NABL 122-04

NATIONAL ACCREDITATION BOARD FOR TESTING AND CALIBRATION LABORATORIES

SPECIFIC CRITERIA for CALIBRATION LABORATORIES IN MECHANICAL DISCIPLINE : Volume

ISSUE No. : 05
ISSUE DATE: 12.08.2014
AMENDMENT No. : 00
AMENDMENT DATE:

AMENDMENT SHEET

Sl no	Page No.	Clause No.	Date of Amendment	Amendment made	Reasons	Signature QO	Signature Director
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							

Sl. No.	Contents	Page No.

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 the specific requirements in calibration of Volumetric Apparatus under mechanical discipline. This part of the document thus amplifies the specific requirements for calibration of Volumetric Appratus and supplements the requirements of ISO/IEC 17025:2005.

1.2 Calibration and Measurement Capability (CMC)

1.2.1 CMC is one of 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 the specific requirements. 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 Volume calibration.

b) B.Sc (with Physics as one of the subject) or Diploma with 6 months experience in Basics of Volume Calibration.

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 field.

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

c) Experience and competence in Volume calibration.

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 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 Volume calibration.

b) B.Sc. (with Physics as one of the subject) or Diploma with 1 year experience in Volume calibration.

1.3.3 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 far 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 - Calibration of Volumetric Apparatus

2.1 Scope: Calibration of Volumetric Apparatus

2.1.1 Specific Requirements for Calibration of Volumetric Apparatus

Sl. No	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.

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.

2.2 National/ International Standards, References and Guidelines

- 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 D28 2004: Conventional value of the result of weighing in air.

- OIML R76-1 2006 Metrological and technical requirements –Non automatic weighing instruments.
- OIML R76-2 2007 Non automatic weighing instruments -Test Report format.
- EURAMET cg-18 V.03 Non automatic weighing instruments.
- **IS/**ISO 4787:2010, 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: 1992 (RA 2004), Reagent Grade water – Specification.
- ISO 3696:1987, Water for analytical laboratory use – Specification and test methods.
- ISO/TR 20461:2000: 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.

2.3 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^3).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 ± 15 hPa. Even though an average (fixed value) of pressure ± 30 hPa is used, whose influence 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.

2.4 Terms and Definitions

Readability

- The smallest scale division or digital interval of the weighing Instrument. For same mechanical weighing Instruments the scale marks may be sufficiently far apart for an estimation to be made of the actual weighing instrument reading when the pointer lies between two scale marks. The estimated readability may therefore be lower than the marked readability.

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.

Conventional Mass

- The conventional mass of a body is the mass of a standard weight of density 8000 kg/m^3 at 20^0C which balances this body in air of density 1.2 kg/m^3. ”

True Mass

- The true mass of a body relates to the amount of material it contains. The prefix true is added to the word mass where it is important to make it clear that a particular mass being considered is not a conventional mass value and it is important to avoid potential ambiguity. The International prototype kilogram, on which the International mass scale is realized is defined as a true mass of exactly 1 kilogram. Most high accuracy comparisons are performed on a true mass basis, and converted to conventional mass when quoted on a certificate.

Buoyancy Correction

- A buoyancy correction is the correction applied when weights of different densities are compared with each other during the calibration process, the buoyancy being a result of the upward force when the weight is immersed in a fluid (such as air) during the weighing process.

Gravimetric Method

- This method consists on weighing the volumetric apparatus under calibration when empty and again when full. The procedure adopted the use of the reference line or marks to have the purpose to provide an exact measure of liquid volume and the draining or drying procedures shall be followed carefully because they all effect the measurements. The difference obtained in the weighing measurements gives the mass of contained or delivered liquid.

Volumetric Method

- In the volumetric method a certain amount of liquid is delivered into a container. The volume is determined at a reference value denoted by a graduation mark. When the standard capacity measure is equipped with an adjustable device or scale the volume can be adjusted to the nominal volume. The calibration can be performed adjusting the level exactly to the reference value, by adding or removing water until the level corresponds to the graduation mark, or calculating the actual volume at the reference value on the basis of the scale reading.

Delivery Time and Waiting Time

- For volumetric instruments used for delivery of a liquid, the volume delivered is always less than the volume contained, due to the film of the liquid left on the inner walls of the volumetric instrument. The volume of this film depends on the time taken to deliver the liquid and the volume delivered decreases with decreasing delivery time .For example the delivered volume of the pipette or burette will decrease if the jet is broken (shorter delivery time) or will increase if the jet is not clean and the outflow of liquid is restricted.

Pipette

- Pipette is used to transfer known volume of liquid from one container to another.

Types of Pipette

- Volumetric Pipette - Used to measure a single volume accurately, typically to 4 significant figures.
- Graduated Pipette - Used to measure various volume with a single pipette, Volume delivered depends on the overall size of the pipette.
- Micro Pipette - Used to accurately measure small volume of liquid, typically vary from 1µl-1000 µl.

