NATIONAL ACCREDITATION BOARD FOR
TESTING AND CALIBRATION
LABORATORIES

SPECIFIC CRITERIA for CALIBRATION LABORATORIES IN MECHANICAL DISCIPLINE : Density and Viscosity Measurement

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1. General Requirement

- The purpose of this document is to specify requirements with which a laboratory has to operate and demonstrate its competency to carry out calibration in accordance with ISO/IEC 17025:2005.

- To achieve uniformity between the laboratories, assessors and assessment process in terms of maximum permissible error, CMC, measurement uncertainty etc in line with National/International standards.

- To achieve uniformity in selection of equipment's, calibration methods, maintaining required environmental conditions, personnel with relevant qualification and experience.

1.1 Scope

This specific criteria lays down those specific requirements in measurement of Density and Viscosity under mechanical discipline. This part of the document thus amplifies the specific requirements for measurement of density and viscosity and supplements the requirements of ISO/IEC 17025:2005.

1.2 Calibration and Measurement Capability (CMC)

1.2.1 CMC is one the parameters that is used by NABL to define the scope of an accredited calibration laboratory, the others being parameter/quantity measured, standard/master used, calibration method used and measurement range. The CMC is expressed as "the smallest uncertainty that a laboratory can achieve when calibrating the best existing device". It is an expanded uncertainty estimated at a confidence level of approximately 95% corresponding to a coverage factor k=2.

1.2.2 For evaluation of CMC laboratories should follow NABL 143 - Policy on Calibration and Measurement Capability (CMC) and Uncertainty in Calibration.

1.3 Personnel, Qualification and Training

1.3.1 Technical Personnel:

1.3.1.1 Qualification required for carrying out calibration activity:

The following are only specific requirement. However, qualification and experience will not be the only criteria for the required activity. They have to prove their skill, knowledge and competency in their specific field of calibration activity.

a) B.E / B.Tech or M.Sc. (having Physics as one of the subject) degree with 3 months experience in Basics of Mass Metrology, Volume Calibration & Density Measurement.

b) B.Sc (with Physics as one of the subject) or Diploma with 6 months experience in Basics of Mass Metrology, Volume Calibration & Density Measurement.

c) ITI with 1 year of experience in Basics of Volume Calibration.

1.3.1.2 Training and experience required:

a) Training may be external/ internal depending on the expertise available in the fields.

b) Training in Mass Metrology, Volume Calibration & Density Measurement and in Uncertainty Measurements, CMC including statistical analysis for Technical Manager.

c) Experience and competence in Mass metrology, Volume calibration & Density measurement.

d) Sufficient knowledge about handling of reference equipment, maintenance, traceability, calibration procedure and effect of environmental conditions on the results of calibration.

e) During training calibration activity should be done under supervision.

1.3.2 Authorised signatory

1.3.2.1 Qualification required for interpretation of results and signing the calibration certificates:

The following are only specific guidelines. However, qualification and experience will not be the only criteria for the required activity. They have to prove their skill, knowledge and competency in analysis and interpretation of calibration results.

a) B.E / B.Tech or M.Sc. (with having Physics as one of the subject) degree with 6 months experience in Mass metrology, Volume calibration & Density measurement.

b) B.Sc. (with Physics as one of the subject) or Diploma with 1 year experience in Mass metrology, Volume calibration & Density measurement.

1.3.2.2 Training and experience required:

a) Training may be external/ internal depending on the expertise available in the field.

b) Training, Experience and Competence in Volume calibration and Training in Uncertainty Measurements, CMC including statistical analysis for Technical Manager.

c) Sufficient knowledge and competence in effective implementation of ISO/IEC 17025, specific criteria and NABL guidelines.

d) Competency in reviewing of results, giving opinion and interpretations.

e) During training the relevant activity has to be done under supervision.

1.4 Accommodation and Environmental Conditions

Accommodation and environmental conditions adversely affect the results of calibration and measurement accuracy unless they are controlled and monitored. Hence, they play a very important role.

The influencing parameters may be one or more of the following i.e. temperature, relative humidity, atmospheric pressure, vibration, acoustic noise, dust, air currents/draft, illumination (wherever applicable), voltage fluctuations, electrical earthing and direct sunlight etc., depending on the nature of calibration services provided. The variables described above can play a major factor on calibration results.

The laboratories are advised to follow the requirement of accommodation and environment depending on the types of services provided as recommended:

- By the manufacturers of the reference equipment
- By the manufacturers of the Unit under calibration
- As specified in the National/ International Standards or guidelines followed for the calibration

The environmental monitoring equipments used should also meet the requirement of manufacturers' recommendations and specifications as per the relevant standards followed.

If, accommodation and environmental conditions are not specified either by manufacturer or by National/International standards / guidelines, the laboratory shall follow the below recommendations.

1.4.1 Vibration

The calibration area shall be 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.

1.4.2 Acoustic Noise

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.

1.4.3 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 250-500 lux on the working table.

