration of UTM at permanent facility (by manufacturer) is acceptable however; verification shall be required after installation and commissioning of UTM at site.*

*Note 2: UTM can also be calibrated as per ASTM E4-10 using Force proving instruments duly calibrated as per ASTM E 74.*

*Note 3: This technical requirement is based on the standard IS 1828 (Part-1). Lab may follow any relevant standard; however, care shall be taken to follow the requirements in totality.*

## 3 Metrological Requirement

- 3.1 The standard IS: 1828 (Part 1), ISO 7500-1 is for both verification and calibration of force measuring system of the Uni axial Testing machine. This specific criterion is meant for only calibration of force measuring system. If the verification is also to be included, then lab has to follow the standard completely along with Annexure -A, B and C of the standard and verification and calibration report can be issued for the machine.
- 3.2 Force Proving instruments used for calibration of uniaxial testing machines as per IS1828 (Part 1), ISO 7500-1 shall have traceability and calibration certificate as per ISO 376 (Or any other standard).
- 3.3 Proving instrument calibrated in compression mode shall not be used for calibration of the machine in Tension mode and vice versa.
- 3.4 The force proving instrument shall be calibrated along with load fittings liked loading pads for compression and ball nut, ball cup and tensile force measuring rod as per A.4 of ISO 376.

## 4 Selection of Reference Standard for UTM Calibration

- 4.1 The proving instruments used for the calibration shall have a certificate from either NMI or accredited laboratory with traceability to the SI units and shall comply with the requirements specified in ISO 376.
- 4.2 The class of the instrument shall be equal to or better than the class for which the testing machine –force indicator is to be calibrated. Initially, the class of the machine may be taken as per the relative resolution of the force indicator (Preferably).
- 4.3 Proving instrument used in compression mode shall have the certificate of calibration in compression mode. Similarly used in tension mode should have certificate of calibration in tension mode.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 67 of 133

- 4.4 The force proving instrument shall be calibrated along with load fittings like loading pads for compression and ball nut, ball cup and tensile force measuring rod as per the required application (or as per manufacturers recommendations).
- 4.5 Proving instrument (proving ring with dial) calibrated in specific forces only should be used for calibrating in the same specific force points and not in between force points. If it has to be used for in between force points, it has to have certificate of calibration with interpolation equation and the calibration certificate for the dial also.
- 4.6 All the elements of force proving instrument shall be individually and uniquely identified.

**5 Recommended Calibration Interval**

Reference Equipment	Interval
Force proving instrument used for calibration	26 months

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

**6 General Required characteristics of Force Proving Instruments:**

Please refer ISO 376 for more details.

**7 Legal Aspects**

Calibration of UTM done by any accredited laboratories is meant for scientific and industrial purpose only. However, if used for commercial trading, additional recognition/ approval shall be complied as required by Dept. of Legal metrology, Regulatory bodies, etc.

**8 Recommended Environmental Conditions**

- 8.1 The calibration shall be carried out between 10°C and 35°C. The temperature at which calibration is carried out shall be noted in the calibration certificate.
- 8.2 The temperature measuring equipment with a resolution 1°C shall be used for monitoring temperature during calibration.
- 8.3 Stability of temperature during measurement is to be mentioned.

**9 Calibration Methods**

- 9.1 Calibration can be carried out with constant indicated force of the machine for all the three series of measurement. When this method is not feasible, the calibration may be carried out with constant true forces. When more than one force proving instrument is required to calibrate a force range, maximum force applied to the smaller device shall be the same as the minimum force applied to the next force proving instrument of the higher capacity. Refer clause 6.5.3 of IS 1828 (part 1) for agreement between two force proving instruments.

**10 Calibration Procedures**

Please refer IS 1828 (Part-1) and other relevant standard for calibration of static Uniaxial Testing Machine.

**11 Scope: Calibration of Tension Creep Testing Machine**

Sl. No	Parameter-Force	Relevant Standard	Permanent Facility	Onsite Calibration
1	Tension Creep Testing Machines	IS1828 (Part 2): 2002 (ISO7500-2:1996)	√*	√

**\*Note 1:** Tension creep testing machine can also be calibrated as per relevant ASTM E 4 standard using Force proving instruments duly calibrated as per ASTM E 74.

**Note 2:** This technical requirement is based on the standard IS 1828 (Part-2). However, lab may follow any other relevant standard/guideline in totality.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 68 of 133

## 12 Metrological Requirement

Please refer IS 1828 (ISO 7500-2)

## 13 Selection of Reference Standard for Tension Creep Testing Machine Calibration

Please ISO 376 Metallic Material calibrations of Force proving Instruments used for the verification of Uniaxial testing Machine for details

### 13.1 Recommended Environmental Conditions

The temperature measuring equipment with a resolution 1°C should be used for monitoring temperature during calibration.

### 13.2 Calibration Procedures

Please refer IS 1828 (Part 2) for Tension Creep Testing Machine.

## 14 Recommended Scope: Calibration of Torsion Testing Machine

Sl. No	Parameter-Force	Relevant Standard	Permanent facility	Onsite calibration
1	Torsion testing Machine	ASTM E 2624-09	√	√*

*\*Note 1: Using torque transducer or load cell with calibrated arm.*

*Note 2: This technical requirement is based on ASTM E 2624-09. However, lab may follow any other relevant standard/guideline.*

## 15 Legal Aspects

Calibration of weights done by any accredited laboratories is meant for scientific and industrial purpose only. However, if load cell is used in equipment for commercial trading, additional recognition/ approval shall be complied as required by Dept. of Legal metrology, Regulatory bodies, etc.

## 16 Recommended National/ International Standards, References and Guideline

- IS1828 (Part 1), ISO 7500-1 -Metallic Materials-Verification of static uniaxial testing machines, Part 1: Tension/Compression Testing Machines-Verification and Calibration of the Force Measuring System.
- ASTM E4-10, Force Verification of Testing Machines.
- ISO: 376 E - Metallic Materials-Calibration of Force -Proving Instruments used for the Verification of Uni-axial Testing Machines.
- ASTM E 74 Standard practices of Calibration of Force-Measuring Instruments for Verifying the Force Indication of Testing Machines.
- OIML R 111- 1 Metrological and Technical Requirement of Weights.
- ASTM E2624 Standard Practice for Torque calibration of Testing Machine and Devices
- BS 7882: Method for calibration and classification of torque measuring devices.
- EURAMET cg 14: Static Torque measuring devices.
- ASTM E 2428 Practice for calibration of torque measuring instruments for verifying Torque indication of Torque testing machines.
- ASTM E 2624 Standard procedure for torque calibration of testing machines and devices.
- ISO 376:2011(E) - Metallic materials-calibration of force -proving instruments used for the verification of Uniaxial testing machines.
- OIML R 111- 1 Metrological and technical requirement of weights.
- ASTM E 74- Standard practice of Calibration of Force-Measuring Instruments for Verifying the Force Indication of Testing Machines.

**Note: - Latest version of the relevant standard(s) should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 69 of 133

# Chapter 1(H): Calibration of Mobile Force Measuring System (Push Pull Gauge)

## 1 Recommended General Requirements:

- 1.1 **Acoustic Noise:** Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 **Illumination:** The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.

## 2 Recommended Requirements – Calibration of Push Pull Gauge

Recommended requirements for the calibration of Mobile Force Measuring System:

Sl. No.	Mobile Force Measuring System	Relevant Standard/ Guidelines	Permanent Facility	On-site Calibration
1	Push Pull Gauge (Analog and Digital), Force Gauge	VDI/VDE 2624 Part 2.1 (December 2008)	√	X

**Note:** This technical requirement is based on the above-mentioned guideline. Lab may follow any relevant standard; however, care shall be taken to follow the requirements in totality.

## 3 Metrological Requirement

Please refer OIML R-111-1

## 4 Recommended Selection of Reference Standard

Please refer OIML R 111-1

## 5 Recommended Calibration Interval

Reference Equipment	Recommended Interval
Dead weight push pull force gauge calibration machine with stainless steel weights	5 years
Dead weight push pull force gauge calibration machine with alloy steel weights	4 years

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

## 6 Calibration Procedures

Please refer VDI/VDE 2624 Part 2.1(December 2008)

## 7 Recommended National/ International Standards, References and Guideline

- VDI/VDE 2624 Part 2.1 (December 2008) - Instructions for calibration of mobile force measuring system.
- OIML R 111-1 Metrological and technical requirement of weights.
- OIML - D28- Conventional value of the result of weighing in air

**Note:** - Latest version of the relevant standard(s) should be followed

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 70 of 133

# Chapter 1(I): Calibration of Verification of Hardness Testing Machine

## 1 Recommended General Requirements

- 1.1 **Acoustic Noise:** Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 **Illumination:** The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.

## 2 Recommended Requirements – Verification of Hardness Testing Machine

Sl. No.	Description	Relevant Standard	Permanent Facility	Onsite Calibration	Mobile Facility
1	Rockwell Hardness	ISO 6508-2	√*	√	X
2	Brinell Hardness	ISO 6506-2	√*	√	X
3	Vickers Hardness	ISO 6507-2	√*	√	X
4	Knoop hardness	ISO 4545-2	√*	√	X
5	Rubber - Calibration and Verification of Hardness Testers - Durometers	ISO 18898-2012, ASTM-D-2240-05	√	X	X

**\*Note 1:** Verification of Hardness Testing Machine at permanent facility is acceptable. However, verification of performance shall be carried out after installation and commissioning of Hardness Testing Machine at site.

**Note 2:** This technical requirement is based on the above-mentioned standards. Lab may follow any relevant standard; however, care shall be taken to follow the requirements in totality.

## 3 Metrological Requirements

- 3.1 The condition of indenters should be monitored by visually checking the aspect of the indentation on a reference block, each day the testing machine is used.
- 3.2 The verification of the indenter is no longer valid when the indenter shows defects, reground or otherwise repaired indenters shall be verified.
- 3.3 In the case of direct verification of hardness testing machine each measurement force applied shall be within  $\pm 1.0\%$  of accuracy.
- 3.4 The force shall be measured by means of a force proving instrument in accordance with ISO 376 - class 1 or IS 4169 - class 0. By balancing against a force, accurate to  $\pm 0.2\%$ , applied by means of calibrated masses or another method with the same accuracy.
- 3.5 For indirect verification, the reference blocks shall be calibrated in accordance with ISO 6506-3 (For Brinell), ISO 6507-3 (For Vickers), ISO 6508-3 (For Rockwell) & ISO 4545-3 (For Knoop Hardness).
- 3.6 For indirect verification, the difference between the mean measured value and the certified mean diameter shall not exceed 0.5%.
- 3.7 The error of the measuring device, expressed as a percentage of the assigned length of the mean indentation diagonal of each reference indentation, shall be not more than 1%.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 71 of 133



- 3.8 IS 4545-2 for verification and Calibration of Knoop Hardness Testing machine covers test forces from 0.09807 N to 19.614 N.
- 3.9 Three readings shall be taken for each test force. Immediately before each reading is taken, the indenter shall be moved in the same direction as during the test. All readings shall be within the tolerances defined in Table 1.
- 3.10 The indenter (Diamond) shall be calibrated, conforming to the requirements of geometrical deviations.
- 3.11 On each reference block, five indentations shall be made & measured.
- 3.12 For the calibration and verification of Hardness Testers- Durometers the measurands of indenter and pressure foot for the instruments to be calibrated are depicted in figures 1 to 6 of ISO 18898 (table 1 to 9).

#### 4 Methods for Verification

The requirements for a comprehensive service to meet the needs of industry for the established scales of hardness, together with the specific requirements of current Standards, have been divided into two categories as follows:

##### 4.1 Indirect verification of testing machines

For each scale of hardness a laboratory will require requisite sets of standardized test blocks and standard indenters, together with the apparatus necessary to carry out the preliminary inspection of the testing machine. The standardized test blocks and indenters will require periodic re-standardization.

##### 4.2 Direct verification of testing machines

Direct verification of testing machines, which have passed indirect verification, requires verification of loads, indenters and the measuring device.

#### 5 Measurement Uncertainty

- 5.1 A procedure for the estimation of uncertainty in hardness measurement by the indirect calibration method:

Step 1: Identifying the Parameters for Which Uncertainty is to be estimated

The first step is to list the quantities (measurands) for which the uncertainties must be calculated. Table 1 shows the parameters that are usually reported in hardness measurements by the indirect calibration method. None of these measurands are measured directly, but are determined from others quantities (or measurements).

**Table 1 Measurands, their units and symbols**

Measurands	Units and Symbol
Rockwell hardness	HR (Scale)
Brinell hardness	HBW
Vickers hardness	HV
Knoop hardness	KH

**Table 2 Measurements, their units and symbols**

Measurements	Units	Symbol
Permanent increase in depth of penetration under preliminary test force after removal of additional force	0.002mm (regular scale) 0.001mm (superficial scale)	H
Single diameter of the indentation	Mm	d1(HB), d2(HB)
Single diagonal of the indentation	mm	d1(HV), d2(HV)

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 72 of 133

Step 2. Identifying all Sources of Uncertainty in the Test

- Uncertainty due to calibration of reference blocks
- Uncertainty of maximum permissible error according the standards
- Uncertainty due to repeatability at certain test conditions

5.2 Mathematical Formulae for Estimating Uncertainties in Hardness Measurement by the Indirect Calibration Method:

Procedure:

5.2.1 Certified value of the CRM (Certified Reference Material)

CRM ... Mean value of five measurements

CRM ... Uncertainty of the mean value calculated by the calibration laboratory.

It is the 2-Sigma uncertainty (confidence level: 95%).

5.2.2 Determination of users machine repeatability

a) 
$$H = \frac{H_1 + H_2 + H_3 + H_4 + H_5}{5}$$
 (3)

b) Repeatability according EN ISO 6506, 6507, and 6508

$$d_5 - d_1 \text{ (Vickers; EN ISO 6507 and Brinell; EN ISO 6506)} \quad (4)$$

c) Empirical standard deviation of a single value

$$s_n = \sqrt{\frac{1}{(n-1)} \sum_{i=1}^n (H_i - \bar{H})^2}, n=5 \quad (5)$$

5.2.3 Assessment according EN ISO 6506, 6507, and 6508

Estimation of the error (E) of a laboratory testing device by n repeated measurements on the same CRM

$$E = X_{CRM} - \bar{H} \quad (6)$$

The error (E) shall not exceed the values given in Table 2 of EN ISO 6506-2, Table 5 of EN ISO 6507-2, and Table 5 of EN ISO 6508-2.

- Permissible repeatability values given in Table 2 of EN ISO 6506-2, Table 4 of EN ISO 6507-2, and Table 5 of EN ISO 6508-2.

5.2.3.1 Measure on a material by n1 repetition

a) Mean Value

$$\bar{X} = \frac{\sum_{i=1}^n x_i}{n_i} \quad (7)$$

b) Empirical standard deviation of a single value

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129		Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)		
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 73 of 133

$$s_n = \sqrt{\frac{1}{(n_1-1)} \sum_{i=1}^{n_1} (x_i - \bar{x})^2} \quad (8)$$

5.2.3.2 In order to estimate the uncertainty of the mean value,  $\bar{x}$ , following mathematical model has been used.

Combined uncertainty

$$u_{C\bar{x}}^2 = E_w^2 + u_{CRM(i\sigma)}^2 + u_{\bar{H}}^2 + u_{\bar{x}}^2 + \dots \quad (9)$$

Expanded uncertainty

$$u(\bar{x}) = k u_c(\bar{x}); k = 2 \quad (10)$$

a) The permissible error  $\pm E_{2\sigma}$  is a 2-Sigma error (normal distribution is assumed), therefore

$$E_{1\sigma} = \frac{E_{2\sigma}}{2} \quad (11)$$

b) The uncertainty  $u_{CRM}$  is a 2-Sigma uncertainty, therefore

$$u_{CRM(1\sigma)} = \frac{u_{CRM(2\sigma)}}{2} \quad (12)$$

c) The uncertainty of  $\bar{H}$  (mean value of users machine on CRM)

$$u_{\bar{H}} = \frac{ts_{\bar{H}}}{\sqrt{n}} \quad (13)$$

t ... Student coefficient for 'n' and P = 68.27%

d) The uncertainty of  $\bar{x}$  (measurements on a material)

$$u_{\bar{x}} = \frac{ts_{\bar{x}}}{\sqrt{n_1}} \quad (14)$$

t ... Student coefficient for 'n1' and P = 68.27%

n1 ... at least 5 repetitions; Eqn. 14 would be valid for  $n_1 \geq 2$

Where

ci	sensitivity coefficient
CoP	Code of Practice
d(x)	diameter or diagonal of the indentation (i.e. d1(HB))
dv	divisor used to calculate the standard uncertainty
E	exactness of impact testing machine
F1	additional force
Fo	preliminary test force
h	permanent increase in depth
HB	Brinell hardness
hM	measurement for the difference in depth
HR	Rockwell hardness
HV	Vickers hardness
k	coverage factor used to calculate expanded uncertainty (normally corresponding to 95% confidence level)
N	number of input parameters xi on which the measurand depends

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129		Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)		
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 74 of 133

- n number of repeat measurements
- p confidence level
- s experimental standard deviation (of a random variable) determined from a limited number of measurements, n
- U expanded uncertainty
- u standard uncertainty
- uc combined standard uncertainty
- V value of the measurand
- xi estimate of input quantity
- x arithmetic mean of the values of the random variable xi
- y test (or measurement) mean result

Contribution to uncertainty of measurement (Knoop Hardness Testing Machine):

1. Direct Verification:

- Uncertainty due to measurement of the hardness testing machine (Type A).
- Uncertainty due to force transducer (From calibration certificate).
- Uncertainty due to test force generated by the hardness testing machine.
- Uncertainty due to temperature.
- Uncertainty due to long-term stability.
- Uncertainty due to interpolation deviation.
- Uncertainty due to object micrometer/optical measuring system (from calibration certificate).
- Uncertainty due to the resolution of the measuring system.

2. Indirect Verification:

- Uncertainty due to measurement of the hardness testing machine (Type A).
- Uncertainty due to reference hardness block (From calibration certificate).
- Uncertainty due to the resolution of the hardness testing machine.

5.2.4 Uncertainty components for Indentation Depth:

- Uncertainty of length measuring device.
- Standard deviation of length measurements.

5.2.5 Uncertainty components for Force Verification:

- Uncertainty of applied force.
- Standard deviation of force measurements.

**5.3 Recommended National/ International Standards, References and Guideline**

- ISO 6508-2 - Metallic material-Rockwell Hardness Test- part-2 –verification and calibration of the testing machine.
- ISO 6506-2– Metallic material-Brinell Hardness Test -part-2 –Verification and calibration of the testing machine.
- ISO 6507-2– Metallic material-Vickers Hardness Test -part-2 –Verification and calibration of the testing machine.
- ISO 4545-2– Metallic material-Knoop Hardness Test -part-2 - Verification and calibration of the testing machine.
- ISO-48-9 Rubber, vulcanized or thermoplastic- Determination of hardness-Calibration and verification of hardness testers.
- ISO 18899 – Rubber – Guide to the Calibration of Test Equipment.
- ASTM-D-2240-05 – Standard test method for Rubber Property – Durometer Hardness.