1.4.4 Environmental Conditions and Monitoring

The environmental conditions for the activity of the laboratory shall be such as not to adversely affect the required accuracy of measurement. Facilities shall be provided whenever necessary for recording temperature, pressure and humidity values prevailing during calibration. The atmospheric conditions maintained in the laboratory during calibration shall be reported in the calibration report/ certificate.

1.5 Special Requirements of Laboratory

1.5.1 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 ± 2.5 Hz or better on the calibration bench.

1.5.2 The reference standards shall be maintained at temperatures specified for their maintenance on order to ensure their conformance to the required level of operation.

1.5.3 The laboratory shall take adequate measures against dust and external air pressure.

1.6 Safety Precautions

1.6.1 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.

1.6.2 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.

1.6.3 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.

1.7 Other Important Points

1.7.1 **Entry to the Calibration Area**: As possible, only the staff engaged in the calibration activity should permitted entry inside the calibration area.

1.7.2 **Space in Calibration Area**: The calibration Laboratory shall ensure adequate space for calibration activity without adversely effecting the results.

1.8 Proficiency Testing

To give further assurance to the accuracy or Uncertainty of measurements, a laboratory will be required to participate, from time to time, in Proficiency Testing Program. The laboratory shall remain prepared to participate in the Proficiency Testing Program through inter-laboratory, inter-comparison schemes wherever it is technically feasible. (Ref. NABL 162, 163 and 164 for further details)

2. Specific Requirements – Measurement of Density of Solids

2.1 Scope: Measurement of Density of Solids

Specific Requirements for Calibration

Sl. No	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.

2.2 National/ International Standards, References and Guidelines

- **OIML G 14, 2011** Density measurement.
- **OIML R111-1, 2004** Metrological and technical requirement of weights classes E_1, E_2, F_1, F_2, M_1, M_2, M_3.
- **OIML R111-2, 2004** Weights Classes E_1, E_2, F_1, F_2, M_1, M_2, M_3- Test report format.
- **OIML R76-1, 2006** Metrological and technical requirements – Non automatic weighing instruments.
- **OIML R76-2, 2007** Non automatic weighing instruments – Test report format.

2.3 Metrological Requirements

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

2.4 Terms and Definitions

Density

- Density is the ratio of mass of an object to its volume.

Equation for Density

- Basic formula for Density = m/V.

  Where m= mass and V= volume

SI Units of Density

- kg/m^3 or g/cm^3

Specific Gravity

- It is the ratio of density of an object to density of tripled distilled water at standard reference temperature.

- Basic formula for specific gravity = density of an object/density of water. When the material has a specific gravity of 0.95 or less we call the material buoyant. When the specific gravity of the material is between 0.95 to 1.05 we call the material as semi buoyant. When the material has a specific gravity more than 1.05 the material will sink.

Resolution (Readability)

- The resolution (readability) is expressed as a portion of the capacity. For example a weighing Instrument with a capacity of 3000 g and a resolution (readability) of 0.1g has a resolution (readability) of 1 part in 30000.

2.5 Selection of Reference for Calibration Density of Solids

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

2.6 Calibration Interval

Recommended calibration interval for reference equipments.

Reference Equipment	Interval
Analytical Balance	1 Year
Thermometer	1 year

2.7 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.

2.8 Environmental Conditions

The ambient temperature shall be (18°C to 27 °C) ± 2 °C.

2.8.1 Recommended Environmental Monitoring Equipments for Density Apparatus

- Temperature with a resolution of 0.1^0C
- Humidity With a resolution of 1% RH
- Barometer with a resolution of 0.1 mbar

However, laboratory shall evaluate the requirement of accuracy, resolution and uncertainty depending on the CMC aimed at.

2.9 Calibration Methods

There are two methods for determination of density solids:

2.9.1 Hydrostatic weighing method: Usually the sample of solid is weighed in Air and weighed in water or known reference density liquid and the difference in weighing is converted in to volume and the Refer the standard OIML G14 for further details. For determination of Standard weights OIML R-111-1 shall be followed.

2.9.2 Density determination using pycnometer (which is of known volume).

2.10 Calibration Procedures

2.10.1 Determination of Density by Hydrostatic Weighing Method:

2.10.1.1 The density of a solid is determined with the aid of a liquid whose density ρ_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 ρ can be calculated from the two weighings as follows:

To find the density of the solid

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

2.10.1.2 To find the volume of the solid

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

Where, ρ = Density of the sample
A = Weight of the sample in air
B = Weight of the sample in liquid
V = Volume of the Sample
ρ_L = Density of the auxiliary liquid
ρ_a = Density of air
ρ_m = Density of mass of the solid

2.10.2 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 $Vw = m_w/\rho_w$

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

Density of the solid matter can be calculated as,

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

2.11 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

2.12 Evaluation of CMC (as per mass)

2.12.1 Refer NABL 143 for CMC evaluation.

2.12.2 CMC value is not the same as expanded uncertainty reported in the calibration Certificate/Report. CMC values exclude the uncertainties which are attributed to the DUC (Device under calibration).