**Note: - Latest versions of the relevant standard(s) should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 75 of 133

# Chapter 1(J): Calibration of Torque Measuring Devices

## 1 Recommended General Requirements:

- 1.1 **Acoustic Noise:** Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 **Illumination:** The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.

## 2 Specific Requirements – Calibration of Torque Measuring Devices

**Scope: Calibration of Torque Measuring Devices** (Torque meters, Torque Calibrators, Torque Transducer/Sensor)

Specific requirements for the calibration of Torque Measuring Devices:

Sl. No	Description	Relevant Standard/ Guidelines	Permanent facility	Onsite calibration
1	Torque Measuring Devices (Electrical, Mechanical, Hydraulic, Optical torque measuring device)	BS 7882	√	X

**Note 1:** Torque measuring devices can also be calibrated as per ASTM E2428-08 which is used for calibration of Torque Testing Machines as per ASTM E2624-09.

**Note 2:** This technical requirement is based on **BS 7882**. Lab may follow any relevant standard; however, care shall be taken to follow the requirements in totality.

## 3 Metrological Requirement for the calibration Torque Measuring Devices

- 3.1 For Each weight, the expanded uncertainty, U, for  $k=2$ , of the true mass.
- 3.2 All weights used for verification of force shall be in Newton.
- 3.3 Preferably all weights will be equivalent or better than F2 standard as per OIML R-111.
- 3.4 'g' value shall be known with sufficient accuracy either by Geological Survey of India or any other relevant source for finer CMC.
- 3.5 Laboratory may also calculate 'g' value knowing latitude and height. However, same shall be validated.
- 3.6 Since mass has to be calibrated in true mass basis, the air buoyancy correction shall be applied.
- 3.7 Knowing the true mass and 'g' value, value in Newton will be determined after applying buoyancy correction.

## 4 Recommended Selection of Reference Standard Torque Calibration Machine

Calibration of torque measuring devices in the torque calibration machines will generally be carried out in accordance with a documented procedure such as BS 7882:2008 and the uncertainty of the calibration results will be dependent on the calibration machine uncertainty (maximum permissible uncertainty of calibration torque applied), as well as on the performance of the instrument during calibration.

The machine shall be capable of calibrating the instruments in clockwise/anti-clockwise, application of torque in both increasing and decreasing steps. It shall ensure axial application of torque.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 76 of 133

## 4.1 Types of Torque calibration Machine

Reference standard calibration machine is required for application of Torque to calibrate Torque measuring device. The calibration machine shall be sturdy enough to receive reaction forces without any deformation and shall have the ability to hold the torque transducer or device at the correct alignment with its measuring axis axial and concentric to the applied torque - at each applied torque will have an effect on the magnitude of the force vector applied to the transducer measuring axis. Machine shall have proper couplings without any side force generation which otherwise may affect torque transducer output.

### 4.1.1 Dead Weight Torque Calibration Machine with calibrated beam (lever arm) supported with antifriction bearing:

For achieving uncertainty of torque applied in the range of  $\pm 0.01\%$  to  $\pm 0.04\%$  it is practically possible with dead weight torque calibration machine with calibrated beam (lever arm) supported with antifriction bearing, Calibrated Newton weights (preferably stainless steel). Local 'g' shall also be known with sufficient accuracy and uncertainty of measurement.

Torque is a product of tangential force and length applied about a known centre of rotation. Since Force is a vector quantity when a beam and weights are used as a calibration equipment, appropriate steps to be taken shall be to ensure that, there is no misalignment of the unit under test which could generate parasitic forces and cause a bias on the system.

The Torque Calibration machine with beam and weights should have provision so that UUT can be mounted in a position such that can be rotated through  $360^\circ$  in  $90^\circ$  increments or  $120^\circ$  increments in both clockwise and anti-clockwise directions about its principal axis of rotation between measurement series. When the design or specification of the UUT does not accommodate for rotation it shall be physically disconnected and removed from the calibration equipment and reconnected between measurement series.

Applied torque depends on following factors:

- Mounting of UUT without any misalignment between the arm and the reaction gearbox used to rotate about its principal axis.
- Quality of the gearbox used for rotation.
- Effect due to bearing friction.
- Class of accuracy and material of the Newton weights used.
- Cosine error, which depends on maintaining the horizontal position of the beam which changes in each step of a series when Newton weight is applied to create torque.
- Maintaining the verticality of the load applied to the horizontal beam which affects the repeatability of measurements.
- Oscillation effect on the hanging weights.
- Local acceleration of gravity.
- Buoyancy correction.
- Operator skill in applying the load slowly and gradually.
- Maintaining the stability of the temperature during calibration.
- Magnitude of vibration, shock or other disturbing conditions during calibration.

To achieve above condition, the torque calibration machine shall have torsionally rigid couplings between the UUC and the beam on one side and UUC and High ratio Gear box (used for rotation) on the other side along with sturdy high-quality bed which can accommodate various sizes of UUC.

While applying Torque, beam shall be maintained horizontal to the axis using spirit level or any other method to minimize cosine error to the applied Torque.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 77 of 133

The calibration machine shall be designed in such a way that, the load is applied exactly at a known distance of beam precisely and vertically.

Weights used shall be preferably of stainless steel and calibrated to F2 or better class of accuracy as per OIML-R-111-1 in Newton. Local value of acceleration due to gravity “g” should be known to sufficient accuracy along with uncertainty and buoyancy correction needs to be applied.

#### 4.1.2 Torque calibration machine with unsupported beam:

- It is generally accepted that a bearing supported beam is used for measurements of the highest accuracy in order to minimize bending effects on the test transducer. The weight of the beam is most significant in relation to the torque being measured. Suitability of a particular beam is also relative to the stiffness of the transducer under calibration. The distance between the beam and the transducer shall be kept minimum. Wherever it is possible, it is always best to connect the beam directly to the transducer in order to minimize any slack in the coupling of the square drives and the transducer itself shall be held as rigidly as possible in the calibration fixture.
- While applying Torque, beam shall be maintained horizontal to the axis using spirit level or any other method to minimize cosine error to the applied Torque.
- The calibration machine shall be designed in such a way that, the load is applied exactly at a known distance of beam precisely and vertically.
- Unsupported calibration beams coupled directly to a transducer is used in industry for verification of low torque applications.
- Un-supported beams generate additional bending moment and side force which makes the calibration conditions very different from pure torque conditions.
- The characteristics of a transducer, its behavior, its output and the way it is mounted are important factors to be considered.
- Sensitivity and bending effects should be considered as part of the uncertainty calculations

##### 4.1.2.1 Method to determine bending effects for unsupported beam:

- The magnitude of bending effect depends on the design and structure of the transducer which may not be known to the calibration lab. The calibration process may damage the torque transducer due to bending effect. Lab has to identify the bending effect before starting the calibration as per below procedure.
- Example for verifying the bending effect of 100 Nm Torque Transducer.
- Apply load in steps of 10% (i.e. 10 Nm) of the full-scale value in clockwise direction and note down the torque value T1.
- Apply additional load of 5 Nm (50% of 10 Nm) on both clockwise and counterclockwise directions i.e. at the end there will be 15 Nm (150% of 10Nm) on clockwise and 5 Nm (50% of 10 Nm) value on counterclockwise direction, but net nominal torque will be same. Now, note down the torque value T2.
- Bending effect =  $[(T2-T1)/T1]*100$ , where T1 and T2 are torque display values.
- 
- If the component of bending effect is significant for the limit of accuracy of the transducer, to proceed with the calibration may cause damage to it. Hence, do not proceed with the calibration process.
- Same shall be repeated for full load in steps of 10 %. If, found linear then.
- 
- It need not be repeated for all calibration steps. Otherwise, it has to be checked at each calibration step. Maximum effect should be taken for uncertainty calculation.

#### 4.1.3 Comparator type torque calibration machine using several reference torque transducers

- Instead of Dead weight torque calibration machine, where a calibrated reference Transducer with indicating device is used for measurement of Torque generated by suitable means either manually or with power, steps shall be taken to ensure that there is no misalignment between the torque generator, UUC, reference torque transducer and the torque absorber.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 78 of 133

- These machines are based on a number of torque transducers, individually calibrated in a dead weight torque standard machine and then loaded in parallel.
- The torque calibration machine shall have torsionally rigid couplings between the UUC and High ratio Gear box (used for rotation) on the other side along with sturdy high quality bed which can accommodate various sizes of UUC.
- Torque transducer or device shall be kept at the correct alignment with its measuring axis axial and concentric to the applied torque - at each applied torque.
- For this type of machine, the uncertainty contributions that need to be considered will include, but are not limited to, the following:
- Uncertainty of Reference Transducers.
- Uncertainty due to Repeatability (3 rotational positions at minimum and maximum of the range to take into account coupling and misalignment effects).
- Uncertainty due to resolution.

#### 4.1.4 Secondary standard Torque calibration Machine

This torque calibration machine is similar to primary torque standard machine having calibrated lever arm, except the load is applied through hydraulic system or motorized system using force proving instrument instead of dead weights. This system is normally used for calibrating torque transducers of high capacity where, dead weight system is not feasible.

For this type of machine, the uncertainty contributions that need to be considered will include, but are not limited to, the following:

- Uncertainty of reference force transducers.
- Uncertainty due to Repeatability (3 rotational positions at minimum and maximum of the range to take into account coupling and misalignment effects).
- Uncertainty of lever arm length.
- Uncertainty due to resolution of indicator.

#### 4.2 Traceability Paths

There are two distinct traceability paths for the torque generated by the torque calibration machine and the method of estimation of the associated uncertainty and evaluation of CMC depend on the following method.

- Traceability Path A:** The torque calibration machine directly from transfer standards calibrated in national force standard machine.
- Traceability Path B:** The torque calibration machine has independent traceability to the base SI units of Mass, Length and 'g' value.

#### Traceability of measurement:

All definitive measurements such as mass, 'g' value, length, temperature, humidity, atmospheric pressure which are used for realization of torque shall have traceability certificate.

Sl. No.	Type of force generating machine	Traceability certificates required for
1	Dead Weight	True Mass value, 'g' value, lever length, temperature, Humidity and atmospheric pressure
2	Comparator type with reference torque/ force transducers	Reference Torque/ Force Transducers.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 79 of 133



### 4.3 Components of Uncertainty

Components of uncertainty for Applied Torque of Calibration Machine

**Dead Weight Torque Calibration Machine:**

#### 4.3.1 Equation for realization of Torque using Dead weight and Beam

$$\text{Torque} = \text{Force} \times \text{Distance} \times (\cos \alpha)$$

$$T = F \times D \times \cos \alpha$$

$$F = m_t \times g_L \times [1 - \rho_a / \rho_m]$$

Where,

T = Torque in Newton- meter

F = Force in N

D = Nominal Length of the Lever Arm in metre

$\alpha$  = Angle

$m_t$  = True Mass used for generating the force

g = Local acceleration due to gravity in m/sec<sup>2</sup>

$\rho_a$  = Density of Air in kg/m<sup>3</sup>

$\rho_m$  = Density of Mass in kg/m<sup>3</sup>

#### 4.3.2 Formula for applying Temperature correction to the length

L =	$l_0 * (1 + \beta_T * \delta_T)$
$l_0$ =	length at calibration temperature, $T_0 = 20^\circ\text{C}$
$\beta_T$ =	Coefficient of linear expansion for the material of arm length
$\delta_T$ =	Difference between the temperature at calibration and during calibration

### Major sources of Uncertainty of Applied Torque

#### Uncertainty contribution of Force:

- Uncertainty of Mass, error in Mass, drift or stability in Mass and Mass stack.
- Uncertainty due to gravity and Uncertainty of gravity due to height of the mass stack.
- Uncertainty due to Air Density.
- Uncertainty due to Density of Mass.

**Note:** ILC may be recommended for the parameters like mass stack,  $\cos \alpha$ , and uncertainty due to height of mass stack.

#### Formula for calculation of uncertainty contribution of generated force

$$[\sigma(F)/F]^2 = [\sigma(m_t)/m_t]^2 + [\sigma(g)/g]^2 + [(\rho_a/\rho_m)^2 * ((\sigma(\rho_m)/\rho_m)^2 + (\sigma(\rho_a)/\rho_a)^2)]$$

#### Uncertainty contribution of Beam Length:

- Uncertainty of beam length  $u(l_0)$ .
- Uncertainty due to angle measurement  $u(\alpha)$ .

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 80 of 133

- Uncertainty due to thermal expansion coefficient  $u(\beta T)$ .
- Difference in temperature of the beam between calibration and usage at lab ( $\theta$ ).
- Uncertainty in temperature measurement  $u(\theta)$ .

**Formula for calculation of uncertainty contribution of Beam length:**

$$\left[ (u(l) / l)^2 \right] = \left[ (u(l_0) / l_0)^2 \right] + \left[ u^2(\alpha) \right] + \left[ \frac{\beta_T \theta}{(1 + \beta_T \theta)} \right]^2 \left\{ \left( \frac{u\beta_T}{\beta_T} \right)^2 + (u\theta / \theta)^2 \right\}$$

**Uncertainty of applied Torque is calculated as below:**

$$\left[ (u(T) / T)^2 \right] = \left[ (u(F) / F)^2 \right] + \left[ (u(l) / l)^2 \right] + \left[ u^2(\alpha) \right]$$

Where,  $u_T$  is uncertainty of Torque,  $u_F$  is uncertainty of Force,  $u_l$  is the uncertainty of length of the beam and  $u(\alpha)$  is uncertainty of cosine error.

### Validation of local 'g' and its uncertainty

Formula for calculation of Acceleration due to gravity.

An approximate value for  $g$ , at given latitude and height above sea level, may be calculated from the formula:

$g =$	$9.780\ 327 (1 + A \sin^2 L - B \sin^2 2L) - 3.086 \times 10^{-6} H m \cdot s^{-2}$
-------	-------------------------------------------------------------------------------------

Where,  $A = 0.005\ 302\ 4$ ,  $B = 0.000\ 005\ 8$ ,  $L$  = latitude,  $H$  = height in meter above sea level.

### 5 To validate this calculated 'g' value the simple steps given below can be followed:

- Find out the actual 'g' value of NMI from the certificate issued by them or by any other source.
- From the google maps click on the location of NMI, find out latitude and height above sea level. (you can know the 'g' value).
- Calculate the 'g' value using the above formula with these latitude and height. The difference between the calculated value of 'g' and the actual value of the NMI should be within 20 to 30 ppm.
- Now, go to the google maps and click on location of the lab and find out the latitude and height of the place as per google (you can know the 'g' value also).
- Calculate the 'g' value for this latitude and height. The value obtained should be within 20 to 30 ppm.
- Then this value can be taken as 'g' value of the lab and uncertainty of 'g' can be assumed to be within  $\pm 50$  ppm .

Air density should be known to sufficient accuracy depending on the required uncertainty of the applied force by measuring temperature, RH & barometric pressure.

Please refer OIML R-111-1: 2004 (page No. 76)

$$\rho_a = \frac{0.34848p - 0.009 \cdot h \cdot e^{(0.061 \cdot t)}}{273.15 + t} \quad (\text{E-3.1 OIML})$$

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 81 of 133

Where, Pressure (P) in mbar, temperature (t) in° C and humidity (h) in % Equation(E-3.1) has a relative uncertainty of  $2 \times 10^{-4}$  in the range  $900\text{hPa} < p < 1100\text{hPa}$ ,  $10\text{ }^\circ\text{C} < t < 30\text{ }^\circ\text{C}$  and  $rh < 80\%$ .

The Magnitude of vibration, Shock or other disturbing conditions shall be such that, they will have a negligible effect on the reading of standard equipment.

## 6 Recommended Calibration Interval

Reference Equipment	Recommended Interval
Dead weight Torque calibration machine Stainless steel weights (both for supported & Unsupported beam)	5 years
Dead weight Torque calibration machine Alloy steel weights (both for supported & Unsupported beam)	4 years
Comparator type with force Transducer with calibrated arm	26 Months
Comparator type with torque Transducer	1 year

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

## 7 Recommended Environment monitoring equipments at calibration laboratory:

- Temperature with a resolution of 0.1 °C.
- Humidity with a resolution of 1% RH.
- Barometer with 1 mbar

However, laboratory shall evaluate the requirement of accuracy, resolution and uncertainty depending on the CMC aimed at.

## 8 Calibration Methods

Torque Measuring Devices can be calibrated as per BS 7882:2008 either with error of indication or with error of interpolation.

## 9 Calibration Procedures

### 9.1 Resolution of the Torque Indicator:

To ensure that the classification is consistent with the resolution of the torque indicator, a lower limit of calibration shall be determined. The calibration shall not be performed below the lower limits given by the equation  $T_{\min} = ar$  where 'a' has following values and 'r' is the resolution of the indicator.

Class	Value of a
0.05	$\geq 4000 \times r$
0.1	$\geq 2000 \times r$
0.2	$\geq 1000 \times r$
0.5	$\geq 400 \times r$
1.0	$\geq 200 \times r$
2.0	$\geq 100 \times r$
5.0	$\geq 40 \times r$
Where r is the resolution	

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 82 of 133

## 9.2 Uncertainty of Applied Torque

Values for the maximum permissible uncertainty of the calibration Torque applied for the determination of different classifications of the torque measuring device shall not exceed the values given in table 1 as per BS 7882:2008. Where a reference standard is used to determine a calibration Torque it shall conform to table 1.

**Table-1:** Uncertainty of Calibration Torques (applied Torque)

Class of Torque measuring device to be Calibrated	Maximum permissible uncertainty of Calibration Torque applied <sup>(A)</sup> %
0.05	± 0.01
0.1	± 0.02
0.2	± 0.04
0.5	± 0.10
1.0	± 0.20
2.0	± 0.40
5.0	± 1.00

(A) Using a coverage factor of  $k=2$  to give a confidence level of approximately 95%.

## 9.3 Classification of the Torque Measuring Device:

- Minimum range of measurement for a selected classification shall be 20% to 100% of the calibration range.
- Where calibration torques have been applied below 20% of maximum applied torque and all of the results meet the requirement, then the range of classification of the selected classification can be extended.
- A second classification of lower class and of an extended range can be awarded, provided that all of the requirements are met in respect of lower class.

Criteria for classification of torque measuring devices as per BS7882:2008

## 10 Measurement Uncertainty

### 10.1 Uncertainty components for Calibration of UUC

- Uncertainty due to applied torque depending upon the type of machine used for calibration.
- Relative repeatability error (R1).
- Relative reproducibility error (R2).
- Relative interpolation error (Eit).
- Relative zero error (residual deflection) (R0).
- Relative reversibility error (R3).
- Relative error of indication (Ei) where interpolation is not used.
- Uncertainty due to bending effect (only for unsupported beam).