2.12.3 For the purpose of CMC evaluation the following components should be considered.

- Repeatability-standard deviation of weighing result (for minimum 10 readings).
- Uncertainty of the reference standard weight.
- Drift in reference standard weight.
- Uncertainty due to air buoyancy correction.
- Uncertainty due to resolution of Balance.

2.13 Sample Scope

An illustrative example: Correct Presentation of Scope

Laboratory: XYZ						Date(s) of Visit:	
Discipline: Mechanical							
Sl	**Parameter* / Device under calibration**	**Master equipment used**	**Range(s) of measurement**	**Calibration and Measurement Capability ****			**Remarks+/ Method used**
				Claimed by Laboratory	**Observed by Assessor**	**Recommended by Assessor**	
1	Density of Solid	Standard weights, Weighing Balance and Known Density distilled water with density kit	1000 kg/m^3 to 9000 kg/m^3	10 kg/m^3	7 kg/m^3	10 kg/m^3	As per OIML G 14, 2011

** Only for Electro-technical discipline; scope shall be recommended parameter wise (where applicable) and the ranges may be mentioned frequency wise.*

*** NABL 143 shall be referred for the recommendation of CMC*

+ Remarks shall also include whether the same scope is applicable for site calibration as well. NABL 130 shall be referred while recommending the scope for site calibration.

Signature, Date & Name of Lab Representative	Signature, Date & Name of Assessor(s)	Signature, Date & Name of Lead Assessor

2.14 Key Points

2.14.1 The laboratory has to demonstrate the CMC values of 1/3rd of the accuracy specified by the manufacturer.

2.14.2 Demonstration of any CMC values doesn't automatically qualify for granting accreditation until the lab satisfies the stipulated requirement given above.

3. Determination of Density of Liquids and Calibration of Density Hydrometers

3.1 Scope: Determination of Density of Liquids and Calibration of Hydrometers

3.1.1 Specific 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.

3.2 National/ International Standards, References and Guidelines

- **OIML G14, 2011** Density measurement
- **IS 3104 -1982 (RA 2008),** Specification for density hydrometers Part –II methods of test & use.
- Recommended guideline for calibration of Hydrometer NIST special Publication No. 250-78.
- **IS 9585 – 1980 (RA 2003),** Specification for Lactometer.
- **Published Article on "Calibration of Hydrometers"** by Mr. Tripurari Lal, Ex Scientist G and Head Mass standards, NPL India, New Delhi.
- **Published Article on "Calibration of Hydrometers by Cuckow's Method"** by Mr. Tripurari Lal, Ex Scientist G and Head Mass standards, NPL India, New Delhi.

3.3 Metrological Requirements

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

3.3.2 The general classifications of Density Hydrometer are as follows:

L20: long scale length, range 20 kg/m^3, the density interval 0.2 kg/ m^3 with 100 graduations
L50: long scale length, range 50 kg/m^3, the density interval 0.5 kg/ m^3 with 100 graduations
M50: Medium scale length, range 50 kg/m^3,the density interval 1 kg/ m^3 with 50 graduations
M100: Medium scale length, range 100 kg/m^3,the density interval 2 kg/ m^3 with 50 graduations
S50: Short scale length, range 50 kg/m^3, the density interval 2 kg/ m^3 with 25 graduations
S50SP: Short scale length, range 50 kg/m^3, the density interval 1 kg/ m^3 with 50 graduations

3.4 Terms and Definitions

Density

- Density is the ratio of mass of an object to its volume.

Equation for Density

- Basic formula for Density = m/V.

  Where m= mass and V= volume

SI Units of Density

- kg/m3 or g/cm3

Density Hydrometer

- A device used to determine density of liquids. The scale of these hydrometers indicates the density in the range of 600 to 2000 kg/m3. These indications are supposed to be correct at a particular temperature and they are categorized as high, medium and low, also refers to within which surface tension range they are supposed to be used. The highest mark on the hydrometer scale refers to the lowest density and the lowest mark on the scale to highest density. The difference between these two marks decides the range of the hydrometer.

Specific Gravity

- It is the ratio of density of an object to density of tripled distilled water at standard reference temperature.

- Basic formula for specific gravity = density of an object/density of water. When the material has a specific gravity of 0.95 or less we call the material buoyant. When the specific gravity of the material is between 0.95 to 1.05 we call it as semi buoyant. When the material has a specific gravity more than 1.05 the material will sink.

Specific gravity Hydrometer

- A device used to determine directly the specific gravity of a liquid. The level of the hydrometer coinciding with the surface of the liquid indicates the no. of times the liquid is heavier or lighter than water, which is specific gravity of the liquid. The hydrometer is based on Archimedes' principle. The level at which the hydrometer floats depends on the density of liquid. The specific gravity hydrometer bears two reference temperatures in the form t1/t2 where t1 refers to the temperature of liquid and t2 that of water. Thus on a specific gravity hydrometer in addition to the specific gravity values the two reference temperatures are also defined.