**Note:**

- The error of interpolation shall only be determined where the deflection is expressed in units other than those of torque (eg in units of Volts or mV).*
- The error indication shall only be determined where the deflection is expressed in units of torque.*

### 10.2 Overall Accuracy of the Device (Oa)

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 83 of 133

Overall accuracy of the device can be obtained by combining the classification accuracy with the expanded uncertainty in quadrature.

$$O_a = \sqrt{[(E_i/\sqrt{3})^2 + (U/2)^2]}$$

Then, overall accuracy  $O_a$  is multiplied by coverage factor  $k=2$  to give  $U_{Oa}$  the classification accuracy is obtained by  $E_i$  column in the table 2 of BS 7882.

**Note:** This applies to the calibration device having deflection in Torque units like Nm, cNm etc.

## 11 Key Points

Laboratory has to demonstrate the expanded uncertainty applied calibration force (at 95% confidence level) as per table below:

Class	Maximum permissible calibration Torque uncertainty with $k=2$ in %	Combined uncertainty with reversibility including error in %	Combined uncertainty with reversibility without error in %	Combined uncertainty without reversibility including error in %	Combined uncertainty without reversibility without error in %
0.05	0.01	0.031	0.029	0.025	0.023
0.1	0.02	0.061	0.058	0.049	0.045
0.2	0.04	0.122	0.115	0.098	0.090
0.5	0.1	0.305	0.288	0.246	0.244
1	0.2	0.610	0.575	0.492	0.448
2	0.4	1.221	1.151	0.985	0.896
5	1.0	3.052	2.877	2.462	2.241

**Note1:** Laboratory shall comply with the applied torque uncertainty required for different class as per the table above.

**Note 2:** The cut off CMC value for class 0.5 or better, for the applied torque of 0.1% beyond this accreditation shall not be granted to qualify as a reference standard for the calibration of torque generating devices.

**Note 3:** The Torque measuring device using for calibration of torque Tools as per IS 6789 & ISO 5393 requires 1 % accuracy including uncertainty (0.33 %). As per the above table the lab which can calibrate for 0.5 class or better of the torque measuring device with applied uncertainty 0.1 % or better can only qualify to carry out the calibration of Torque Tools as per the standard.

Demonstration of any CMC values doesn't automatically qualify for granting accreditation until the lab satisfies the stipulated requirement given in this document.

## 12 Recommended National/ International Standards, References and Guideline

- BS 7882 Method for calibration and classification of torque measuring devices.
- EURAMET cg 14: Static Torque measuring devices.
- ASTM E 2428 Practice for calibration of torque measuring instruments for verifying Torque indication of Torque testing machines.
- ASTM E 2624 Standard procedure for torque calibration of testing machines and devices.
- OIML R111-1 Metrological and technical requirement of weights Classes  $E_1$ ,  $E_2$ ,  $F_1$ ,  $F_2$ ,  $M_1$ ,  $M_2$ ,  $M_3$ .
- OIML D28: Conventional value of the result of weighing in air.

**Note:** - Latest versions of the relevant standard(s) should be followed

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 84 of 133

# Chapter 1(K)- Calibration of Torque Generating Devices

## 1 Recommended General Requirement

- 1.1 **Acoustic Noise** - Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 **Illumination** - The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.

## 2 Specific Requirements - Calibration of Torque Generating Devices

### Scope: Calibration of Torque Generating Devices

Sl. No.	Description	Relevant Standard	Permanent Facility	Onsite Calibration	Mobile facility
<b>I Hand Torque Tools</b>					
1	Torque Generating Devices– (Type I & II)	IS/ISO 6789	√	√	√
2	Hydraulic torque wrenches (Type –I)	IS/ISO 6789	√	√	√
3	Torque Multipliers	Comparison method	√	√	√
<b>II Rotary Torque Tools</b>					
4	Continuously rotating pneumatic, electrical hydraulic and Oil pulse tools	ISO 5393	√	√	√
5	Handheld pneumatic assembly tools	ISO 6544	√	√	√
6	Hydraulic impulse tools	ISO 17104	√	√	√
7	Torque controlled DC nut runners	ISO 5393	√	√	√
8	Impact torque wrenches/Tools	ISO 17104	√	√	√
9	Rotary and percussive pneumatic Tools	ISO 12563 (ISO 2787)	√	√	√

**Note 1:** Torque wrench tester using dead weights (mass) and 'g' is not recommended as a master for the permanent, site and mobile calibration of torque wrench (Type II) because this type of setup cannot ensure gradual application of torque from 80 % to 100 % within 0.5 s to 4 s.

**Note 2:** This technical requirement is based on the above-mentioned standards. Lab may follow any relevant standard; however care shall be taken to follow the requirements in totality.

## 3 Metrological Requirement

### 3.1 Hand torque tools

- The maximum permissible uncertainty of measurement of the calibration device shall be  $\pm 1\%$  of the indicated value including error.
- Lab is not expected to calibrate higher accuracy DUC with lower accuracy master equipment.

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 85 of 133

- 1<sup>st</sup> Peak hold mode in the display unit with minimum 20000 counts is required for type-II click type torque wrenches with a provision to measure clicking time between 0.5 s to 4 s between 80% to 100 % as per the standard either inbuilt or an external timer.

### 3.2 Rotary Torque Tools

- Performance of pneumatic tools is affected by different ambient conditions such as atmospheric pressure and temperature. Moreover the temperature of the compressed air influences the behavior of the tool. Hence, test conditions are important.
- Proper rigid fixtures and setup is required for the calibration of rotary tools with regulated input pressure or regulated electrical power input depending on the type of tool calibrated.
- Accurate measurement of the compressed air pressure to the pneumatic tool is of very great importance since the tool performance is strongly influenced by this factor.

## 4 Recommended Selection of Reference Standard

- Torque measuring device used for calibration shall be calibrated from an accredited laboratory as per BS 7882 or any other National/International standard.
- The horizontality shall be maintained between torque wrench and the torque transducer while applying the calibration torque with the help of adjustable fixtures to minimize the cosine error and other side forces. Similarly, verticality shall be maintained while calibrating screw driver type torque tools.

### 4.1 Equipment for Calibration of Torque Wrenches as per IS/ISO 6789

- Torque sensor/transducers of different capacities and digital display unit with gradual loading system for application of torque for Type –I indicating torque tools.
- Mechanical Torque tester with Dead weight and graduated lever and Fulcrum for calibration of Type –I torque tools only as per IS/ISO 6789.
- Torque sensor/transducers of different capacities and digital display unit for Type-II setting torque tools. The display unit shall have facility to read torque in Normal, first peak and Peak mode with minimum 20000 counts. Rigid fixture for gradual application of load and to satisfy 0.5 s to 4s from 80% to final target value of the torque. The display unit may also have built-in timer to measure this clicking time, alternatively external timer may also be used. The traceability for timer may not be required as it is not a precision measurement and the span is quite large. Accuracy for the timer can be verified with other available timer / stop watch.
- The maximum permissible uncertainty of measurement of the calibration device shall be  $\pm 1\%$  of the indicated value including error. For e.g. 100 to1000 Nm calibration of the measurement uncertainty including error shall be 1 % at 100 Nm as well as 1000 Nm (in % of rdg, not in FSD).

### 4.2 Equipment for Calibration of Hydraulic Torque Wrench as per IS/ISO 6789

- Reaction torque sensor of various capacities.
- Digital Display unit to measure torque with peak hold facility. The accuracy including uncertainty of the transducer and the amplifier within  $\pm 1\%$  of the test torque level.
- Rigid test fixture to mount reaction torque transducer and hydraulic torque wrench under calibration.
- Hydraulic power pack supplied with Hydraulic torque Wrench.

### 4.3 Equipment for Calibration of Torque Multiplier by Comparison Method

- Digital Torque Indicator and Torque Transducer for input torque measurement.
- Digital Torque Indicator and Torque Transducer for output torque measurement.
- Torque Multiplier calibration rigid fixture with reaction plate for the output side and high ratio gear box having zero backlash with suitable couplings for the application of torque via input torque transducer.

### 4.4 Equipment for Calibration of Rotary Torque Tools like Continuously Rotating, Pneumatic, Electrical and Hydraulic tools including D.C. Nut runners as per ISO 5393

- Rotary Torque sensor (or reaction torque sensor)

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 86 of 133

- Digital display unit with peak hold facility. The accuracy of the transducer and the amplifier within  $\pm 1\%$  of the test torque level and frequency response shall be -3dB at 500 Hz, with a roll off of 50 dB per decade.
- Pressure gauge.
- Rigid test fixture with provision of soft joint and hard joint.
- Air compressor with filter and pressure regulator (for pneumatic tools).
- Regulated A.C. power supply (for Electrical Tools).
- D.C. Power supply (supplied with the tool)
- Accessories like hose pipes, adapters etc.

#### 4.5 Equipment for Calibration of Handheld Pneumatic Assembly Tools as per ISO 6544

- Rotary Torque Transducer
- Digital display unit with peak hold facility, it should have frequency response of  $\pm 1\%$ .
- From 0-1000 Hz.
- Angle sensor with display unit having resolution of  $1^\circ$
- Fast recording device to plot Torque Vs time and Torque Vs Angle.
- Rigid test fixture with soft and hard joint assembly.
- Pressure Gauge.
- Air compressor with filter and pressure regulator.
- Accessories like hose pipes, adapters etc.

#### 4.6 Equipment for Calibration of Hydraulic impulse tools as per ISO/TS 17104

- Rotary torque angle sensor with display unit having analog or digital output for both torque and angle (accuracy and frequency response should comply with ISO 5393 for torque measurement).
- Clamp force sensor with display unit having analog or digital output with peak hold facility. The repeatability of the device should be  $\pm 1\%$  of the test clamp force level. The frequency response of the clamp force device and amplifier shall be -3dB at 500 Hz, with a roll off of at least 50dB per decade.
- Digital Display unit to measure impulse/min and speed.
- Rigid test fixture to hold tool and the rotary torque sensor
- Digital Pressure gauge.
- Air compressor with filter and pressure regulator.
- Accessories like hose pipes, adapters etc.

#### 4.7 Equipment for Calibration of Impact Wrenches as per ISO/TS 17104

- Reaction torque sensor of various capacities.
- Digital Display unit to measure torque, impacts/min, speed with peak hold facility.
- Rigid test fixture to mount reaction torque transducer and impact wrench under calibration.
- Speed measuring set up.
- Hard & soft Joints.
- Air compressor with filter and pressure regulator.
- Accessories like hose pipes, adapters etc.,

### 5 Recommended Calibration Interval

Reference Equipment	Recommended calibration Interval
Torque measuring system used for calibration of hand torque tools and rotary torque tools	1 year

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 87 of 133



## 6 Recommended Environmental conditions required for calibration and requirement of environmental monitoring system

### 6.1 For Hand Torque Tools as per IS/ISO 6789

- The ambient temperature shall be maintained in the range of 18°C to 28°C. The temperature shall not vary more than  $\pm 1^\circ\text{C}$  throughout a measurement series.
- Maximum relative humidity 90% RH.

### 6.2 Recommended Environment Monitoring Equipment

- Temperature with a resolution of  $1^\circ\text{C}$
- Humidity with a resolution of 1% RH

However, laboratory shall evaluate the requirement of accuracy, resolution and uncertainty depending on the CMC aimed at.

### 6.3 For all Rotary Torque Tools

The test conditions should be in the range of values given below:

- Atmospheric pressure  $960 \pm 100$  mbar.
- Ambient temperature  $20^\circ\text{C} \pm 2^\circ\text{C}$ .
- Compressed air temperature  $20^\circ\text{C} \pm 5^\circ\text{C}$ .
- In general, pneumatic tools shall be tested at an effective (gauge) Air pressure of  $6.3 \pm 0.15$  bar. If the tool has been designed for a different pressure (for example 4 bar) this may be used and shall be stated in the test report. The working pressure shall be maintained under all the test conditions.

## 7 Calibration Procedure

Calibration of Hand Torque Tools follow the procedure as per IS/ISO 6789.

## 8 Measurement Uncertainty

### 8.1 Uncertainty components to be considered but not limited to the following:

- Uncertainty of the reference torque tester/calibrator (sensor).
- Standard deviation of the repeated readings.

**Note 1:** *Uncertainty contribution due to input and output torque measuring device to be considered for the calibration torque multiplier.*

**Note 2:** *Uncertainty contribution from pressure measuring device for pneumatic/ hydraulic tools and electrical input for electrical tools has to be considered wherever applicable.*

## 9 Recommended National/ International Standards, References and Guideline

- IS/ ISO 6789: Assembly tools for screws and Nuts – Hand Torque tools requirements and test methods for design conformance testing, quality conformance Testing and recalibration procedure.
- ISO 5393 Rotary tools for threaded fasteners-performance test method.
- ISO/TS 17104 Rotary Tools for threaded fasteners – Hydraulic impulse tools performance test method
- ISO 6544 Hand-held pneumatic assembly tools for installing threaded fasteners- Reaction torque and torque impulse measurements
- IS 12563 (ISO 2787) Rotary and percussive pneumatic Tools- performance test

**Note:** - Latest versions of the relevant standard(s) should be followed

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 88 of 133

# Chapter 1(L): Calibration of Pressure Balance/Dead Weight Tester

## 1 Recommended General Requirement

- 1.1 Acoustic noise level in the laboratory shall be maintained to facilitate proper performance of calibration work. Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 The calibration area shall have adequate level of illumination. Where permissible, fluorescent lighting is preferred to avoid localized heating and temperature drift. The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.
- 1.6 The ambient temperature shall be between 15 °C to 25 °C. The temperature shall not vary more than  $\pm 1^\circ\text{C}$  / hour during measurement.
- 1.7 It is preferable to maintain Relative humidity 60% RH  $\pm$  20% RH.

## 2 Recommended Requirements – Calibration of Pressure Generating Device

### 2.1 Scope: Calibration of Pressure Generating Device (Dead Weight Tester)

Requirements for the calibration of Pressure Generating Devices with following details:

Sl. No	Description	Relevant Standard/ Guidelines	Permanent Facility	Onsite Calibration
1.	Hydraulic Dead Weight Testers/Pressure Balance	EURAMET cg-3	√	X
2.	Pneumatic Dead Weight Testers/Pressure Balance		√	X
3.	Vacuum/Absolute Dead Weight Testers/ Pressure Balance		√	X

**Important Note:** This technical requirement is based on the above-mentioned guideline. Lab may follow any relevant standard; however, care shall be taken to follow the requirements in totality.

## 3 Metrological Requirement

- Unit of measurement of pressure is pascal, Pa SI Unit of measurement of pressure is Pascal, (Pa)
- Pressure gauges, vacuum gauges, Pressure-Vacuum gauges are to be calibrated in Pa, kPa, MPa, GPa, as per SI units. However, Units like bar and mbar, may also be used.
- All weights shall be traceable in SI Units for deriving Pressure in Pascal or bar.
- For Each weight, the expanded uncertainty, U, for k=2, of the true mass.
- Preferably all weights shall be equivalent or better than F2 standard as per OIML R-111-1.
- 'g' value shall be known with sufficient accuracy either by Geological Survey of India or any other relevant source for finer CMC.
- Laboratory may also calculate 'g' value knowing latitude and height as per the formula. However, same shall be validated (as per 2.8.2.1 & 2.8.2.2).
- A suitable air buoyancy correction shall be applied if the weight are calibrated either by conventional basis or by true mass basis.
- Knowing the true mass, piston cylinder area value and 'g' value, Pressure value will be determined after applying buoyancy correction.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 89 of 133

- When the masses are submitted to vacuum, the balance measures an absolute pressure. The residual pressure in the bell jar around the masses creates a force in opposition to the measured pressure. The residual pressure has to be measured and added to the pressure created by the masses.
- When overall masses are submitted to the atmosphere which also applies to the top of the piston, the balance measures the gauge pressure.

In some cases, an adopter allows the reversal of the piston cylinder mounting, the balance then measures the negative gauge pressure (below atmospheric pressure) and generates an upward force opposed to the gravitational force.

**Accuracy Class** - Pressure balances are classified into the following accuracy classes 0.005, 0.01, 0.02, 0.05, 0.1, 0.2. The accuracy class of a pressure balances shall be determined by calibration.

#### 4 Selection of Reference Equipment

Reference Standard Pressure generating system

##### 4.1 Hydraulic Dead Weight Tester

Accessories:

- Proper connecting pipes and adopters.
- Float level indicators for both reference and DUC.
- Suitable arrangements to measure temperatures both of the PCU using thermometers/PRTs in close proximity to PCUs.
- Preferably constant volume valve or equivalent device can be used.

##### 4.2 Pneumatic Dead Weight Tester

Accessories:

- Proper connecting pipes and adopters.
- Float level indicators for both reference and DUC.
- Suitable arrangement to measure temperatures of both the PCUs using thermometers /PRTs in close proximity to PCUs.
- Vacuum pump (if negative calibration is done).
- Gas supply.

##### 4.3 Absolute Pressure Dead Weight Tester (for Vacuum and Absolute Pressure)

Accessories:

- Proper connecting pipes and adopters.
- Vacuum generating pump.
- Float level indicators for both reference and DUC.
- Suitable arrangement to measure temperatures of both the PCUs using thermometers / PRTs in close proximity to PCUs.
- Gas supply
- 

**Note:** The reference balance should have better or at least equal uncertainty or the pressure balance under calibration

#### 5 Calibration Interval

Reference Equipment	Recommended interval
Dead Weight Tester	2 years

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

#### 6 Legal Aspects

Calibration of Pressure Dead weight testers done by any accredited laboratories is meant for scientific and industrial purpose only. However, if used for commercial trading, additional recognition/ approval shall be

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 90 of 133

complied as required by Dept. of Legal metrology, Regulatory bodies, etc. This should be clearly mentioned in the calibration certificate issued to the customer.

## 7 Effect of Gravity "g" on Calibration

'g' value shall be known to sufficient accuracy. It is very important to establish the gravitational value of the laboratory since it is one of the major quantity during realization of force. The effect of not doing this could be a variation in force produced by the weight perhaps 0.5% of the force. It is therefore recommended that; the Pressure calibration laboratory establishes local value of gravity (g) and use weights that have been calibrated at that gravitational constant.

### 7.1 Effect of gravity 'g' on calibration, when Dead Weight Testers are used

For measurement uncertainty of applied force, 'g' value shall be known. For realization of applied force more than 0.01%, 'g' value shall be calculated using the formula given in below for better than 0.005%, 'g' value shall be measured by appropriate authority.

### 7.2 Validation of local 'g' and its Uncertainty

Formula for calculation of Acceleration due to gravity.

An approximate value for  $g$ , at given latitude and height above sea level, may be calculated from the formula:

$$g = 15.17 (1 + A \sin^2 L - B \sin^2 2L) - 3.086 \times 10^{-6} H m \cdot s^{-2}$$

Where,  $A = 0.0053024$ ,  $B = 0.0000058$ ,  $L$  = latitude,  $H$  = height in meter above sea level.

To validate this calculated 'g' value the simple steps given below can be followed:

- Find out the actual 'g' value of NMI from the certificate issued by them or by any other source.
- Find out the actual 'g' value of NMI from the certificate issued by them or by any other source.
- From the web search engine maps click on the location of NMI, find out latitude and height above sea level. (You can know the 'g' value).
- Calculate the 'g' value using the above formula with these latitude and height. The difference between the calculated value of 'g' and the actual value of the NMI should be within 20 to 30 ppm.
- Now, go to the web search engine maps and click on location of the lab and find out the latitude and height of the place as per web search engine (you can know the 'g' value also)
- Calculate the 'g' value for this latitude and height. The value obtained should be within 20 to 30 ppm.
- Then this value can be taken as 'g' value of the lab and uncertainty of 'g' can be assumed to be within  $\pm 50$  ppm.

## 8 Effect of Temperature

Piston cylinder of the pressure balance is temperature sensitive and must, therefore, be corrected to a common temperature datum. Variation in the indicated pressure resulting from changes in temperature arises from the change in effective area of the piston due to the expansion or contraction caused by temperature changes. The solution is a straight forward application of the thermal coefficients of the material of the piston and cylinder. Hence the effect of temperature is critical in pressure realization and correction for the area is to be done.

## 9 Estimation of Air Density

Air density should be known to sufficient accuracy depending on the required uncertainty of the applied force by measuring temperature, RH & barometric pressure.

(Approximation formula as per OIML R-111-1: 2004 pg. No. 76)

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 91 of 133

$$\rho_a = \frac{0.34848p - 0.009 \cdot h \cdot e^{(0.061 \cdot t)}}{273.15 + t} \quad (\text{E3.1 OIML})$$

Where, Pressure (P) in mbar, temperature (t) in ° C and humidity (h) in % Equation(E-3.1) has a relative uncertainty of  $2 \times 10^{-4}$  in the range  $900\text{hPa} < p < 1100\text{hPa}$ ,  $10^\circ \text{C} < t < 30^\circ \text{C}$  and  $\text{rh} < 80\%$

## 10 Calibration Methods

Cross float method applies to both hydraulically and pneumatically operated balances. In both cases the method is a comparative one. Consisting of comparing the balance to be calibrated and the standard instrument both are subjected to same pressure and same environmental conditions is also called as cross floating method.

### 10.1 Method A - Pressure Generated Method

The scope of this method is to determine the bias error and the repeatability of the Calibrated pressure balance. This is done by determining the generated pressure corresponding to well-identified weights and effective area of the reference standard. In this method the weighing of the masses of the instrument under calibration is optional. This method is not employed where the smallest uncertainty is required.

### 10.2 Method B - Effective Area Determination Method

This method determines the mass of the piston, weights of the balance and effective area of the Piston cylinder assembly. This method is employed where the smallest uncertainty of better than 0.01% is required.

The scope of this method is to determine:

The value of the mass of all the weights, including the piston of the pressure balance, if removable.

- The effective area  $A_p$  referred to  $20^\circ \text{C}$  or another reference temperature  $t_r$  of the piston-cylinder assembly of the pressure balance as a function of pressure. At high pressure, this area can be expressed from the effective area at null pressure  $A_0$  and the pressure distortion coefficient.
- Repeatability as a function of the measured pressure.

## 11 Calibration Procedure and Measurement Uncertainty

### Method A (Pressure Generation Method)

- For cross floating the reference and test dead weight testers (pressure balance) are to be connected with suitable connections so that, there is no leakage (either hydraulic or pneumatic)
- Three series of increasing and decreasing pressures are applied from 10 % to 100% of the calibration range.
- The serial numbers of weights placed on both reference and the device under calibrations are noted along with the temperature of both the piston cylinder unit.
- Since both the pistons are balanced, the pressure generated will be equal. The generated pressure with reference to the reference is calculated as both mass and area are known.

## 12 Equation for Calculation

### 12.1 Generated Pressure for a Hydraulic DWT using true mass

$$p_e = \frac{[\sum_i m_i \cdot (1 - \rho_a / \rho_m)] \cdot g + \sigma_c}{A_0 (1 + \lambda \cdot p) [1 + (\alpha_p + \alpha_c) \cdot (t - t_r)]} (+) \rho_f \cdot g \cdot \Delta h \quad (\text{A-1})$$

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 92 of 133

$p_e =$	Pressure in pascal (at a calibration step)
$g =$	Local acceleration due to gravity in $m/s^2$
$\rho_a =$	Density of Air in $kg/m^3$
$\rho_m =$	Density of mass in $kg/m^3$
$\rho_{m1} =$	Density of mass of PCU in $kg/m^3$
$A_0 =$	Effective area of the Piston Cylinder Assembly (PCU) at reference temperature and zero pressure in $m^2$
$\lambda =$	Pressure distortion co-efficient of Piston Cylinder Assembly (PCU) in /pa
$\alpha =$	Linear thermal expansion coefficient of the piston in / °C
$\sigma =$	Surface tension of the oil in N/m
$c =$	Circumference of emergent piston in m
$\rho_f =$	Density of Hydraulic Fluid in $kg/m^3$
$p =$	Applied Nominal Pressure in pa

$\Delta h$  = Difference between height  $h_1$  of the reference level of the balance and the height  $h_2$  of the balance under calibration:  $\Delta h = h_1 - h_2$  in m [ $\Delta h$  is +ve if, the unit under test is below the reference PCU level and -ve if, it is above the reference PCU level]

**Note 1:** In case of Pressure generated from a pneumatic dead weight tester, Fluid head correction factor ( $\rho_f \cdot g \cdot \Delta h$ ) may be considered negligible.

**Note 2:** In case of Pressure generated from a pneumatic dead weight tester, pressure distortion coefficient ( $\lambda$ ) may be considered negligible if not reported in the certificate.

## 12.2 Generated Pressure for a Hydraulic DWT using conventional mass

$$P_e = \frac{[\sum_i m_{ci} * (1 - (\rho_{0a}/\rho_0) + (\rho_{0a} - \rho_a)/\rho_m)] * g + \sigma c}{A_0 (1 + \lambda * p) [1 + (\alpha_p + \alpha_c) * (t - t_r)]} (+) \rho_f \cdot g \cdot \Delta h \quad (A-2)$$

Where,  $m_{ci}$  = individual conventional mass value of each applied weight on the piston  
 $\rho_{0a} = 1.2 \text{ kg/m}^3$ , conventional value of air density  
 $\rho_0 = 8000 \text{ kg/m}^3$ , conventional value of mass density  
 Other quantities are as referred above.

## 12.3 Generated absolute Pressure for a Pneumatic DWT using true mass

$$p_{abs} = \frac{[\sum_i m_i * (1 - \rho_a/\rho_m)] * g}{A_0 [1 + (\alpha_p + \alpha_c) * (t - t_r)]} + \mu \quad (A-3)$$

Where,  $p_{abs}$  is the absolute pressure measured at the bottom of the piston  
 $m_i$  = individual true mass value of each applied weight on the piston,  
 $\mu$  = is the residual pressure surrounding weights  
 Other quantities are as referred above.

## 13 Measurement Uncertainty

### 13.1 Estimation of type A Uncertainty

Standard deviation of Repeatability of the balance for estimated pressure.

### 13.2 Estimation of type B Uncertainty (Method A):

#### Components associated with applied pressure

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 93 of 133

- a) **Uncertainties associated with Mass of Reference Dead Weight Tester(u1)**
  - Mass placed on the carrier [including piston]
  - Mass instability in time
  - Cross floating sensitivity in terms of masses
  - Local acceleration due to gravity "g"
  - Air Density
  - Mass Density
  
- b) **Uncertainties associated with Effective area of PCU at Atmospheric Pressure and Reference Temperature (u2)**
  - Piston Circumference
  - Surface Tension of the fluid (for hydraulic only)
  - Drift in effective area
  - Pressure distortion coefficient
  - Thermal expansion coefficient of piston
  - Thermal expansion coefficient of cylinder
  
- c) **Other associated Uncertainties (u3)**
  - Variation of Temperature of Reference PCU during calibration
  - Head Correction (Level)
  - Fluid Density(for hydraulic only)
  - Verticality of Piston

**Total B type uncertainty**

$$u_B = \sqrt{u_1^2 + u_2^2 + u_3^2}$$

**Combined Uncertainty for Gauge Calibration**

$$u_c = \sqrt{u_A^2 + u_B^2}$$

**Expanded Uncertainty U = k x uc**

Where, k= coverage factor corresponding to the effective degree of freedom

**Reporting of Results (Method A)**

The certificate of calibration should be issued with Pressure values converted to standard 'g' 9.80665m/s<sup>2</sup> and reference temperature of 20 °C (if or otherwise not specified 'g' value and temperature value on the dead weight tester or requested by the user).

The following equation should be given to enable the customer to convert the pressure to the local 'g' value ( at the lab) and the reference temperature

$p_e =$	$p_s * g_L * [1+(t-20)*\alpha]$
	9.80665

Where,'t' is the temperature during calibration and gravity 'gL' local acceleration due to gravity (g' value of the lab) and 'α' is the linear thermal expansion coefficient of the Piston / °C.

**14 Calibration Procedure Measurement Uncertainty**

**14.1 Method –B (Effective Area Method)**

Pressure generated by the reference dead weight tester is calculated as its area and mass applied is known at each point of calibration [using the equation A-1, A-2, A-3 depending on the case].

$$p_e = \left[ \sum_i m_i * (1 - \rho_a / \rho_m) \right] * g + \sigma_c \quad (+) \rho_f * g * \Delta h \quad (A-1)$$

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 94 of 133

$$A_0(1 + \lambda * p) * [1 + (\alpha_p + \alpha_c) * (t - t_r)]$$

- $p_e$  = Pressure in Pascal (for each calibration point)
- $g$  = Local acceleration due to gravity in  $m/s^2$
- $\rho_a$  = Density of Air in  $kg/m^3$
- $\rho_m$  = Density of mass in  $kg/m^3$
- $\rho_{m1}$  = Density of mass of PCU in  $kg/m^3$
- $A_0$  = Effective area of the Piston Cylinder Assembly (PCU) at reference temperature and zero
- $\lambda$  = Pressure distortion co-efficient of Piston Cylinder Assembly (PCU) in /pa
- $\alpha$  = Linear thermal expansion coefficient of the piston in/ °C
- $\sigma$  = Surface tension of the oil in N/m
- $c$  = Circumference of emergent piston in m
- $\rho_f$  = Density of Hydraulic Fluid in  $kg/m^3$
- $p$  = Applied Nominal Pressure in pa

$\Delta h$  =Difference

between height  $h_1$  of the reference level of the balance and the height  $h_2$  of the balance under calibration:  $\Delta h = h_1 - h_2$  in m [ $\Delta h$  is + ve if, the unit under test is below the reference PCU level and -ve if, it is above the reference PCU level]

**Note 1:** In case of Pressure generated from a pneumatic dead weight tester, Fluid head correction factor ( $\rho_f \cdot g \cdot \Delta h$ ) may be considered negligible.

**Note-2:** In case of Pressure generated from a pneumatic dead weight tester, pressure distortion coefficient ( $\lambda$ ) may be considered negligible if not reported in the certificate.

**Note 3:** Two to three series of measurement for five to seven points equally divided through the range should be carried out.

Now the Effective area of the device under calibration, at the calibration temperature and the generated pressure from the reference is calculated using the below given equation

$$A_{2t} = \frac{[\sum_i m_{i2} * (1 - \rho_a / \rho_{mi2})] * g}{P_{1i} * [1 + (\alpha_p + \alpha_c) * (t - t_r)]} \quad (B-1)$$

Where,

$A_{2t}$  is the effective area in  $m^2$  of the Test PCU at reference temperature

$m_2$  is the mass applied on the Test PCU and  $\rho_{mi2}$  density of the mass of test PCU

$p_{1i}$  is the calculated pressure at reference temperature at a calibration point

By plotting a graph of applied pressure against the calculated area for each calibration point, we can calculate the effective area  $A_0$  at zero pressure for the test PCU and the pressure distortion constant along with their uncertainties.

## 14.2 Estimation of Uncertainty for Method B (Area Method)

### Estimation of type A Uncertainty ( $u_A$ components):

Repeatability of the balance, estimated as a function of pressure from the values of the standard deviation of the effective area as expressed in the table in 6.3.3. Alternatively, the type A uncertainty of pressure can be presented by an equation based on the variances and covariance of  $A_0$  and  $\lambda$ .

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 95 of 133



**Estimation of type B Uncertainty (uB components):**

- Uncertainty of the reference pressure;
- Uncertainty of the masses;
- Uncertainty due to the temperature of the balance;
- Uncertainty due to the thermal expansion coefficient of the piston-cylinder assembly;
- Uncertainty due to the air buoyancy;
- Uncertainty due to the head correction;
- Uncertainty due to the surface tension of the pressure-transmitting fluid;
- Uncertainty due to tilt (negligible if perpendicularity was duly checked); Uncertainty due to spin rate and/or direction, if applicable
- Uncertainty of the residual pressure (absolute mode only).

**Reporting of Results (Method B)**

- A certificate of individual weights along with their uncertainty either for true mass or for conventional as per the customer's requirement shall be issued.
- The calibration certificate shall be issued with the effective area at zero pressure (A0) and pressure distortion coefficient with their uncertainties.
- Calibration certificate should report- values same as given in Clause 2.10.6.5 and overall expanded uncertainty for the device.

**Note:** For Area/Mass method scope should also be in % of reading (Pressure).

**15 Recommended National/ International Standards, References and Guidelines**

- EURAMET cg-3, Version 1.0 –“Calibration of pressure balances”(Guidelines).
- OIML R 110 “Pressure balances”
- OIML R-111-1,edition 2004 Metrological and technical requirement of weights
- OIML D28 Conventional value of the result of weighing in air

**Note:** - Latest version of the relevant standard(s) should be followed

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129		Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)		
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 96 of 133

# Chapter 1(M): Calibration of Pressure Indicating Devices

## 1 Recommended General Requirement

- 1.1 Acoustic noise level in the laboratory shall be maintained to facilitate proper performance of calibration work. Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 The calibration area shall have adequate level of illumination. Where permissible, fluorescent lighting is preferred to avoid localized heating and temperature drift. The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.
- 1.6 **For Pneumatic and Hydraulic Pressure Measurements:** During calibration temperature shall be 18°C to 28°C. Preferably 23°C  $\pm 1.5^\circ\text{C}$  should not change more than 1°C during calibration.
- 1.7 **For Vacuum Pressure (as per Standard ISO 27893):** During calibration temperature shall be 20°C to 26°C. Preferably 23°C  $\pm 1.5^\circ\text{C}$  should not change more than 1°C during calibration.
- 1.8 Temperature measuring instrument shall have an expanded uncertainty  $\leq 0.5^\circ\text{C}$  at k=2.
- 1.9 ‘

## 2 Recommended Requirements: Calibration of Pressure Indicating Devices

### 2.1. Recommended Facility of Scope:

Sl. No	Description	Relevant Standard/ Reference	Permanent Facility	Onsite Calibration	Mobile Facility
1.	Precision Pressure Calibrator with pump (digital and Analog)	OIML R-101 OIML R-97 (Barometer) EURAMET Cg-17, Version 2.0 (03/2011) / DKD R-6-1 IS 3624	√	√	√
2.	Pressure transducer with digital pressure indicator		√	√	√
3.	Pressure transducer with Voltage, current or frequency output		√	√	√
4.	Pressure transmitters with Voltage (5 V,10V....) , Current (4-20mA,0-20mA,...) Frequency Digital format (RS232...)		√	√	√
5.	Industrial Pressure gauges with analog/digital indication.		√	√	√
6.	Barometer		√	√	√
7.	Oxygen Pressure gauges*		√*	X	X
8.	Pressure Switches		√	√	√
9.	Magnahelic Gauge, Pirani Gauge		√	√	√
10.	Vacuum Gauge		ISO 3567 ISO 27893	√	√

\* Special separate arrangements shall be required when calibrating oxygen pressure gauge.

**Note 1:** Onsite and Mobile calibration with Dead weight testers as a master is not recommended.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 97 of 133

**Note 2:** This technical requirement is based on the above-mentioned guideline. Lab may follow any relevant standard; however care shall be taken to follow the requirements in totality.

### 3 Metrological Requirement

#### Pressure Gauges to use with oxygen & Acetylene

- Oxygen under pressure forms an explosive mixture with oil or grease, and a serious explosion may result if the two are brought together. When Oxygen gauges are calibrated Oil and grease should not be allowed to touch or enter the gauge. They should be tested only with dry and clean air and used for that purpose alone, and no other gauges should be calibrated on this equipment to avoid the risk of oil contamination (refer appendix C clause 9.1.5 of IS 3624: 1987, RA 2004).
- Acetylene in conjunction with copper form an explosive compound .Care shall be taken during calibration.
- SI Unit of measurement of pressure is Pascal, (Pa).
- Pressure gauges, vacuum gauges, Pressure-Vacuum gauges are to be calibrated in Pa, kPa, MPa, GPa, as per SI units. However, Units like bar and mbar, may also be used.
- 'g' value shall be known with sufficient accuracy either by Geological Survey of India or any other relevant source for finer CMC.
- Laboratory may also calculate 'g' value knowing latitude and height as per the formula in annexure A. However, same shall be validated.
- A suitable air buoyancy correction shall be applied if the weights are calibrated either by conventional basis or by true mass basis.
- Knowing the true mass, piston cylinder area value and 'g' value, Pressure value will be determined after applying buoyancy correction.
- The reference equipment used for calibration of pressure gauges or electro manometer should be such that, its accuracy including uncertainty better than 1/3 of the accuracy class of the device under calibration.
- The laboratory is not recommended to calibrate the higher accuracy DUC with lower accuracy reference equipment.

### 4 Recommended Selection of Reference Equipment

Make sure that the reference equipment used for calibration of pressure gauges or electro manometer should be such that, its accuracy including uncertainty better than 1/3 of the accuracy class of the device under calibration.

### 5 Calibration Interval

Laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

### 6 Legal Aspects

Calibration of Pressure gauge done by any accredited laboratories is meant for scientific and industrial purpose only. However, if used for commercial trading, additional recognition/ approval shall be complied as required by Dept. of Legal metrology, Regulatory bodies, etc. This should be clearly mentioned in the calibration certificate issued to the customer.