Lactometer

- Lactometer is used to check concentration of milk.

Alcoholometer

- An alcoholmeter is a hydrometer which is used for determining the alcoholic strength of liquids.

Specific Gravity Meter

- To read specific gravity of any liquid.

Pycnometer (pyknometer)

- Is a device used to measure the density of liquids. It is usually made of glass.

Brix Hydrometer

- To measure percentage of sugar by weight in a solution.

Baume Hydrometer

- To measure density of Oil.

Twaddle Hydrometer

- To measure density of liquids higher than water.

Sikes Hydrometer

- To measure the strength of spirits to levy Excise duty on production of alcohol.

Hydrometer for Liquid Petroleum

- To measure density of petroleum products lighter than water.

Resolution

- The readability expressed as a portion of the capacity. For example a weighing Instrument with a capacity of 3000g and a readability of 0.1g has a resolution of 1 part in 30000.

Reference Temperature

- It is the temperature at which the hydrometer, viscometer, lactometer and alcoholmeter is intended to measure the density of respective liquids.

3.5 Selection of Reference for Calibration of Hydrometers

3.5.1 Hydrostatic Weighing (Cuckow's) Method

Weighing Balance, Standard weights, Temperature Sensor with indicator, Tridecane, Water bath, Laser light etc.

3.5.2 Comparison Method based on Archimedes Principle

The calibration set up consists of: Standard Hydrometers, Standard liquid of known density and other support equipment.

3.5.2.1 Standard Hydrometers: Precise hydrometers with known scale error and the graduations preferably similar to the hydrometer under test are to be selected.

3.5.2.2 Standard Liquids: The standard liquids of known density and preferably with low surface tension are to be used for calibration of hydrometers. The low surface tension liquid wet the hydrometer stem reliably and thereby reduces the variation due to contamination. In comparison these liquids need not have the traceable calibration status. Some of the standard liquids used in calibration of Hydrometers are Petroleum ether, Tetra Chloromethylene, Xylene and Methylene bromide etc.

3.5.2.3 Support Equipment

Hydrometer Vessel: Rectangular Jar which stands firmly on its base and preferably with optical walls inside to reduce distortion of the liquid surface.

Stirrer: To stir thoroughly the reference liquid in the vessel to remove the entrapped air in the liquid. It should be preferably be perforated rectangular metal type.

Thermometer: Long total immersion type thermometer covering a range of -0.5°C to 40.5°C, with a resolution of 0.1°C along with a certificate of scale correction is preferable.

Chamber: The hydrometer vessel set-up should be housed in a closed chamber with exhaust system to suck out the fumes of the liquid used.

Exhaust: To keep the atmosphere reasonably free from toxic fumes that may emerge from the calibration liquids and to draw the vapour downwards thus taking advantage of their high density.

Wooden rectangular board: The board with top half painted black and bottom with white colour with sharp horizontal line to assist in proper reading of the scales.

3.6 Calibration Interval

Recommended interval for reference equipments.

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

3.7 Legal Aspects

Calibration of Viscometer, Hydrometer, Lactometer, and Alcoholmeter 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.

3.8 Environmental Conditions

The ambient temperature shall be within 20°C to 27 °C. The temperature shall be kept constant within ± 2 °C.

3.9 Calibration Methods

3.9.1 There are two methods for determination of density of liquids

a. Using pycnometer (specific gravity bottle)

b. Using a hydrometer by comparison

3.9.2 There are two methods for calibration of hydrometers

a. Hydrostatic weighing (Cuckow's) method

b. 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.

3.10 Calibration Procedures

3.10.1 Density of Liquids using Sinker of known volume

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 ρ can be determined from the two weighing as follows:

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

Where, ρ = Density of the liquid
A = Weight of the sinker in air
B = Weight of the sample in liquid
V = Volume of the Sinker
ρ_L = Density of the auxiliary liquid
ρ_a = Density of air
α = Air buoyancy correction

3.10.2 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

ρ_{tw} = Density of water at temperature t °C (from the table)
C = Buoyancy correction = ρ_{ta} x m_w
ρ_{ta} = Air density

3.10.3 Calibration of Hydrometers

By Cuckow's Method

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

i) In the case the hydrometer floats free in a liquid of ρ_x density at reference temperature of T_0, the equilibrium equation is:

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

Where,
m - mass of the hydrometer,
g - local acceleration due to gravity
D - diameter of stem of hydrometer
ρ_{a1} – density of air at the moment of reading hydrometer's indications
γ_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.

ii) 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:

$$\mathbf{M_a x g \left(1- \rho_{a2}/ \rho_w\right) = m x g - g^*(V+v)^* [1+\beta(T_2-T_0)] \rho_{a2}} \qquad (2)$$

Where, M_a - mass of the hydrometer in air
ρ_w - Density of standard weight
β - coefficient of cubic expansion of the glass used to construct the hydrometer
T_2 - air temperature during the weighing
ρ_{a2} - density of air during weighing

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

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

iii) The Hydrometer is weighed being partially immersed in a reference liquid of density ρL , at the same mark as in the case (i) at the temperature T3 (thus the volume remains unchanged). Two types of forces against it: the weight of the hydrometer and the force due to surface tension γL 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:

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

Where,

M_L – is the mass of hydrometer in the reference liquid
ρ_{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`≅ g, the equation (4) becomes.

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

3.10.4 Calibration of Hydrometer by Comparison Method

3.10.4.1 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 ρ_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 π DT

Upward forces

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

Under equilibrium, Downward Force = Upward force

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

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.

3.10.4.2 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

3.10.5 Classification of Density Hydrometers:

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

- 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.

3.11 Measurement Uncertainty

3.11.1 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

3.11.2 Comparison method: Contributions of Uncertainty are due to:

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

3.12 Evaluation of CMC

3.12.1 Refer NABL 143 for CMC Evaluation.

3.12.2 CMC value is not the same as expanded uncertainty reported in the Calibration Certificate/Report.CMC values exclude the uncertainties which are attributed to the UUT (Unit under test/calibration).

3.12.3 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.

3.12.4 Comparison Method: Contributions of uncertainty are due to:

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

- Reference standard hydrometer
- Temperature correction
- Surface tension correction
- Meniscus correction

3.13 Sample Scope

An illustrative example: Correct Presentation of Scope

Laboratory: XYZ					Date(s) of Visit:		
Discipline: Mechanical							
Sl	**Parameter* / Device under calibration**	**Master equipment used**	**Range(s) of measurement**	**Calibration and Measurement Capability ****			**Remarks+/ Method used**
				Claimed by Laboratory	**Observed by Assessor**	**Recommended by Assessor**	
1	Density of Liquid	Liquid Density calibration setup including Weighing Balance	700 kg/m^3 to 2000 kg/m^3	5 kg/m^3	4 kg/m^3	5 kg/m^3	IS 3104-1982(part 2) (RA – 2008)
2	Density-Hydrometer (Brix, Baume, Twaddle, Hydrometer for liquid petroleum)	Reference Weights, Weighing balance and density of Distilled water	700 kg/m^3 to 1500 kg/m^3	5 kg/m^3	4 kg/m^3	5 kg/m^3	As per relevant standards

** Only for Electro-technical discipline; scope shall be recommended parameter wise (where applicable) and the ranges may be mentioned frequency wise.*

*** NABL 143 shall be referred for the recommendation of CMC*

+ Remarks shall also include whether the same scope is applicable for site calibration as well. NABL 130 shall be referred while recommending the scope for site calibration.

Signature, Date & Name of Lab Representative	Signature, Date & Name of Assessor(s)	Signature, Date & Name of Lead Assessor

3.14 Key Points

3.14.1 Demonstration of any CMC values doesn't automatically qualify for granting accreditation until the lab satisfies the stipulated requirement given above.

3.14.2 Laboratory shall demonstrate at least the lower grade of hydrometer.

4. Measurement of Viscosity and Calibration of Viscometers

4.1. Scope: Determination of Viscosity and Calibration of Viscometers

4.1.1. Specific Requirements for Calibration

Sl. No	Description	Relevant Standard	Permanent facility	Onsite calibration	Mobile facility
1	Calibration of Master Viscometer	ASTM D2162-13, ASTM D1480, ASTM D1250	√	X	X
2	Determination of kinematic and Dynamic Viscosity of liquids		√	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***.

4.2. National/ International Standards, References and Guidelines

- **ASTM D 2162-13** "Standard practice for basic calibration of Master Viscometers and Viscosity Oil Standards"

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"
- **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"

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 -1982, RA 2005** "Method for Determination of Flow Time for Flow Cups"
- **ISO 2555 :1989** "Plastics –Resins in the Liquid State or as Emulsions or Dispersions **-**Determination of Apparent Viscosity by the Brookfield Test Method"
- **ISO1652:2011** "Rubber Latex- Determination of Apparent Viscosity by Brookfield Test Method"
- **OIML R 69, 1985** "Glass Capillary Viscometers for the Measurement of Kinematic Viscosity-Verification Method"
- **OIML D 17, 1987** Hierarchy Scheme for Instruments Measuring the Viscosity of Liquids.

4.3. 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).

4.4. Terms and Definitions

Viscosity

- Viscosity is a fundamental characteristic property of all fluids. When a liquid flows, it has an internal resistance to flow. It is a measure of this resistance to flow or shear. It can also be termed as drag force and is a measure of functional properties of the liquid. Viscosity is a function of temperature and pressure. Viscosity is expressed two distinctive forms (a) Absolute or dynamic viscosity (b) Kinematic viscosity.