#### 6.1.1 Effect of gravity "g" on calibration when Dead weight testers are used.

- 'g' value should be known to sufficient accuracy.
- It is very important to establish the gravitational value of the laboratory since it is one of the major quantity during realization of force. The effect of not doing this could be a variation in force produced by the weight perhaps 0.5% of the force. It is therefore recommended that, the Pressure calibration laboratory establishes local value of gravity (g) and use weights that have been calibrated at that gravitational constant.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 98 of 133

- For measurement uncertainty of applied force, 'g' value shall be known. For realization of applied force more than 0.01%, 'g' value shall be calculated using the formula given in below For better than 0.005%, 'g' value shall be measured by appropriate authority.
- Validation of local 'g' and its uncertainty
- Formula for calculation of Acceleration due to gravity

An approximate value for  $g$ , at given latitude and height above sea level, may be calculated from the formula:

$g =$	$9.780 \quad 7 (1 + A \sin^2 L - B \sin^2 2L) - 3.086 \times 10^{-6} H m \cdot s^{-2}$
-------	----------------------------------------------------------------------------------------

Where,  $A = 0.005 \ 302 \ 4$ ,  $B = 0.000 \ 005 \ 8$ ,  $L$  = latitude,  $H$  = height in meter above sea level

### 6.1.2 To validate this calculated 'g' value the simple steps given below can be followed:

- Find out the actual 'g' value of NMI from the certificate issued by them or by any other source.
- Find out the actual 'g' value of NMI from the certificate issued by them or by any other source.
- From the maps click on the location of NMI, find out latitude and height above sea level. (you can know the 'g' value).
- Calculate the 'g' value using the above formula with these latitude and height. The difference between the calculated value of 'g' and the actual value of the NMI should be within 20 to 30 ppm.
- Now, go to the maps and click on location of the lab and find out the latitude and height of the place as per the location (you can know the 'g' value also).
- Calculate the 'g' value for this latitude and height. The value obtained should be within 20 to 30 ppm.
- Then this value can be taken as 'g' value of the lab and uncertainty of 'g' can be assumed to be within  $\pm 50$  ppm.

## 6.2 Estimation of Air Density

Air density should be known to sufficient accuracy depending on the required uncertainty of the applied force by measuring temperature, RH & barometric pressure.

(Approximation formula as per OIML R-111-1: 2004 pg. No. 76)

$$\rho_a = \frac{0.34848p - 0.009 \cdot h \cdot e^{(0.061 \cdot t)}}{273.15 + t} \quad \text{(E-3.1 OIML)}$$

Where, Pressure (P) in mbar, temperature (t) in ° C and humidity (h) in % Equation(E-3.1) has a relative uncertainty of  $2 \times 10^{-4}$  in the range  $900 \text{hPa} < p < 1100 \text{hPa}$ ,  $10^\circ \text{C} < t < 30^\circ \text{C}$  and  $rh < 80\%$

## 7 Recommended Calibration Methods

### Pressure gauges can be calibrated with one of the following methods:

- Using Dead weight tester by calculating the actual pressure generated with the help of area of the piston, local 'g' and applied known mass while comparing.
- Using Dead weight tester by comparison method using the nominal pressure values mentioned on the pressure weights of the Dead weight tester.
- Using Digital Pressure Calibrator.
- By comparison method using a Digital /analog pressure gauge and pressure generating system-  
Method I: Reading as per set on DUC  
Method II: Reading as per set on standard

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 99 of 133

## 8 Calibration Procedure & Measurement Uncertainty

### 8.1 Recommended calibration sequences based on their accuracy:

Calibration Sequence	Measurement Uncertainty aimed at in % of the meas. Span (*)	Number of meas. Points with zero up/down	Number of press. loadings	Load change +waiting time sec. (**)	Wafting time at upper limit of meas. Range minutes (***)	Number of meas. Series	
						Up	Down
A	< 0.1	9	3	>30	2	2	
B	0.1.....0.6	9	2	>30	2	1	
C	> 0.6	5	1	>30	2	1	

(\*) Reference to span is used to allow the sequence to be selected from the table as the accuracy specifications of the manufacturers are usually related to the measurement span.

(\*\*) One has to wait in any case until steady-state conditions are reached.

### Calibration of Industrial Pressure Gauges by using Dead Weight Tester and calculating the actual pressure generated at each point

1. After noting down the applied mass, temperature at 5 calibration point of the steps and series as per the above referred table (8.10.1), the pressure generated to balance the UUT can be calculated using the pressure formula given below:

Equation to calculate the Generated Pressure from a Dead Weight Tester:

$$P_e = \frac{[\sum_i m_i * (1 - \rho_a / \rho_{mi})] * g + \sigma c}{A_0 (1 + \lambda * p) * [1 + (\alpha_p + \alpha_c) * (t - t_r)]} \quad (+) \rho_f \cdot g \cdot \Delta h \quad (A-1)$$

g =	Local acceleration due to gravity in m/s <sup>2</sup>
$\Omega \rho_a$ =	Density of Air in kg/m <sup>3</sup>
$\rho_m$ =	Density of mass in kg/m <sup>3</sup>
$\rho_{m1}$ =	Density of mass of PCU in kg/m <sup>3</sup>
$A_0$ =	Effective area of the Piston Cylinder Assembly (PCU) at reference temperature and zero pressure in m <sup>2</sup>
$\lambda$ =	Pressure distortion co-efficient of Piston Cylinder Assembly (PCU) in /pa
$\alpha$ =	Linear thermal expansion coefficient of the piston in/ °C
$\sigma$ =	Surface tension of the oil in N/m
c =	Circumference of emergent piston in m
$\rho_f$ =	Density of Hydraulic Fluid in kg/m <sup>3</sup>
p =	Applied Nominal Pressure in pa

**Note 1:** In case of Pressure generated from a pneumatic dead weight tester, Fluid head correction factor ( $\rho_f \cdot g \cdot \Delta h$ ) may be considered negligible and pressure distortion correction ( $1 + \lambda \cdot p$ ) may be considered negligible if not reported in the certificate

**Note 2:** In case of absolute pressure measurements, the atmospheric pressure should be added to the generated pressure. The uncertainty component of atmospheric pressure measuring device should also be included while estimating the measurement uncertainty of calibration.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 100 of 133

- **Calibration of Industrial pressure gauges by using Dead Weight Tester and Reference Pressure Value**

1. Check the calibration certificate of the Dead weight tester whether pressure value is given for standard 'g' value 9.80665 m/s<sup>2</sup> or local 'g' value (corrected to the lab 'g' value). If the pressure values mentioned is for standard 'g' value then convert them to the local 'g' value using the following equation for accurate measurements.
2. Also add the uncertainty components of temperature and 'g' value in terms of pressure to the uncertainty given in the certificate.
3. The pressure corrected to the local 'g' value and temperature at which calibration is performed using the following equation.

$P_e =$	$P_s * g_L * [1+(t-20)* \alpha]$
	9.80665

Where, 't' is the temperature during calibration and gravity 'g<sub>L</sub>' local acceleration due to gravity (g' value of the lab) and 'α' is the linear thermal expansion coefficient of the Piston / °C.

4. After noting down the applied pressure at each calibration point of the steps and series as per the above referred table (2.10.1) error and uncertainties are estimated.
5. Uncertainty components of the calibration to be considered but not limited to:

- Applied pressure uncertainty ( after correction if required) (u<sub>1</sub>)
- Uncertainties associated with DUC-
- a) Repeatability (u<sub>2</sub>)
  - b) Reproducibility(u<sub>3</sub>) - Optional
  - c) Resolution (u<sub>4</sub>)
  - d) Zero error (u<sub>5</sub>)
  - e) Hysteresis (u<sub>6</sub>)

6. Equation for combined uncertainty calculation:  

$$u_c = \sqrt{(u_1^2 + u_2^2 + u_3^2 + u_4^2 + u_5^2 + u_6^2)}$$

7. Expanded uncertainty U = k x u<sub>c</sub>  
 Where, k= coverage factor corresponding to the effective degree of freedom.

- **Calibration of Pressure Gauges using Digital Pressure Calibrator or Reference Pressure Gauge and Pressure Generating Pump**

1. After noting down the applied pressure at each calibration point of the steps and series as per the above referred table (11.10.1) error and uncertainties are estimated.
2. Uncertainty components of the calibration to be considered but not limited to

- Applied pressure as per the calibration certificate of digital pressure calibrator (u<sub>1</sub>)

Uncertainties associated with DUC

- Repeatability (u<sub>2</sub>)
- Reproducibility(u<sub>3</sub>) -Optional
- Resolution (u<sub>4</sub>)
- Zero error (u<sub>5</sub>)
- Hysteresis (u<sub>6</sub>)

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 101 of 133

If Digital multi meter when used to record values in units other than pressure the associated uncertainty should also be added to the above.

3. Equation for combined uncertainty calculation

$$u_c = \sqrt{(u_1^2 + u_2^2 + u_3^2 + u_4^2 + u_5^2 + u_6^2)}$$

4. Expanded uncertainty  $U = k \times u_c$

Where, k= coverage factor corresponding to the effective degree of freedom.

5. Calibration Procedure for Vacuum Gauges

ISO 27893 & DKD R-6-1 are recommended to be followed

## 9 Recommended National/ International Standards, References and Guidelines

- IS: 3624 Specifications for Pressure and Vacuum gauges”
- OIML R 101 Indicating and recording pressure gauges, vacuum gauges and pressure- Vacuum gauges with elastic sensing elements (ordinary instruments)
- EURAMET Cg-17, Version 2.0 “Guidelines on the calibration of Electromechanical Manometers”
- DKD R-6-1 Guidelines on Calibration of Pressure Gauge
- DKD R-6-2 Guidelines on Calibration of measuring devices for Vacuum
- ISO 3567 Vacuum Gauges- Calibration by direct comparison with a reference gauge
- ISO 27893 Vacuum Technology-Vacuum gauges-Evaluation of results of calibrations by direct comparison with reference gauge
- OIML R-97 Barometers (International Recommendation)

**Note: - Latest versions of the relevant standard(s) should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 102 of 133

# Chapter 1(N): Verification of Impact Testing Machine

## 1 Recommended General Requirement

- 1.1 Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5\text{Hz}$  or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.

## 2 Specific Requirements: Verification of Impact Testing Machine

### Recommended Specific Requirements:

Sl. No.	Description	Relevant Standard	Permanent Facility	Onsite Calibration	Mobile Facility
1	Impact Testing Machine (Charpy)	ISO 148-2	√*	√	X
2	Impact Testing Machine (Izod)	ISO 180	√*	√	X

\*Verification of Impact Testing Machine at permanent facility is acceptable. However, verification of performance shall be carried out after commissioning and installation of Testing Machine at site.

**Important Note:** This technical requirement is based on the above mentioned guideline. Lab may follow any relevant standard; however care shall be taken to follow the requirements in totality.

## 3 Metrological Requirements

- Indirect verification including a limited direct verification shall be performed at the time of installation or after moving the machine or striker.
- Indirect verification shall be performed at intervals not exceeding 12 months.
- Direct verification shall be performed when the machine is new and when the results of an indirect verification are unsatisfactory.
- The procedures given in 6.4.5.1 and 6.4.5.2 of the standard should be performed at the beginning of each day during which the machine is used because they provide a quick indication as to whether the performance of the machines has been impaired e.g. by dirt in bearings.

## 4 Selection of Reference Standard

The requirements for the reference test pieces are found in ISO 148-3.

## 5 Calibration Interval

The Calibration Interval of Indirect verifications shall be performed at not exceeding 12 months.

Note - Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 103 of 133



**Note:** Direct verification shall be performed when the machine is new and when the results of an indirect verification is unsatisfactory. A limited direct verification shall be performed prior to Performing an indirect verification.

## 6 Methods for Verification

### 6.1 Methods for Verification of Impact Calibration

Impact machines used for industrial, general or research laboratory testing of metallic materials in accordance with this part of ISO 148 are referred to as industrial machines. Those with more stringent requirements are referred to as reference machines. Specifications for the verification of reference machines are found in ISO 148. Please refer ISO 148 and ISO 148-3 for details.

#### 6.2 There are two methods of verification:

- a) The **direct method**, which is static in nature, involves measurement of the critical parts of the machine to ensure that it meets the requirements of this part of ISO 148. Instruments used for the verification and Calibration is traceable to national standards. Direct methods are used when a machine is being installed or repaired, or if the indirect method gives a non-conforming result.

Direct verification of the machine involves the inspection of the following items:

- i. foundation/installation;
- ii. machine framework;
- iii. pendulum, including the hammer and the striker;
- iv. anvils and supports;
- v. indicating equipment.

- b) The indirect method, which is dynamic in nature, uses reference test pieces to verify points on the measuring scale. A pendulum impact testing machine is not in compliance with this part of ISO 148 until it has been verified by both the direct and indirect methods and meets the requirements of Clauses 6 and 7 of ISO 148-2.

Indirect verification consists of verifying points on the measuring scale using reference test pieces.

#### 6.3 These reference test pieces are used:

- a) For comparison between test results obtained with the machine under consideration and test results obtained with a particular reference machine or set of reference machines, or with an ISO 148 traceable K value;
- b) To monitor the performance of a machine over a period of time, without reference to any other machine.

#### 6.4 Before each indirect verification a limited direct verification shall be performed, which includes:

- a) inspection of the machine.
- b) inspection (visual at least) of the striker and anvils for excessive wear;
- c) measurement of the gap;
- d) measurement of the angularity, only when the striker or supports are changed;
- e) measurement of the losses due to bearing friction and air resistance;
- f) measurement of the loss due to pointer friction.

### 6.5 Bias and Repeatability

**Maximum permissible values for repeatability and bias values:**

Absorbed energy level	Repeatability	Bias   Bv
≤40	≤6	≤4
≥ 40	≤ 15 % kV <sub>R</sub>	≤ 10 % kV <sub>R</sub>

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 104 of 133

## 7 Measurement Uncertainty

7.1 In theory, one can use an equation such as the following for the estimation of  $z$ , the combined instrument bias:

$$z = R + A + C + E + V + (l - l_1) + H + S \quad (\text{B.1})$$

Where

$R$  is the bias in  $K$  (in energy units) due to bias of the radius of tup or striker;

$A$  is the bias in  $K$  (in energy units) due to bias of anvil and supports geometry;

$C$  is the bias in  $K$  (in energy units) due to bias of the centre of strike;

$E$  is the bias in  $K$  (in energy units) due to the energy calculation from measured angles;

$V$  is the bias in  $K$  (in energy units) due to bias of the impact velocity;

$(l - l_1)$  is the bias in  $K$  (in energy units) due to bias of the difference between pendulum length and centre of percussion;

$H$  is the bias in  $K$  (in energy units) due to the correction for friction loss;

$S$  is the bias in  $K$  (in energy units) due to the bias of the energy read from an analogue or digital scale.

The effects the factors ( $R, A, C, E, V, l - l_1, H, S$ ) on the absorbed energy are assumed to be small if they are within the tolerances required for direct verification of the machine, and if the pendulum impact test is performed according to the standard procedure (see ISO 148-1).

However, there are uncertainties associated with the assessment of the individual factors contributing to  $z$ . Assuming that all quantities are independent, the combined standard uncertainty of  $z$  would be:

$$u_c(z) = \sqrt{u^2(R) + u^2(A) + u^2(C) + u^2(E) + u^2(V) + u^2(l - l_1) + u^2(H) + u^2(S)}$$

### 7.2 Contributions to the uncertainty of the indirect verification result:

#### 7.2.1 Bias

The primary result of an indirect verification is the estimate of the instrument bias,  $B_V$ :

$$B_V = \overline{KV_V} - KV_R$$

Where,

$\overline{KV_V}$  is the mean value of the reference test pieces broken during the indirect verification;

$KV_R$  is the certified  $KV$  value of the reference test pieces.

#### 7.2.2 Uncertainty of the Bias Value

The standard uncertainty of the bias value is equal to the combined standard uncertainties of the two terms.

$u_{RM}$ , the standard uncertainty of the certified reference value,  $KV_R$ , is calculated from the expanded uncertainty,  $U_{RM}$ , indicated on the certificate of the reference test pieces, by dividing  $U_{RM}$  with the appropriate coverage factor (also indicated on the certificate).

The uncertainty associated with  $KV_V$  is calculated as:

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 105 of 133

$$u(\overline{KV}_V) = \frac{s_V}{\sqrt{n_V}}$$

where  $s_V$  is the standard deviation of the results of the  $n_V$  reference test pieces. Therefore,  $u(B_V)$ , the standard uncertainty of  $B_V$ , is calculated as:

$$u(B_V) = \sqrt{\left(\frac{s_V}{\sqrt{n_V}}\right)^2 + u_{RM}^2}$$

### 7.2.3 Determining the combined uncertainty of the indirect verification result, $u_V$

As a general rule, bias should be corrected for. However, due to wear of the anvil and hammer parts, it is difficult to obtain a perfectly stable bias value throughout the period between two indirect verifications. This is why the measured bias value is considered an uncertainty contribution, to be combined with its own uncertainty to obtain the uncertainty of the indirect verification result,  $u_V$ :

$$u_V = \sqrt{u^2(B_V) + B_V^2}$$

## 8 Key Points

Demonstration of any CMC values doesn't automatically qualify for granting accreditation until the lab satisfies the stipulated requirement given in this document.

## 9 Recommended National/ International Standards, References and Guideline

- ISO 148-1, Metallic materials – Charpy pendulum impact test – Part 1: Test method.
- ISO 148-2 – Metallic materials – Charpy pendulum impact test – Part 2: Verification of test machines.
- ISO 148-3, Metallic materials– Charpy pendulum impact test– Part 3: Preparation and characterization of Charpy V-notch; test pieces for indirect verification of pendulum impact machines.
- ISO 180: Plastics – Determination of Izod Impact Strength.
- IS 1757 Method for Charpy Impact Test on Metallic Materials.

**Note: Latest versions of the relevant standards should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 106 of 133

## **Chapter 1(O): Calibration of Duro Meter (Rubber Hardness Testers)**

### **1 General Requirements:**

- 1.1 Acoustic noise level in the laboratory shall be maintained to facilitate proper performance of calibration work. Noise level shall be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 The calibration area shall have adequate level of illumination. Where permissible, fluorescent lighting is preferred to avoid localized heating and temperature drift. The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 2.5$  Hz or better on the calibration bench.

### **2 Specific Requirements – Calibration of Durometer - Rubber Hardness Tester**

#### **2.1 Scope: Calibration of Durometer - Rubber Hardness Tester:**

Accreditation can be sought for verification of indentation depth or for force verification or for both depending on the laboratories capability.

<b>Sl. No</b>	<b>Rubber Hardness Testing Machine</b>	<b>Relevant Standard, Guidelines</b>	<b>Permanent facility</b>	<b>On-site calibration</b>
1	Rubber - Calibration and Verification of Hardness Testers - Durometers	ISO 18898-2006	√	X

**Important Note:** This technical requirement is based on the above mentioned standard, taking into account only the salient features required during calibration. Lab may follow any relevant standard; however care shall be taken to follow the requirements in totality.