Fluid Density

- Density is defined as the mass of a fixed volume of a substance. Generally when the fluid expands when heated and contract when cooled. Therefore density decreases when there is increase in temperature and increase with decrease in temperature

Specific Gravity

- It is the ratio of density of a sample at a specific temperature to density of some standard at the same temperature.

Dynamic Viscosity

- Dynamic or absolute viscosity is defined as the ratio of the shear stress to the shear rate of a fluid. Dynamic viscosity (η) is measured in mPa.s. An alternative unit in popular usage is the Poise (P) where 10^{-2} P = 1 cP =1 mPa.s.

Kinematic Viscosity

- Kinematic Viscosity is defined as the dynamic or absolute viscosity divided by the density. Kinematic viscosity (v) is reported in mm^2/s. An alternative unit in popular use is the centistrokes (cSt) where 1 cSt = 1 mm^2/s [1 St= 1 cm^2/s.

Viscometer

- Is an instrument used to measure the viscosity of a fluid.

Rheometer

- Is an Instrument which measures the viscosity of liquids which vary with flow conditions.

Type of Viscometers

- U-tube viscometers, Falling sphere viscometers, Falling piston viscometers, Oscillating piston viscometer, Vibration viscometers, Rotational viscometers, Bubble viscometers etc.

Capillary Viscometer

- The principle of operation of the glass capillary viscometer is a known volume of liquid is timed passing through a precision –formed capillary. The volume of oil which passes through the capillary is held in a bulb marked with calibration lines and positioned above the capillary and, when released flows through the capillary under gravity.

Falling Ball Viscometer

- The falling ball viscometer operates on the principle of timing a ball as it falls through the sample. The viscosity is then calculated using the following formula:

$$\mu = k(\rho t - \rho)\, t$$

where,
μ = viscosity in centipoises (cp)
ρt = density of the ball in g/cm^3
ρ = density of the liquid in g/cm^3
t = time of decent in seconds
k = viscometer constant

Rotational Viscometers

- Rotational viscometers measure dynamic viscosity by sensing the torque required to rotate a spindle at constant speed whilst immersed in the sample fluid. The torque is proportional to the viscous drag and thus viscosity.

Mineral Oil

- Any petroleum oil, as opposed to animal or vegetable –based oils.

Newtonian Fluid

- A fluid is which the ratio of shear stress to the rate of shear is constant. Examples are water and thin motor oils.

Flow Cups

- Flow cups are viscometers consisting of a cylinder of fixed volume with an orifice of specified dimensions at the bottom. These are crude measurement devices used for the measurement of the kinematic viscosity of paints, inks etc. Measurements are normally taken at approximately 20°C. Types of flow cups encountered include Zahn, Ford, Shell and ISO. Formulas available in the relevant standards giving the relationship between efflux time and the kinematic viscosity. The use of flow cups for the determination of kinemateic viscosity is limited to Newtonian or near-Newtonian liquids.

Near-Newtonian

- A liquid in which the variation in viscosity with shear rate is small and the effect on viscosity of mechanical disturbances such as stirring is negligible.

Non-Newtonian

- Non-Newtonian fluids are those fluids not having a constant ration of shear stress to shear rate. Examples are lubricating greases, multi grade engine oils and household paints.

Digital Contact Thermometer (DCT)

- An electronic device consisting of temperature measuring sensor in contact with a material that provides an output to a digital display of measured value.

Basic Calibration, n

- Calibration based on the primary standard, water. Pure water has a kinematic viscosity of 1.0034 mm^2/s at 20° C (ISO 3666).

Master Viscometer, n

- Glass capillary viscometer with a liquid driving head of at least 400 mm.

Viscosity Oil Standard, n

- Stable Newtonian liquid, the kinematic viscosity of which has been related to the kinematic viscosity of water through the step-up procedure described in this practice.

4.5. Selection of Reference Equipment

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 ± 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 ± 0.01°C.
- Devices for measurement of flow time of the liquid in a standard or reference viscometer with an error not exceeding ± 0.01s and in a viscometer to be verified with an error not exceeding ± 0.2 s.
- Thermometers for the measurement of the temperature in the thermostatic bath with an error not exceeding ± 0.01°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.

4.6. Calibration Interval

4.6.1. Recommended Calibration Interval

Reference Equipment	Recommended Interval
Temperature measuring devices	1 Year
Timer	1 Year
Master viscometer	1 Year

4.7. Legal Aspects

Calibration of Viscometers 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.

4.8. Environmental Conditions

4.8.1. 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 ± 4 °C.

4.8.2. Recommended Environmental Monitoring System

- Temperature with a resolution of 1°C.

4.8.3. 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.

4.8.3.1. 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:

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

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

4.8.3.2. 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 ± 50 ppm.

4.9. Calibration Procedures

The different types of viscometers are as listed below:

A.1 Calibration of Kinematic Glass Viscometers

- Modified Ostwald Viscometers used for Newtonian liquids upto 20000mm^2/s.