### **3 Calibration Interval**

<b>Reference Equipment</b>	<b>Recommended Interval*</b>
Force measuring Instruments	1 year
Force weights	3 Years
Depth Measuring Instruments	1 Year

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

### **4 Metrological Requirements**

The measurands of indenter and pressure foot for the instruments to be calibrated are depicted in figures 1 to 6 of ISO 18898 (table 1 to 9 of the standard ISO 18898 consolidated as below):

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 107 of 133

Sl. No.	Type of Durometer	Force on Indenter	Depth of indentation in mm	Duration of force application
1	A	Force in mN, $F = 550 + 75H_A$ , $\Delta F = \pm 37.5$ where $H_A$ = hardness reading on type A durometer	0 to 2.5, $\Delta L = \pm 0.02$	3 or 15 s
2	D	Force in mN, $F = 445H_D$ , $\Delta F = \pm 222.5$ where $H_D$ = hardness reading on type D durometer	0 to 2.5, $\Delta L = \pm 0.02$	3 or 15 s
3	AO	Force in mN, $F = 550 + 75H_{AO}$ , $\Delta F = \pm 37.5$ where $H_{AO}$ = hardness reading on type AO durometer	0 to 2.5, $\Delta L = \pm 0.02$	3 or 15 s
4	AM	Force in mN, $F = 324 + 4.4H_{AM}$ , $\Delta F = \pm 8.8$ where $H_{AM}$ = hardness reading on type AM durometer	0 to 1.25, $\Delta L = \pm 0.01$	3 or 15 s
5	IRHD Dead load tester Method-N	a. Contact force on indenter $0.3N \pm 0.02N$ , b. Total force on indenter $5.7N \pm 0.03N$	Incremental indentation depth $L=f(\text{IRHD})$ $\Delta L = \pm 0.01$	Contact force-5 s Total force -30 s
6	IRHD Dead load tester Method-H	a. Contact force on indenter $0.3N \pm 0.02N$ , b. Total force on indenter $5.7N \pm 0.03N$	Incremental indentation depth $L=f(\text{IRHD})$ $\Delta L = \pm 0.01$	Contact force-5 s Total force -30 s
7	IRHD Dead load tester Method-L	a. Contact force on indenter $0.3N \pm 0.02N$ , b. Total force on indenter $5.7N \pm 0.03N$	Incremental indentation depth $L=f(\text{IRHD})$ $\Delta L = \pm 0.01$	Contact force-5 s Total force -30 s
8	IRHD Dead load tester Method-M	a. Contact force on indenter $8.3mN \pm 0.5mN$ , b. Total force on indenter $153.3mN \pm 1 mN$	Incremental indentation depth $L=f(\text{IRHD})$ $\Delta L = \pm 0.002$	Contact force-5 s Total force -30 s
9	IRHD Pocket Meter	a. Contact force on indenter $2.65 N \pm 0.15 N$ , b. Total force on indenter $5.7N \pm 0.03N$	Depth of indentation $L=f(\text{IRHD})$ $\Delta L = \pm 0.02$	Contact force-5 s Total force -30 s

**Note-1:** If indication is magnified by a factor of 6 (by mechanical means) before the measurement is made then ( $\Delta L = \pm 0.01mm$ ).

**Note-2:** Care should be taken that the force from the hardness tester is applied vertically to the force measuring device and a hinged support may be used to aid force application.

**Note-3:** Duration time for force application: A tolerance on the time of application of the force is only given for the case of a durometer in a stand with an automatic timing device. The tolerance is then  $\pm 0.3$  s. The device shall be calibrated in accordance with ISO 18899.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 108 of 133

## 5 Calibration Procedure

### 5.1 Calibration of Durometer by measuring depth of Indentation:

The permissible calibration values along with errors ( $\Delta L$ ) are as given below for verification depth of indentation in different types of durometers:

Shore value	Shore A	Shore D	Shore AO	Shore AM
	Value of indentation depth L in mm ( $\Delta L = \pm 0.02\text{mm}$ )	Value of indentation depth L in mm ( $\Delta L = \pm 0.02\text{mm}$ )	Value of indentation depth L in mm ( $\Delta L = \pm 0.02\text{mm}$ )	Value of indentation depth L in mm ( $\Delta L = \pm 0.01\text{mm}$ )
0	2.500	2.500	2.500	1.250
10	2.250	2.250	2.250	1.125
20	2.000	2.000	2.000	1.000
30	1.750	1.750	1.750	0.875
40	1.500	1.500	1.500	0.750
50	1.250	1.250	1.250	0.625
60	1.000	1.000	1.000	0.500
70	0.750	0.750	0.750	0.375
80	0.500	0.500	0.500	0.250
90	0.250	0.250	0.250	0.125
100	0.000	0.000	0.000	0.250

IRHD Pocket Meter		IRHD Method N	
Shore value	Value of indentation depth L in mm ( $\Delta L = \pm 0.01\text{mm}$ )	Shore value	Value of indentation depth L in mm ( $\Delta L = \pm 0.01\text{mm}$ )
100	0	100.0	0
90	0.191	80.2	0.350
80	0.323	70.4	0.510
70	0.473	60.1	0.710
60	0.653	50.2	0.960
50	0.884	40.1	1.300
40	1.195	30.0	1.800
30	1.650		

IRHD Method H		IRHD Method L		IRHD Method M	
Shore value	Value of indentation depth L in mm ( $\Delta L = \pm 0.01\text{mm}$ )	Shore value	Value of indentation depth L in mm ( $\Delta L = \pm 0.01\text{mm}$ )	Shore value	Value of indentation depth L in mm ( $\Delta L = \pm 0.002\text{mm}$ )
100.0	0	34.9	1.1	100.0	0
98.8	0.1	21.3	1.8	80.2	0.058
95.4	0.2	14.1	2.5	70.4	0.085
91.1	0.3	9.9	3.18	60.1	0.118
84.8	0.4			50.2	0.160
				40.1	0.217
				30.0	0.300

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 109 of 133

## 5.2 Force Verification Method:

The permissible calibration values along with errors ( $\Delta F$ ) are as given below for verification of spring Force in different types of durometers:

Shore value (A, D, AO or AM as per the selected durometer)	Shore A	Shore D	Shore AO	Shore AM	IRHD Pocket Meter
	Value of spring force F, in mN $\Delta F = \pm 37.5$ mN	Value of spring force F, in mN $\Delta F = \pm 222.5$ mN	Value of spring force F, in mN $\Delta F = \pm 37.5$ mN	Value of spring force F, in mN $\Delta F = \pm 8.8$ mN	Value of spring force F, in N $\Delta F = \pm 0.15$ N
0	550	-	550	324	-
10	1300	4450	1300	368	-
20	2050	8900	2050	412	-
30	2800	13350	2800	456	0.795
40	3550	17800	3550	500	1.060
50	4300	22250	4300	544	1.325
60	5050	26700	5050	588	1.590
70	5800	31150	5800	632	1.855
80	6550	35600	6550	676	2.120
90	7300	40050	7300	720	2.385
100	8050	4450	8050	764	2.65

IRHD Methods N, H, L		IRHD (Method M)
Value of spring force F, in N		Value of spring force F, in N
Apply the contact force	$F_c = 0.3 \pm 0.02$ N	$F_c = 8.3 \pm 0.5$ mN
Apply the indenting force $F_i = F_t - F_c$	$F_i = 5.4 \pm 0.01$ N	$F_i = 145.0 \pm 0.5$ mN
Total force $F_t$	$F_t = 5.7 \pm 0.01$ N	$F_t = 153.3 \pm 1$ mN

## 6 Measurement Uncertainty

### 6.1 Uncertainty components for Indentation Depth:

- ✓ Uncertainty of depth measuring device
- ✓ Standard deviation of depth measurements

### 6.2 Uncertainty components for Force Verification:

- ✓ Uncertainty of applied force
- ✓ Standard deviation of force measurements.

## 7 Recommended National/ International Standard, References and Guidelines

- ISO 18898 – Rubber – Calibrations and Verification of Hardness Tester
- ISO 18899 – Rubber – Guide to the Calibration of Test Equipment
- ASTM-D-2240-05 – Standard test method for Rubber Property – Durometer Hardness
- OIML R111-1 Metrological and technical requirement of weights Classes E<sub>1</sub>, E<sub>2</sub>, F<sub>1</sub>, F<sub>2</sub>, M<sub>1</sub>, M<sub>2</sub>, M<sub>3</sub>
- OIML D28 Conventional value of the result of weighing in air

**Note: Latest versions of the relevant standards should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 110 of 133

## 2. Fluid Flow Calibration

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 111 of 133



## 1. Recommended General Requirements

- 1.1 Acoustic Noise -Noise level is recommended to be maintained less than 60 dBA, wherever it affects adversely the required accuracy of measurement.
- 1.2 The recommended level of illumination is 250-500 lux on the working table.
- 1.3 The calibration laboratory should make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is  $\pm 2\%$  or better, and Frequency variation  $\pm 1$  Hz or better on the calibration bench.
- 1.4 Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart is recommended to be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 1.5 Effective mains earthing may be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.
- 1.6 Adequate safety measures against electrical, chemical fire hazards must be available at the work place as per the statutory requirements.
- 1.7 The environmental condition during calibration, temperature shall be preferably within (10–40) °C. The environmental conditions for the activity of the laboratory shall be such as not to adversely affect the required accuracy of measurement. Facilities should be provided whenever necessary for recording temperature, pressure and humidity values prevailing during calibration. The atmospheric conditions maintained in the laboratory during calibration should be reported in the calibration report/certificate. The laboratory may specify limits on the environmental conditions to be achieved in the laboratory. The condition shall appropriate to the level of accuracy required for calibration undertaken by the laboratory. The environmental conditions shall be monitored at appropriate intervals and calibrations stopped when the environmental conditions fall outside of specified limits. Temperature extremes of the working fluid must be avoided and difference between the fluid temperature and the ambient air temperature should not exceed 10°C.

Environmental conditions of fluid flow liquid and control room to be maintained as per the applicable standard(s)

## 2. Recommended facility of the scope

Sl.	Device under calibration	Relevant Standard/ Reference	Permanent Facility	Onsite Calibration	Mobile Facility
1.	Differential pressure flow meters (Flow Element (Orifice Plate, Venturi, Critical Flow Venturi, Flow Nozzles, Pitot Tubes etc.)	National / International standards / AGA reports / OIML recommendations	√	√	x
2.	Flowmeters Magnetic, Turbine, Fluidic, Swirl Meter, etc.)		√	√	x
3.	Pitot Tube, Anemometers		√	x	x
4.	Coriolis, Ultrasonic		x	√	

## 3. Recommended Reference Standards

The reference standard for calibration can be selected from below mentioned to achieve TUR ratio preferably of 3:1

- a. Piston Prover
- b. Bell Prover
- c. Gravimetric system
- d. Volumetric system
- e. Pivot type system
- f. Sonic nozzles
- g. Reference flowmeter
- h. Ultrasonic

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 112 of 133

**4. Recommended Calibration Methods**

Relevant National / International standards / AGA reports / OIML recommendations may be followed for calibration.

**5. Recommended Measurement Uncertainty Components / CMC**

**Table 1- Uncertainty budget of electromagnetic flowmeter calibration at 150 m<sup>3</sup>/h (calibration medium: water)**

Quantity X <sub>i</sub>	Estimate x <sub>i</sub>	Limits ±Δ x <sub>i</sub>	Probability distribution Type A/B	Standard uncertainty ±u <sub>i</sub>	Sensitivity coefficient c <sub>i</sub>	Uncertainty contribution u <sub>i</sub> (y)= c <sub>i</sub> · u <sub>i</sub>	Degree of freedom v <sub>i</sub>
Repeatability of MUC	150 m <sup>3</sup> /h	-	Normal, Type A	0.0123 %	1	0.0123 %	4
Weighing System	2000 kg	0.02 %	Normal, Type B	0.01 %	1	0.01 %	∞
Timer	60 s	0.01 %	Normal, Type B	0.005 %	1	0.005 %	∞
Diverter Error	60 s	-	Normal, Type B	0.025 %	1	0.025 %	∞
Density Measuring System	1000 kg/m <sup>3</sup>	0.01%	Normal, Type B	0.005 %	1	0.005%	∞
Digital Multimeter	20 mA	0.02 %	Normal, Type B	0.01 %	1	0.01 %	∞
Error (e)	-0.34 %						
Combined uncertainty u <sub>c</sub> (e)						0.032 %	184
Expanded uncertainty U <sub>(e)</sub>						0.064%	184
At coverage factor k=2 for 95.45% confidence level							

**Table 2- Uncertainty budget of Rotameter calibration at 140 L/min (calibration medium: air)**

Quantity X <sub>i</sub>	Estimate x <sub>i</sub>	Limits ±Δ x <sub>i</sub>	Probability distribution Type A/B	Standard uncertainty ±u <sub>i</sub>	Sensitivity coefficient c <sub>i</sub>	Uncertainty contribution u <sub>i</sub> (y)= c <sub>i</sub> · u <sub>i</sub>	Degree of freedom v <sub>i</sub>
Repeatability of MUC	110 L/min	-	Normal, Type A	0.0208 %	1	0.0208 %	4
Resolution of MUC	3 L/min	1.0714 %	Rectangular, Type B	0.6186 %	1	0.6186 %	∞
Zero drift of MUC	0 L/min	0 L/min	Rectangular, Type B	0 %	1	0 %	-
Reference Standard	112. 720 L/min	0.20 %	Normal, Type B	0.10 %	1	0.10 %	∞

**National Accreditation Board for Testing and Calibration Laboratories**

Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 113 of 133

Reference Pressure Monitor	100 kPa	0.003%	Normal, Type B	0.0015 %	1	0.0015%	$\infty$
Thermometer	25 °C	0.04 %	Normal, Type B	0.02 %	1	0.02 %	$\infty$
Error (e)	-1.943 %						
Combined uncertainty $u_c(e)$						0.6287 %	3904620
Expanded uncertainty $U(e)$	At coverage factor $k=2$					1.26%	3904620

For the purpose of CMC evaluation refer NABL 143 “Policy on Calibration and Measurement Capability (CMC) and Uncertainty in Calibration”

## 6. Other Relevant Information

The following details give broad guidelines relating to Fluid Flow Facility installation, operation and maintenance: In all cases, the flow conditions at the test section shall be steady, the velocity distribution across the test cross-section is symmetrical and the flow is free from swirl. This may be achieved by use of flow conditioners and by providing sufficient upstream and downstream length requirements of DUC (Device under Calibration) as per relevant standard.

Working fluid should be clean and the degree of cleanliness will be determined by the type of flow meters being calibrated. The sump tank & constant level over tank shall be cleaned periodically. The working liquid must be of uniform composition and temperature.

The liquid temperature stability criteria must be met for the calibration media. In case, the variation is more than  $\pm 1$  °C/hour, density value will change and results shall be corrected appropriately.

Density and viscosity (wherever felt necessary by laboratory) of the fluid must be measured at the temperature at which the calibration is performed and recorded. It is a common practice to check these variables at a recognized reference temperature in addition to the working temperature.

Accurate measurement of density is essential, in case calibration is based on volumetric basis. Measurement of density of liquids shall be made by equipments like densitometer (online/ offline), hydrometer, weighing balance etc. If the liquid to be measured is reasonably pure and clean it is acceptable to measure its temperature and to derive its density from a table of physical properties.

Fluid flow control circuit must be such that:

- Presence of flow controlling device shall have no effect on flow meter calibration. The flow control valve may be installed in the downstream or upstream judiciously.
- Any drop in pressure at the flow meter does not cause dissolved gases to come out of solution/ cavitations to occur.
- The flow meter and complete system continue to run full under all conditions with no stagnant volumes in the circuit.

The laboratory must be adequately kept clean and well maintained. It should be adequately ventilated to prevent accumulation of flammable vapors wherever necessary.

For On-site Calibration activities, laboratory shall comply with NABL requirements mentioned in **NABL 130**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 114 of 133

**“Specific Criteria for Site Testing and Site Calibration Laboratories”**. If there is any problem envisaged like the absence of authorized signatory at on-site calibration, the laid down procedures ensuring compliance to such procedures must be specially addressed by the laboratory.

The following details give broad guidelines relating to Fluid Flow Facility installation, operation and maintenance:

- In all cases, the flow conditions at the test section shall be steady, the velocity distribution across the test cross-section is symmetrical and the flow is free from swirl. This may be achieved by use of flow conditioners and by providing sufficient upstream and downstream length requirements of DUC (Device under Calibration) as per relevant standard.
- Working fluid must be clean and the degree of cleanliness will be determined by the type of flow meters being calibrated. The sump tank & constant level over tank shall be cleaned periodically.
- The working liquid must be of uniform composition and temperature.
- The liquid temperature stability criteria must be met for the calibration media. In case, the variation is more than  $\pm 1$  °C/hour, density value will change and results shall be corrected appropriately.
- Density and viscosity (wherever felt necessary by laboratory) of the fluid must be measured at the temperature at which the calibration is performed and recorded. It is a common practice to check these variables at a recognized reference temperature in addition to the working temperature.
- Accurate measurement of density is essential, in case calibration is based on volumetric basis. Measurement of density of liquids shall be made by equipments like densitometer (online/ offline), hydrometer, weighing balance etc. If the liquid to be measured is reasonably pure and clean it is acceptable to measure its temperature and to derive its density from a table of physical properties.
- Fluid flow control circuit must be such that:
- Presence of flow controlling device shall have no effect on flow meter calibration. The flow control valve may be installed in the downstream or upstream judiciously.
- Any drop-in pressure at the flow meter does not cause dissolved gases to come out of solution/ cavitations to occur.
- The flow meter and complete system continue to run full under all conditions with no stagnant volumes in the circuit.
- The laboratory must be adequately kept clean and well maintained. It should be adequately ventilated to prevent accumulation of flammable vapours wherever necessary.
- For On-site Calibration activities, laboratory shall comply with NABL requirements mentioned in NABL 130 “Specific Criteria for Site Testing and Site Calibration Laboratories”. If there is any problem envisaged like the absence of authorized signatory at on-site calibration, the laid down procedures ensuring compliance to such procedures must be specially addressed by the laboratory.

## 7.1 Uncertainty of the Equipment

There are two ways of stating measurement error and uncertainty for the entire range of measuring instrument.

- Percent of full scale or FS
- Percent of reading or indicated value

The difference between the two concepts becomes highly significant when an instrument is operating near the bottom of its turn down range.

The flowmeter error/ uncertainty may be assigned in Full Scale (for compliance to specification), if it is not to be used as a reference standard for further calibration, otherwise it should be reported in percentage of reading.

**7.2** It is the responsibility of the laboratory to arrange dead weights for calibration of weighing systems as per OIML R-76 or any other standards. These weights should be calibrated from NMI or any accredited laboratories.