A.2 Constant Volume at Filling Temperature

- Cannon – Fenske Routine Viscometer
- 2.Cannon- Manning Semi Micro Viscometer
- Pinkevitch Viscometer

A.3 Constant Volume at Test Temperature

- Zeitfuchs Viscometer
- SIL Viscometer

- BS/U Tube Viscometer
- BS/U Tube Miniature Viscometer

For more details, construction, operation and calibration set up of above type of viscometers refer Annexure A-1 **of ASTM D446-12** Or Annexure A of **ISO 3105:1994**.

B.1 Suspended Level Viscometer

For transparent and Newtonian liquids upto 100000 mm^2/s.

For more details calibration of above viscometers follow the Annexure –A2 of standard ASTM D446-12 or Annexure B **of ISO 3105:1994**.

C.1 Reverse Flow Viscometer

For opaque & transparent liquids 300000 mm^2/s.

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 3105:1994 Or ISO 3104:1994**.

D.1 Flow cups- as per IS 3944 -1982, RA 2005 Or ASTM D 1200

- For calibration follow IS 3944 -1982, RA 2005 "Method for Determination of Flow Time for Flow Cups" Or ASTM D1200.

E.1 Rotational Viscometers –as per ISO 2555 1989 & ISO 1652 2011

- For calibration follow the standard ISO 2555 1989 "Plastics –Resins in the Liquid State or as Emulsions or Dispersions-Determination of Apparent Viscosity by the Brookfield Test Method".

- For calibration follow the standard ISO 1652 2011 "Rubber Latex-Determination of Apparent Viscosity by Brookfield Test Method"

4.9.1. General procedure for selection of reference viscometer and reference standard viscosity for calibration is as given below. For more details refer annexes of the standards ASTM D446 or ISO 3104: 1994 or ISO3105:1994.

Reference Viscometers

4.9.1.1. Select a clear petroleum oil, free from solid particles and possessing Newtonian flow characteristics, with a kinematic viscosity within the range of both reference and viscometer and the viscometer to be calibrated. The minimum flow time shall be greater than that specified in the appropriate table of the annex in both the reference viscometer and the viscometer which is to be calibrated in order that the kinetic energy correction may be less than 0.2%.

4.9.1.2. Select a calibrated viscometer of known viscometer constant C_1 as follows:

$$C_1 = (t_2 \text{ X } C_2)/t_1$$

Where, C_1 is the constant of the viscometer being calibrated
t_1 is the flow time, to the nearest 0.1 S, in the viscometer being calibrated
C_2 is the constant of the calibrated viscometer
T_2 is the flow time, to the nearest 0.1 S, in the calibrated viscometer

4.9.1.3. Repeat with a second oil whose flow times are at least 50% longer than the first oil. If two values of C1 differ by less than 0.2% for those viscometers listed in Annexes A & B, and less than 0.3% for those viscometers listed in annex C, use the average as the viscometer constant for the viscometer being calibrated, If the constants differ by more than this value, repeat the procedure taking care to examine all possible sources of errors.

4.9.1.4. The calibration constant C, is dependent upon the gravitational acceleration at the place of calibration and this must , therefore, be supplied by the standardization laboratory, together with the instrument constant. Where the acceleration of gravity, g, differs from more than 0.1%, correct the calibration constant as follows:

$$C_2 = (g_2 / g_1)\, C_1$$

Where, g_1 indicates the acceleration due to gravity of the standardization laboratory and g_2 that of the calibration laboratory.

4.9.2. Viscosity Reference Standards

4.9.2.1. Kinematic viscosity reference standards are available in certain countries from national laboratories or other authorized sources. Select a viscosity reference standard with a kinematic viscosity range of the viscometer to be calibrated and a minimum flow time greater than that specified in the appropriate table of the annex. Determine the flow time , to the nearest 0.1s in accordance with ISO 3104, and calculate the viscometer constant, C as follows:

$$C = \nu/t$$

Where, ν is the kinematic viscosity, in mm^2/s for the reference liquid.
t is the flow time, in seconds

4.9.2.2. Repeat with a second reference standard whose flow times are at least 50% longer than the first reference standard. If the two values of C differ by less than 0.2% for those viscometers listed in the annexes A and B and less than 0.3% for those viscometers listed in Annex C, use the average as the viscometer constant for the viscometer being calibrated. If the constants differ by more than this value repeat the procedure, taking care to examine all possible sources of errors.

4.10. Measurement Uncertainty

The relative overall uncertainty in the measurement of kinematic viscosity (Uv) calculated as:

$$U_v = 2 * \sqrt{(S_k)^2 + (S_{9\gamma})^2 + 1/2\,[\,(S_T)^2 + (S_t)^2\,]}$$

S_k - relative uncertainty constant k (from the certificate of calibration).
S_t - relative uncertainty of the time device in test Viscometer.
$S_{9\gamma}$ - relative uncertainty of thermometer, gradient of temperature in the thermostat bath and temperature coefficient of liquid viscosity.
S_T - relative uncertainty of flow time measurements in standard viscometer.