**7.3** For on-site calibration using ultrasonic flowmeter, the measurement uncertainty will not be recommended better than 1%

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 115 of 133

## 8. Recommended relevant National/ International Standards, References and Guidelines in Fluid Flow Calibration

- ISO 4185 Measurement of liquid flow in closed conduits - Weighing method
- ISO 9368-1 Measurement of liquid flow in closed conduits by the weighing method - Procedures for checking installations - Part 1: Static weighing systems
- ISO 9300 Measurement of gas flow by means of critical flow Venturi nozzles
- ISO 5167-1 Measurement of fluid flow by means of pressure differential devices inserted in circular cross-section conduits running full - Part 1: General principles and requirements
- ISO 5167-2 Measurement of fluid flow by means of pressure differential devices inserted in circular cross-section conduits running full - Part 2: Orifice plates
- ISO 5167-3 Measurement of fluid flow by means of pressure differential devices inserted in circular cross-section conduits running full - Part 3: Nozzles and Venturi nozzles
- ISO 5167-4 Measurement of fluid flow by means of pressure differential devices inserted in circular cross-section conduits running full - Part 4: Venturi tubes
- ISO 5167-5 Measurement of fluid flow by means of pressure differential devices inserted in circular cross-section conduits running full - Part 5: Cone meters
- ISO 5168 Measurement of fluid flow - Procedures for the evaluation of uncertainties
- ISO 8316 Measurement of liquid flow in closed conduits -Method by collection of the liquid in a volumetric tank
- ISO 9104 Measurement of fluid flow in closed conduits - Methods of evaluating the performance of electromagnetic flow-meters for liquids
- IEC 61400-12-1 Wind turbines - Part 12-1: Power performance measurements of electricity producing wind turbines
- ISO 4006 Measurement of fluid flow in closed conduits - Vocabulary and symbols
- ISO 3966 Measurement of fluid flow in closed conduits - Velocity area method using Pitot static tubes
- ISO 7194 Measurement of fluid flow in closed conduits - Velocity-area methods of flow measurement in swirling or asymmetric flow conditions in circular ducts by means of current-meters or Pitot static tubes
- ASTM D5096-02 Standard Test Method for Determining the Performance of a Cup Anemometer or Propeller Anemometer<sup>1</sup>
- ISO 17713-1 Meteorology—Wind measurements - Part 1: Wind tunnel test methods for rotating anemometer performance
- ASTM D6011-96 Standard Test Method for Determining the Performance of a Sonic Anemometer/Thermometer
- ASTM D6011-96 Standard Test Method for Determining the Performance of a Sonic Anemometer/Thermometer
- SP 250-49 NIST calibration services for Gas Flowmeters - Piston Prover and Bell Prover Gas Flow Facilities
- SP 250-63 Gas Flowmeter Calibrations with the 34 L and 677 L PVT Standards
- SP 250-73 NIST Calibration Services for Water Flowmeters - Water Flow Calibration Facility
- SP 250-80 Gas Flowmeter Calibrations with the Working Gas Flow Standard
- SP 250-1039 Hydrocarbon Liquid Calibration service
- MAPAN, JMSI Vol. 28(1) Comparative Analysis of Different Air Density Equations

**Note: - Latest version of the relevant standard(s) should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 116 of 133

### 3. Radiological Calibration

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 117 of 133

## 1. Scope of Accreditation

- 1.1. An accredited laboratory shall be classified as calibration laboratory for the accredited parameters in the field of Radiological Measurements in accordance to its measurement capability.
- 1.2. A calibration laboratory seeking accreditation to offer calibration services in the field of Radiological Measurements will generally carry out calibration of exposure / dose / count, exposure rate / dose rate / count rate or any other relevant parameters in the equipment, which include:
  - Radiation Monitors (e.g.-radiation survey meter, area gamma monitor)
  - Radiation Dosimeters (e.g.-pocket dosimeter, chemical dosimeter, thermoluminescent dosimeter)

## 2. Recommended Environmental Conditions

Lab shall have low background radiation and be preferably air-conditioned.

1. Calibration area shall not be used for any other activity. Eg. Records, sample storage, etc.
2. Standard Environmental Condition for the Radiological Calibration Laboratory shall be as follows:
  - a) Temperature  $25^{\circ}\text{C} \pm 3^{\circ}\text{C}$ ; and
  - b) RH 45% to 75%
- 2.1 **Vibrations:-** The calibration area shall be adequately free from vibrations generated by central air-conditioning plants, vehicular traffic and other sources to ensure consistent and uniform operational conditions. The laboratory shall take all special / protective precautions like mounting of sensitive apparatus on vibration free tables and pillars etc., isolated from the floor, if necessary.
- 2.2 **Acoustic Noise:-** Acoustic noise level in the laboratory shall be maintained to facilitate proper performance of calibration work. A threshold noise level of 60 dBA is recommended unless otherwise stated. - The laboratory should be categorized under Central Pollution Control Board (CPCB) guidelines (industry, commercial, residential and silence zone) and the limit can be specified accordingly. 65 dBA is specified for commercial, which can be used here).
- 2.3 **Illumination** The calibration area shall have adequate level of illumination. Where permissible, fluorescent lighting is preferred to avoid localized heating and temperature drift. The recommended level of illumination is 250-400 lux on the working table with glare index of 19 – For normal activities about 1000 lux is recommended and in case of precision or detailed works probably more like about 2000 lux.
- 2.4 **Dimension of the Calibration Lab:** The radiation enclosure room should have a appropriate dimension to house the radiation exposure device. A separate control room can be provided for operating the Source Exposure Device remotely and recording the instrument reading using the CCTV camera
- 2.5 **Entry to the Calibration Area:-** As far as possible, only the authorized staff engaged in the calibration activity shall be permitted entry inside the calibration area. The access to the areas shall be controlled through physical protection systems. Entry for others for planned activities shall be regulated on case by case basis through administrative control procedures.
- 2.6 Regulatory consent from AERB is required to operate the laboratory.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 118 of 133

### 3. Reference Standard

Each calibration laboratory should have measurement standards or equipment of required accuracy in respect of each parameter covered by it in order to be able to realize and to substantiate the corresponding measurement capability claimed. Stability of the standards, accuracy of the values realized through them and repeatability shall be regularly monitored.

- 3.1 Any bias resulting from ageing of standards should be precisely determined. Instructions for operating each standard and equipment/ instrument (SOP) shall be readily available for use by the laboratory staff members.
- 3.2 The standards/ measuring equipment of the laboratory shall be calibrated at regular intervals with higher accuracy standards. The calibration certificates, performance history sheets in respect of the reference standard/ working standards and measuring equipment shall be held safely by the laboratory.
- 3.3 Radioactive Source: A radiation source having suitable strength to cater the requirements for the calibration of instrument in all the ranges should be available, which is to be procured with due approval from AERB. The radiation source should be housed in a suitable Source Exposure Device as mentioned in the following section 4.5
- 3.4 Source Exposure Device: The radiation level on the surface of Source Exposure Device should not be more than 0.01 mSv/h (1mR/hr). The collimator of the Source Exposure Device should be suitable to ensure that the radiation beam is wide enough to cover the detector and the scattered radiation is not more than 5 % of the primary radiation. Apart from type approval of the radiation source exposure device, design approval of radioactive source housing device for the safe transport of radioactive material is required from AERB.
- 3.5 Reference Standard Instrument: A reference standard instrument having established the reference radiation field with an uncertainty of  $\pm 5\%$  should be available. A reference standard instrument should have traceability to the national standards maintained by Bhabha Atomic Research Centre (BARC), Mumbai.
- 3.6 Auxiliary Equipment's for Reference Standard Instrument: Other auxiliary equipment such as Thermometer and barometer having capability to measure ambient temperature and ambient pressure with a least count of at least 0.1° and 0.1 mbar respectively should be available.
- 3.7 Distance Measuring Tool: It should be available with a capability of measuring distance with a least count of at least 1 mm.
- 3.8 Phantom: A suitable water phantom will be required for the calibration for personnel dosimeters namely direct reading dosimeters. The dimensions of water slab phantom can be obtained from the IAEA Publication - Safety Report Series – 16: 2000.

### 4. Recommended General Requirements of Laboratory

- 4.1 The calibration laboratory shall make arrangement for regulated and uninterrupted power supply. The recommended regulation level is  $\pm 1\%$  or better on the calibration bench. [Conventionally specify 240 V AC +/- 10 % and 50 Hz +/- 1.5 Hz )
- 4.2 Adequate arrangements shall be made by the laboratory so as to ensure temperature gradient not exceed in 1.5 °C per hour inside the laboratory in case of power failure.
- 4.3 Special care shall be taken about the location of magnetic field sources like, transformers, looped wires, ferrous materials etc., in order to minimize magnetic interference in the measurements.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 119 of 133



- 4.4 Adequate screening of the laboratory against electromagnetic interference shall be done, if necessary. By pass filters should also be provided to minimize conducted interference effect on the electronic equipment. Special shielding chambers should be provided in the laboratory for measurements, particularly when signal to noise is a disturbing factor for accurate measurements.
- 4.5 The reference standards shall be maintained at temperatures in case specified for their maintenance in order to ensure their conformity to the required level of operation and traceability. The laboratory should have specific facilities required for carrying out the calibrations of parameters chosen.
- 4.6 Relevant fire extinguishing equipment for possible fire hazards shall be available in the corridors or convenient places in the laboratory. Adequate safety measures against electrical/ chemical fire hazards must be available at the work place. Laboratory rooms/ areas where highly inflammable materials are used/ stored should be identified. Access to relevant fire equipment should be assured near these rooms/ areas.
- 4.7 BIS Specifications SP.31–1986, Treatment for Electric Shock, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart should be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- 4.8 Means shall be provided for Safety against radiation hazards in the laboratory. Special warning and protection devices shall be available for the safety of workers as per Atomic Energy (Radiation Protection) Rules, 2004.
- 4.9 The radioactive sources used for calibration shall be kept in safe and secure manner. The source shall have a valid calibration certificate from the supplier.
- 4.10 Use and disposal of source shall be as per Atomic Energy act 1962 and rules promulgated thereunder.
- 4.11 The person who is handling the source during calibration should have adequate knowledge of radiation safety and should take all radiation safety precaution while handling. The person should have certification from AERB to handle the radioactive sources as per Atomic Energy (Radiation Protection) Rules, 2004.
- 4.12 The person handling the radioactive source should use Thermoluminescent Dosimeter (TLD) badge for estimation of exposure of radioactive source.

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 120 of 133

## 4. Electro-Technical Calibration

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 121 of 133

## 1. Recommended General Requirements

The following minimum standard Environmental Conditions to be maintained in the laboratory:

- Temperature: In-house:  $25^{\circ}\text{C} \pm 4^{\circ}\text{C}$  (or better depending on the sensitivity of the measurements)
- RH: 30% to 75%. For HV Measurement the Humidity shall be < 60%.
- The recommended level of illumination is 250-500 lux on the working table.
- Earth Resistance: <  $1\Omega$  (Shall be measured and recorded at least once in a year.)
- N-E Voltage: < 1 V
- THD: <5% (Ref.std: IS:13875, IS: 4722)
- Acoustic noise: <60dBA.
- Power Supply regulation:  $\pm 2\%$  or Better on Calibration Bench.
- Frequency: 50 Hz  $\pm 1$  Hz.
- The calibration laboratory shall make arrangements for regulated and uninterrupted power supply to provide backup to calibration bench.
- During calibration of Inductance parameter care shall be taken about the location of magnetic field sources like, transformers, looped wires, ferrous materials etc., in order to minimize magnetic interference in the measurements.
- Adequate screening of the laboratory against electromagnetic interference may be done if necessary. By pass filters should also be provided to minimize conducted interference effect on the electronic equipment.
- Adequate protective measures, like use of transient suppressors etc, shall be taken by the laboratory to ward off high current spikes and transients emanating from switching on and off, of heavy machines, surges in power lines and other such reasons, from reaching the electronics equipment in general and computer based systems involving data storage facilities in particular.

Note: For calibration of Inductance (Low Frequency) and DC Resistance, the temperature variation must be controlled such that the MU does not exceed 10% of MU due to temperature variation.

### For High Voltage Facility

- The inductive voltage divider should be protected against the effects of AC magnetic fields. For High Voltage calibration, ensure humidity shall be <60%. The laboratory may use, if necessary, isolation transformers and filters etc. to ensure minimization of ground current and effects of mains hum interference.
- The power supply to the calibration laboratory should be preferably directly obtained from the substation as far as possible and should be preferably not be on the same feeder line which is supplying power to workshops and other production areas which require operation of heavy duty machines.
- Laboratory should have dedicated earth line from the earth pit in high voltage laboratory, to ensure earth resistance <  $1\Omega$ .
- Relevant fire extinguishing equipment for possible fire hazards, shall be available in the corridors or convenient places in the laboratory. Adequate safety measures against electrical, chemical fire hazards must be available at the work place. Laboratory rooms/ areas where highly inflammable materials are used/ stored shall be identified. Access to the relevant fire equipment shall be assured near these rooms/ areas.
- Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 122 of 133

## 2. Recommended Facility of the Scope

Specific Requirements for Electro-Technical calibration are as follows:

Sl.	Description	Permanent Facility	Onsite Calibration Facility	Mobile Facility
1	<b>ALTERNATING CURRENT (&lt;1 GHz)</b> Attenuation, Capacitance, Current, Dielectric loss angle, Energy, Inductance, Impedance, FM Modulation, AM Modulation, Phase angle, Power, Power factor, Resistance, Reflection Coefficient, Voltage, High Voltage and others	√	√	√ (except High Voltage)
2	<b>DIRECT CURRENT</b> Current, Power, Energy, Resistance, Voltage, Capacitance, High Voltage and others	√	√	√ (except High Voltage)
3	<b>RF/Microwave (1 GHz and Above)</b> Attenuation, Impedance, Frequency Modulation, Amplitude Modulation, Power, VSWR, Phase Modulation and Others	√	√ (except VSWR, Power, Phase Modulation)	√
4	<b>TIME &amp; FREQUENCY</b> Frequency (LF and HF), Time interval, Time Period and others	√	√	√
5	<b>EMI/ EMC</b> Antenna Factor, Attenuation, Automotive Transient Generator, Coupling Factor/ Coupling Loss Directivity, Conducted RF, Combination Wave Surge, Damped Oscillatory Wave Generator, Decoupling of Common Mode Disturbance, Electrostatic Discharge, Electrical Fast Transients, EMI Test Receiver, Isolation, Impulse Voltage, Impulse/ Immunity Generator, Insertion Loss/ RF Attenuation, Impedance, Longitudinal conversion Loss, Preamplifier Gain, Phase angle, RF Power Amplifier, Ring Wave Generator, Telecom Surge Test System, Return loss (VSWR), Voltage Dips/ Interruptions, Voltage Division Factor and others	√	√ (except Antenna factors)	X
6	<b>ELECTRICAL EQUIPMENT</b> Current Transformers, Voltage Transformers, Oscilloscopes, Bridges, CT-VT Comparator, Tr. Ratio Standard, Tan Delta (eg. Dissipation factor), Gauss Meter and Others	√	√	√ (Only oscilloscope and Bridges)
7	<b>TEMPERATURE SIMULATION</b>	√	√	√

## 3. Recommended Calibration Methods

Laboratory may use standard methods as per national and International standards or as specified by reputed technical organization, or as per relevant scientific texts or journals or as specified by reputed manufacturer of the equipment. While performing calibration to achieve CMC, Test uncertainty Ratio (TUR) of preferably 3:1 must be followed. In exceptional cases coarser can be accepted with proper technical justification.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 123 of 133

### 3.1 Comparison Method

Applicable for calibration of Measuring Device (eg. DMM) with another Measuring Device (eg. DMM) having better uncertainty

The laboratory while using the comparison method of calibration (using a nominal source of good short-term stability and a measurement standard/master) shall evaluate stability at short intervals (within 24 hours) devise methods to evaluate the short-term stability of the sources/supply. Records of the short-term stability shall be available with the laboratory for verification during Assessment.

The stability data generated by the laboratory for the source /supply needs to be used as one of the influencing components under a Type-B during estimation of uncertainty in measurement. This shall be verified by the assessor during assessment.

The suitability of the comparison method (especially in case of low voltage, low/high current, low/high resistance, high frequency voltage measurements) shall be established by the laboratory by studying the effects of factors like capacitive / inductive / resistive loading, allowable current, compliance voltage etc. on measurement. Assessor shall verify the same during assessments.

Also, Methods like Difference Method, Null Method, Substitution Method, etc can be used for Comparison.

**Note:** Lab having only measurement capability (By Direct and Comparison Method) can calibrate source/ DMM.

### 3.2 Direct Method

Direct measurement with reference Equipment eg. Multi function calibrator or DMM

### 3.3 Automated Method

Any software used by laboratories for performing automated calibration shall be validated so that all parameters and ranges intended to be calibrated using the software are taken care of. Records for the same shall be available with the laboratory during assessment. Such software needs to be verified by the user laboratory periodically. Periodicity of these verifications may be decided by the user laboratory. Re-validation of software is required whenever there is a change in the version of the software used.

### 3.4 V/I Method

Use for Measurement of Low Value Resistance (Mostly DC Resistance) for Precise Measurements.

## 4. PARAMETERS FOR CMC CALCULATION - For Permanent Facility Calibration

4.1 CMC value is not the same as expanded uncertainty reported in the calibration Certificate/Report (Issued by the laboratory). CMC values exclude the uncertainties which are attributed to the DUT (Device under Calibration).

4.2 For the purpose of CMC evaluation, the following components shall be considered however this is not limited; any other relevant component may be included.

- Repeatability (10 readings at least at minimum and maximum Points within the range, wherever applicable).
- Uncertainty of master (Lab to verify whether error has been adjusted or not as mentioned in the calibration certificate).
- Accuracy/Uncertainty declared by Manufacturer/Stability estimated by laboratory (ref. 2.5.1).
- Resolution of the readout unit.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 124 of 133

For the purpose of CMC evaluation refer NABL 143 "Policy on Calibration and Measurement Capability (CMC) and Uncertainty in Calibration"

## 5. Measurement Uncertainty

### 5.1 Minimum Required Type 'B' Components for Uncertainty Calculations:

The following Type B components shall be necessarily considered as a minimum for estimation of uncertainty in measurement:

U1: Uncertainty reported in the calibration certificate of the standard(s) / master(s). (Lab to verify whether error has been adjusted or not as mentioned in the calibration certificate.) *Also Lab to mention the error separately if not adjusted in the calibration certificates issued to their customers.*

U2: Uncertainty arising from Long Term Stability/ drift data of the measurement standard(s)/master(s) used for calibration (Detailed explanation for this component is provided below). If this data is not available accuracy/uncertainty provided by the manufacturer shall be used (only up to two years).

U3: Uncertainty due to resolution of the Device/Unit under Calibration.

U4: Uncertainty due to other applicable influential factors such as temperature, power supply regulation, voltage coefficient etc affecting the measurements.

For the purpose of CMC evaluation refer **NABL 143** "Policy on Calibration and Measurement Capability (CMC) and Uncertainty in Calibration"

**LONG TERM STABILITY:** Long term stability data shall be generated by laboratories by preparation of control /trend charts based on successive calibration of their standard(s)/master(s) (preferably without adjustments)\*. This shall be established by laboratories within two years based on minimum four calibrations from the date on which laboratories apply for NABL accreditation. For the accredited laboratories, this shall be established within a period of two years w.e.f. the date of issue.