4.11. Evaluation of CMC

4.11.1. Refer NABL 143 for CMC evaluation.

4.11.2. CMC value is not the same as expanded uncertainty reported in the calibration Certificate/Report. CMC values exclude the uncertainties which are attributed to the UUC (Unit under test/calibration).

4.11.3. For the purpose of CMC evaluation of Viscometer the components shall be considered are same as above given at 4.11 since most of the uncertainty components are process related and not of the UUC.

4.12. Sample Scope

An illustrative example: Correct Presentation of Scope

Laboratory: XYZ					Date(s) of Visit:	
Discipline: Mechanical						
Sl	**Parameter* / Device under calibration**	**Master equipment used**	**Range(s) of measurement**	**Calibration and Measurement Capability ** Claimed by Laboratory**	**Observed by Assessor**	**Recommended by Assessor**	**Remarks+/ Method used**
1	**Viscosity** (Capillary Viscometer)	Standard viscometers and Newtonian liquids	**Reverse Flow** (0.001 to 0.5) mm^2/s^2	0.7 %	0.6 %	0.7 %	Reverse Flow at 20°C to 100°C Procedure based on ASTM D446 & ASTM D 2162
	Measurement of Viscometer Constant		**Direct Flow** (0.001 to 5) mm^2/s^2	0.7 %	0.6 %	0.7 %	Direct Flow at 20°C to 100°C Procedure based on ASTM D446 & ASTM D 2162

** Only for Electro-technical discipline; scope shall be recommended parameter wise (where applicable) and the ranges may be mentioned frequency wise.*

*** NABL 143 shall be referred for the recommendation of CMC*

+ Remarks shall also include whether the same scope is applicable for site calibration as well. NABL 130 shall be referred while recommending the scope for site calibration.

Signature, Date & Name of Lab Representative	Signature, Date & Name of Assessor(s)	Signature, Date & Name of Lead Assessor

4.13. 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.

Annexure -1

Master Viscometer Calibration

1. Selection of Reference Equipment

1.1. Two or more master viscometers (Cannon or Ubbelohde type) having calibrating constants in the 0.001 to 0.003 mm^2/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^2/s^2 duly calibrated at 40°C with two standard oils and its calibration factor calculated at standard conditions for water at 20°C.

1.2. Digital Contact Thermometer to meet or exceed following requirements:

- 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.

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.

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.

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.

2. Calibration Procedure

2.1. Calibration of Master Viscometers with Water at 20 °C as per ASTM D 2162-13

Follow clause 4.1 to 4.12 to calculate the viscometer constants as follows:

$$c = 1.0034/t$$

where c= calibration constant of viscometer with water at 20 °C, mm^2/s^2
t = efflux time in seconds

Note 1: Normally master viscometers are calibrated and used in the same location.

Note 2: If, subsequently a viscometer is used at a laboratory other than the calibrating one, c constant should be corrected for the difference in acceleration of gravity 'g' at the 2 locations as follows:

$$c_2 = g_2/g_1 * c_1$$

where,
c_2 & g_2 = calibration constant and gravity at new location
c_1 & g_1 = calibration constant and gravity at original location

Failure to correct a viscometer constant for change of gravity can result in errors as high as 0.5 %, which is more than the error permitted between checks in this test method

Certificates of viscometer shall state the value of 'g' at the location of the calibrating laboratory.

Calibration of Viscosity Oil standards as per 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.

3. Equations for Viscosity Calculation

Kinematic Viscosity Calculation:

a) **For cannon master viscometer**, calculate the kinematic viscosity of the oil standard as follows:

$$\boldsymbol{\nu = (1+c_b + c_T + c_s) * C_t}$$

where,
ν = kinematic viscosity of the oil at 40°C
c_b= Buoyancy correction (see 10.1 of the standard)
c_T= temperature correction(see 10.2 of the standard)
c_s=Surface tension correction (see 10.3 of the standard)
c_t =calibration constant of master viscometer with water at 20°C, mm^2/s^2

b) **For Cannon-ubbelohde master viscometer,** calculate the kinematic viscosity of the oil standard as follows:

$$\boldsymbol{\nu = (1+c_b) * C_t}$$

where, ν, c_b and C_t are as given above.

Calibration of Additional Master Viscometers and Viscosity Oil standards at 40°C.

c) Calibration constant for Cannon Master Viscometers:

$$C = \nu / [(1 + c_b + c_T + c_s) * t]$$

Where,
C= Calibration constant factor for water at 20°C, mm^2/s^2
ν= Kinematic viscosity of the oil standard mm^2/s
c_b= Buoyancy correction (see 10.1 of the standard)
c_T= Temperature correction(see 10.2 of the standard)
c_s= Surface tension correction (see 10.3 of the standard)
t= Average efflux time, s

d) Calibration constant for Cannon-Ubbelodhe Master Viscometers:

$$C = \nu / [(1 + c_b) * t]$$

where, C, ν, c_b and t are as given above.

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