The laboratories may need to get their standard(s)/master(s) calibrated more frequently to generate the stability data within the above stipulated time.

Till two years, the stability data provided by the manufacturer of the standard(s)/master(s) can be utilized for estimation of uncertainty. In case the stability data from the manufacturer is also not available, the accuracy specification as provided by the manufacturer can be used. However, manufacturer's data will not be acceptable after the two year period as mentioned above since the laboratories are expected to establish their own stability data by that period.

\*In cases where the standard(s)/master(s) are adjusted during its calibration pre- adjustment data needs to be used for preparation of control/trend charts.

## 6. Reference Standards

### 6.1 Minimum capabilities a lab shall possess in terms of equipment's in order to get accreditation is:

### 6.2 List of equipment/ process for which accreditation shall not be granted:

- a) Reference Multimeters having < 5 ½ Digit Display.
- b) Clamp on meters/ Clamp Meter with DMM as Standard for measuring capability of high current.
- c) Measuring (DC Volt/Current & Resistance) Capability of Temperature Process calibrator used for Temp. Simulation.

**Note:** *If calibrated DMM is using as Null detector for calibrating Source by Comparison Method, same to be recommended in sourcing capability.*

- a) Calibration of Stop watches using identical stop watch. For that lab should have Digital time interval Meter/Counter.
- b) Counter meter Calibration.

### 6.3 Source calibration using Calibrated DMMs by comparison method to be recommended in Measure Mode & a footnote may be mentioned in form 73 that lab can calibrate sources with the recommended scope.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 125 of 133

- 6.4 Verification of Current Coil along with master calibrator (set up) should be done at least once in 2 years using Clamp meter.
- 6.5 HV Probe shall be calibrated along with DMM.
- 6.6 For Energy Calibration, Time should be an integrated system with source.
- 6.7 Tan Delta bridge calibration using Tan Delta Calibrator is not acceptable. However, for the complete calibration of Tan Delta Bridge, lab should have rated capacitance to cover the range.
- 6.8 Transformer turns ratio meter using TTR Calibrator is not acceptable. However, same can be done using V/V Method.
- 6.9 Lab should have compensating cable (Thermocouple wise) for Temperature Simulation thermocouple wise.
- 6.10 Potential Transformer (PT) and Current Transformer (CT) calibration shall be done with Burden Box of suitable ratings.
- 6.11 For Temperature Simulation CMC (Coarser) to be recommended in single value.

**7. Calibration interval**

	Reference Equipment	Recommended interval
a.	Calibrators (for calibration of 4 ½ Display DMMs) and 6½ Display DMMs for calibration of sources. Calibrator 6½ Display (which can be used to calibrate 6½ Display DMM if TUR ratio is better than 1:3 for all the parameters.), and all other electronics instruments. Note: Periodicity can be extended by once only for one year based on control chart of last 4 to 5 calibrations if they are not adjusted during calibration & also in all previous calibrations.	1Year
b.	Passive equipments such as CT/PT fixed single value resistances inductors and capacitors boxes	2 year
c.	3 Phase Power and Energy System Note: If lab is having different equipment for Testing and Calibration, periodicity of calibration can be 2 year.	1 year

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

**8. NATIONAL/ INTERNATIONAL STANDARDS, REFERENCES AND GUIDELINES**

- IS 1248 - Part 9 Analog Indicating Instruments Voltmeter, Ammeter, Ohmmeter, Frequency meter, Power Meter, PF Meter.
- IS 13875 – Part 1 & 2 Digital Indicating Instruments. Voltmeter, Ammeter, Ohmmeter, Frequency meter, Power Meter, PF Meter
- IS 13779, (Class 1 & 2) IS 14697 (Class 0.2 & 0.5) IS 12346 (Master Equipment requirements) -Energy Meter
- Euromet Guide CG-07 for Calibration of Oscilloscopes.
- NIST 960-12 Timer and Stop watch Calibrations (Though committee has decided not to allow stop watch as master equipment)
- Euromet Guide – CG-11 Calibration guide for Temperature simulation.

**Note: - Latest versions of relevant standard(s) should be followed**

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 126 of 133

## 5. Thermal Calibration

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 127 of 133



## 1. Recommended General Requirements

- Temperature: In-house: 25°C± 4°C (or better depending on the sensitivity of the measurements)
- RH: 30% to 75%.
- Earth Resistance: < 1Ω (Shall be measured and recorded at least once in a year.)
- N-E Voltage: < 1 V
- THD: <5% (Ref.std: IS:13875, IS: 4722)
- Acoustic noise: <60dBA.
- Power Supply regulation: ±2 % or Better on Calibration Bench.
- Frequency: 50 Hz ±1 Hz.
- The recommended level of illumination is 250-500 lux on the working table.
- The calibration laboratory shall make arrangements for regulated and uninterrupted power supply of proper rating. The recommended voltage regulation level is ±2% or better, and Frequency variation ± 2.5Hz or better on the calibration bench.
- Specification SP 31- 1986, a special publication in the form of a wall chart, giving the method of treatment in case of electric shock, should be followed. The chart shall be placed near the power supply switchgear and at other prominent places as prescribed under Indian Electricity Rules 1956.
- Effective mains earthing shall be provided in accordance with relevant specification IS: 3043. This shall be periodically checked to ensure proper contact with earth rod.
- Laboratory using fluids (oil/alcohol used in the calibration bath) may produce noxious or toxic fumes under certain circumstances. Fluid manufacturer's MSDS (Material Safety Data Sheet) may be referred. Exhaust hood of sufficient capacity should be provided to pull oil fumes away from the operator.

## 2. Recommended Facility of the Scope

Specific Requirements for Thermal calibration with following details.

Sl.	Description	Relevant Standard/ Guidelines	Permanent facility	Onsite calibration	Mobile facility
Temperature					
1	SPRT, PRT using Fixed Point Cell	ITS -90	√	X	X
2.	RTD sensors with/without indicator, Temperature Transmitter with/without indicator	DKD-R-5-1	√	√	√
3.	Thermocouple with/without indicator	ASTM E220-13/EURAMET cg-8	√	√	√
4.	Liquid-In-Glass Thermometer, Dial Temperature Gauge	IS 6274, IS 2480, OIML R 133	√	√	√
5.	Indicator of Liquid bath, Furnace, Oven, Freezer, Dry block Bath, Cold Room, Chamber	DKD-R5-7	√	√	X
6.	Liquid bath, Furnace, Oven, Freezer, , Cold Room, Environmental Chamber	IEC 60068 (Part 3-6), Part 11, DKD-R5-7	√	√	X
7.	Dry block Bath	EURAMET cg -13	√	√	X
8.	Infrared radiation thermometer/ Pyrometer	MSL technical guide 22, VDI/VDE 355 Part 4.3	√	√	√

### National Accreditation Board for Testing and Calibration Laboratories

Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 128 of 133

9.	Black Body Source		√	√	√
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Relative Humidity					
1.	Humidity Sensor/ Transducer/Transmitter with Indicator		√	√	√
2.	Indicator of Humidity Chamber, Environmental Chamber	DKD –R5-7	√	√	X
3.	Humidity Chamber, Environmental Chamber	DKD-R5-7	√	√	X

**Note 1:** This technical requirement is based on above referred standard taking into account only the salient features required during calibration. Lab may follow any relevant standard; however care shall be taken to follow the requirements in totality.

**Note 2:** Larger effective Volume of chamber should not be calibrated for single position value. Only multi position (Mapping) is allowed.

Sl.	Effective Volume of Chamber	No. of Sensor used	Reference standard
1.	≤2000 l m <sup>3</sup>	Minimum 9	IEC60068-3-5
2.	>2000 l m <sup>3</sup>	Minimum 15	IEC60068-3-5

### 3. Selection of Reference Standards

- RTD Standard should be minimum Class A or better (4 wire)
- Thermocouple should be of noble metals like R, S and B.
- For Temperature Mapping base metal thermocouple (preferably N Type) are acceptable (AMS 2750/ API 20H)
- Certified Humidity salt Solutions, Humidity Chamber for RH probe Calibration.
- Reference RH probe with indicator for RH Probe calibration.

**Note:** In case of thermohygrometer, temperature and humidity may be calibrated separately as per the desired range.

### 4. Calibration Interval

Reference Equipment	Recommended interval
Triple Point of water (Fixed Cells)	1 year
SPRT,PRT with/without Indicator	1 year
Thermocouple with/without Indicator	1 year
Pyrometer	1 year
Humidity Indicator with Sensor	1 year

Note: Recommendation is based on present practice of NPL, India. However, laboratory may refer ISO 10012 and ILAC G24 for deciding the periodicity of calibration other than above recommendations.

### 5. Legal Aspects

Calibration of Liquid-in-Glass Thermometer done by any accredited laboratories is meant for scientific and industrial purpose only. However, if used for commercial trading, additional recognition/ approval shall be complied as required by Dept. of Legal Metrology, Regulatory Bodies, etc.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 129 of 133

## 6. Calibration methods

### a. Fixed Point Method

This absolute method is used for the realization of the International temperature scale, ITS-90. The thermometer is calibrated by measurements at a series of temperature fixed points: e.g. freezing/melting points, triple points, vapour pressure points. This method consists in setting up a thermometer in a fixed-point cell that provides an isothermal environment. Methods for highly accurate fixed point realizations are described in detail in "Supplementary Information for the ITS-90" [CCT (1990)]

### b. Comparison Method

A reference thermometer and the thermometer/thermocouple to be calibrated are immersed in a suitable, stirred liquid bath (organic liquid, water, oil or salt) or put in a suitable laboratory furnace, and the readings of the thermometers are compared. The choice of calibration temperatures should cover the range which is used in the temperature measurement. It is important that the sensing head of the thermometer is calibrated and that of the reference thermometer is at the same temperature. Metal blocks which have drilled holes for thermocouples, or other sensing elements, can be used in furnaces for homogenizing the temperature during calibration. If two thermocouples are compared in calibration, it is important to use a stable reference junction temperature.

All the Thermal sources shall be studied/known for their stability and uniformity data periodically at least once in a year, in order to use the data to evaluate uncertainty in the measurement.

For calibration of relative humidity sensor, the sensor should be placed in the chamber of the humidity generator, the test chamber should be maintained at a stable temperature and measurements are obtained when thermal and water vapor pressure equilibrium conditions are reached. For psychrometer calibration, distilled water should be used in the reservoir of the sensor head.

### Non-contact Calibration by Radiation thermometry/Pyrometry

The laboratories establishing with calibration facilities for Infrared radiation thermometer/pyrometers shall satisfy the following conditions/guidelines.

- i. The thermal source used for the calibration should satisfy the condition of blackbody having a known emissivity in order to confirm suitable temperature as measured by standard IR-radiation thermometer.
- ii. It should be preferred to calibrate a radiation thermometer against a standard radiation thermometer using a blackbody radiation source and not against an ordinary furnace/thermal heating source. Lab may follow any other relevant standard, however care shall be taken to follow the requirements in totality
- iii. The thermal uniformity and stability shall be measured and evaluated by non-contact technique in order to confirm accuracy in temperature measurement.

### Calibration of thermal sources

When calibration of thermal sources (Baths/Furnace/Blackbody source/Dry block calibrators) is required to be performed by a laboratory, the following guidelines are to be followed:

- i. For calibration procedure some national/international standard should be followed.
- ii. For the calibration of source, the stability and uniformity are the two major and specific parameters which are really in practice used.
- iii. Calibration of its set temperature and digital display values shall also be calibrated for effective operation of the source. However, these parameters are not important/ required to be used for practical application of uncertainty evaluation.

### Calibration of Liquid-in-Glass Thermometers:

There are specific international and national documentary standards (IS specifications) available for calibration of glass thermometers. These are two different types of liquid-in-glass thermometers. One the solid stem and the other enclosed scale stem type of glass thermometers. Similarly according to the practical use, there are further three different categories called as Total Immersion or full immersion thermometers (TI), the Partial Immersion (PI) thermometers and the Complete Immersion (CI) thermometers.

1. Calibration of LIG Thermometers shall not be calibrated on Dry well calibrator sources.
2. Only Temperature liquid baths are employed for glass thermometers.

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 130 of 133

3. Total Immersion or Partial Immersion thermometers are calibrated as per their condition of immersion only.
4. For calibration of total immersion thermometers, Liquid baths of high depth (~450mm to 500mm) are required. These cannot be calibrated on shallow depth baths.
5. Partial Immersion LIG thermometers are required to be calibrated at specified immersion condition of thermometer stem as marked or mentioned over the stem.
6. In the calibration of Glass thermometers, readings shall be observed by using a Reading Telescope with suitable magnification (x10 or x20) and not by a magnifying glass. This avoids the parallax error giving faulty reading while reading the thermometer.

## 7. Recommended Example of Measurement Uncertainty

Sl.	Description	Uncertainty components
1	SPRT, PRT using Fixed Point Cell	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard (s)</li> <li>• Drift of reference standard(s)</li> <li>• Resolution of readout</li> <li>• Uncertainty of resistance measuring unit (Bridge/DVM)</li> <li>• Uncertainty due to heating effect</li> <li>• Uncertainty due to immersion error</li> <li>• Uncertainty due to choice of fixed point (for FP realization)</li> <li>• Uncertainty due to purity of fixed point material.</li> <li>• Uncertainty due to propagation of error due to TP water.</li> <li>• Uncertainty due to atmospheric pressure variation of the cell</li> <li>• Uncertainty due to hydrostatic head of the FP cell.</li> </ul>
2.	RTD sensors with/without indicator, Transmitter with/without indicator	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard (s)</li> <li>• Uncertainty of readout unit in case of sensor calibration</li> <li>• Drift of reference standard(s)</li> <li>• Resolution of readout</li> <li>• Stability of temperature source</li> <li>• Uniformity of temperature source</li> <li>• Self-heating error</li> <li>• Immersion depth</li> </ul>
3.	Thermocouple with/without indicator	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard (s)</li> <li>• Uncertainty of readout unit in case of sensor calibration</li> <li>• Drift of reference standard(s)</li> <li>• Resolution of readout</li> <li>• Stability of temperature source</li> <li>• Uniformity of temperature source</li> <li>• Stability of ice point for reference junction</li> <li>• Variation error in the ice point</li> <li>• In-homogeneity of thermocouple</li> </ul>
4.	Liquid-In-Glass Thermometer, Dial Temperature Gauge	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard (s)</li> <li>• Uncertainty of readout unit in case of sensor calibration</li> <li>• Drift of reference standard(s)</li> <li>• Resolution of Liquid-In-Glass Thermometer /readout</li> <li>• Stability of temperature source</li> <li>• Uniformity of temperature source</li> </ul>

### National Accreditation Board for Testing and Calibration Laboratories

Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 131 of 133

5.	Indicator of Liquid bath, Furnace, Oven, Freezer, Dry block Bath, Cold Room, Environmental Chamber	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard (s)</li> <li>• Drift of reference standard(s)</li> <li>• Temperature Gradient/Temperature Fluctuation</li> <li>• Resolution of indicator of source</li> </ul>
6.	Liquid bath, Furnace, Oven, Freezer, , Cold Room, Environmental Chamber	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard (s)</li> <li>• Drift of reference standard(s)</li> <li>• Temperature Gradient/stability</li> <li>• Uniformity of source to be calibrated</li> <li>• Resolution of readout</li> <li>• wall radiation effect according to various sensor constructions</li> <li>• loading effect due to standard and accessories</li> </ul>
8.	Infrared radiation thermometer/Pyrometer	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard pyrometer(s)</li> <li>• Drift of reference standard pyrometer (s)</li> <li>• Resolution of pyrometer under calibration</li> <li>• Stability of temperature radiation source</li> <li>• Uniformity of temperature source</li> <li>• Uncertainty due to emissivity correction of the source</li> <li>• Uncertainty due to size of source effect</li> </ul>
9.	Black Body Source	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard (s)</li> <li>• Drift of reference standard(s)</li> <li>• Temperature Gradient/stability</li> <li>• Uniformity of source to be calibrated</li> <li>• Resolution of readout</li> <li>• Emissivity variation of the radiation source</li> <li>• Size of source effect</li> </ul>
10.	Humidity Sensor/ Transducer/Transmitter with Indicator	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard hygrometer(s)</li> <li>• Drift of reference standard hygrometer (s)</li> <li>• Resolution of readout hygrometer</li> <li>• Stability of humidity generator/source</li> <li>• Uniformity of humidity generator/source</li> </ul>
11.	Indicator of Humidity Chamber, Environmental Chamber	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard hygrometer (s)</li> <li>• Drift of reference standard hygrometer</li> <li>• Humidity Gradient/Fluctuation</li> <li>• Resolution of humidity source</li> </ul>
12.	Humidity Chamber, Environmental Chamber	<ul style="list-style-type: none"> <li>• Uncertainty in Measurement repeatability</li> <li>• Uncertainty of reference standard hygrometer (s)</li> <li>• Drift of reference standard hygrometer (s)</li> <li>• Temperature Gradient/stability of humidity source</li> <li>• Uniformity of source to be calibrated</li> <li>• Resolution of indicator of the RH source</li> </ul>

National Accreditation Board for Testing and Calibration Laboratories				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 132 of 133

**8. Recommended National/ International Standards, References and Guidelines**

- IS: 6274 - method of calibrating Liquid-In Glass Thermometer.
- IS: 7358 – Specification of thermocouple.
- EURAMET cg-8: Calibration of Thermocouple
- ASTM E220 13: Standard Test method for calibration of Thermocouple by comparison method.
- DKD-R5-1: Calibration of Resistance Thermometer.
- DKD-R5 -7: Calibration of Climate chamber
- API (6) standard 20H: Calibration of Industrial Furnace
- JCGM 200 International vocabulary of metrology – Basic and general concepts and associated terms (VIM)
- ISO 10012 Part 2: Measurement management systems —Requirements for measurement processes and measuring equipment.
- MSL technical Guide 22: Calibration of Low temperature Infrared Thermometers
- VDI/VDE 3511 Part 4.3 – High Temperature

Note: Latest versions of relevant standard(s) should be followed

<b>National Accreditation Board for Testing and Calibration Laboratories</b>				
Doc. No: NABL 129	Specific Criteria for Accreditation of Calibration Laboratories (Mechanical, Fluid flow, Radiological, Electro-Technical & Thermal Calibration)			
Issue No: 01	Issue Date: 02-Apr-2019	Amend No: --	Amend Date: --	Page 133 of 133

**National Accreditation Board for Testing and Calibration Laboratories (NABL)**

**NABL House**

Plot No. 45, Sector- 44,  
Gurugram – 122002, Haryana

Tel.: +91-124 4679700

**Fax: +91-124 4679799**

Website: [www.nabl-india.org](http://www.nabl-india.org)