Volumetric Flask

- It is used to prepare a standard solution or dilution of a solution.

Burette

- It is used to accurately deliver the variable amount of liquid.

Volume at Reference Standard Temperature

- It is the temperature at which the volumetric apparatus is intended to contain or deliver its volume (capacity), either it shall be 27^0C for tropical countries like India (for others 20^0C).

Calibration Liquid

- Distilled or deionized water complying with ISO 3696, Grade 3 should be used for calibration.

Receiving Vessel

- Conical flask with ground joint, manufactured from glass, e.g. in accordance with ISO 4797. The nominal volume of the conical flask shall correspond to the volume of liquid to be measured.

2.5 Selection of Reference Apparatus and Reference Liquid for Volumetric Calibration

2.5.1 Balance

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

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

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

Selected volume under test[a] *V*	**Resolution** mg	**Standard deviation (repeatability)** mg	**Linearity** mg
100 µl $< V \leq$ 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
[a] - For practical purposes, the nominal volume may be used to choose the balance			

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

Selected volume[a] of apparatus under test *V*	**Resolution** **mg**	**Repeatability and linearity** **mg**	**Standard uncertainty of measurement** **mg**
1µl ≤ *V*≤ 10µl	0.001	0.002	0.002
10µl < *V*≤ 100µl	0.01	0.02	0.02
100µl < *V*≤ 1000µl	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
[a] For practical purposes, the nominal volume may be used to choose the balance.			

2.5.2 Other Apparatus Required

Name of the Device	**For Volumetric instruments as per IS/ISO4787:2010 Max. required measurement error**	**For Piston operated volumetric apparatus as per ISO 8655-6:2002 Max. required uncertainty**
Timing Device	≤ 1s	≤ 1s with k = 1
Thermometer for measuring temperature of the calibration liquid (water)	0.2°C upto 1000 ml 0.1°C ≥ 1000 ml	≤ 0.2°C with k = 1
Hydrometer	5 % RH	≤ 10% RH with k = 1
Barometer	1 kpa	≤ 0.5 kpa with k = 1

2.5.3 Use distilled or deionized water conforming grade3 as specified in ISO 3696 or IS 1070 should be used for calibration. Water shall be at room temperature.

2.5.4 **Receiving vessel** (required as per IS/ISO 4787:2010), conical flask with ground joint, manufactured from glass, e.g. in accordance with ISO 4797. The nominal volume of the conical flask shall correspond to the volume of the liquid to be measured.

2.5.5 **Liquid reservoir** with sufficient capacity for all the test liquid likely to be required for the complete series of test.

2.5.6 **Weighing Vessel**, (required as per ISO 8655-6), suitable for the test procedure selected from clause 7 (of ISO 8655-6). Care shall be taken regarding the loss of water by evaporation during the dispensing and weighing procedure. It is recommended that, especially for testing apparatus of the lowest volume, the height-to-diameter ratio of the weighing vessel be at least 3:1 or that a weighing vessel with a lid be used.

2.6 Calibration Interval

Recommended Calibration Interval for Reference Weighing Balance and Weights

Reference Weights	Recommended Interval	Reference Balance	Interval
Weights of E_1 class	3 years	Electronic weighing balance	1 Year
Weights of class E_2 to F_2	2 years		
Weights of class M_1	1 Year		

2.7 Legal Aspects

Calibration of volumetric measures 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 Calibration, Regulatory bodies, etc.

2.8 Environmental Conditions

The test shall be carried out in a draught-free room with stable environment. The test room shall have a relative humidity between 35% to 85% and shall provide a temperature locally constant ±1°C and temporally constant to ±0.5°C between 15°C and 30°C. Prior to this test, the volumetric instrument to be tested and the test water shall have stood in the room for sufficient time (1h to 2h) to reach equilibrium with the room conditions. Test water should be covered to avoid evaporation cooling. Temperatures (room and calibration liquid), atmospheric pressure and humidity should be recorded.

However, laboratory shall maintain relative humidity above 50% in order to minimize the evaporation effect if, the calibration is carried out as per ISO 8655-6.

2.9 Calibration Methods

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

2.9.1 Gravimetric Method

2.9.2 Volumetric Method

2.10 Calibration Procedure (Based on Gravimetric Method)

2.10.1 Based on IS/ISO 4787:2010 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.

2.10.2 Based on ISO 8655-6,2002 for piston operated volumetric apparatus:

This is applicable for ≤ 1µl 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.

2.10.3 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.

2.11 Equation for Calculation of Volume

2.11.1 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);

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

ρ_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;

ρ_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).

γ **-**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.

2.11.1.1 Coefficient of Cubical Thermal Expansion 'γ'

Material	Coefficient of cubical thermal expansion, γ^a (x10-6/ ^{0}C)
Borosilicate glass 3.3 Borosilicate glass 5.0 Soda-lime Glass	9.9 15 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.

2.11.1.2 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**[a]
Water temperature	± 0.5 °C	± 10^{-4}
Air pressure	± 8 mbar (0.8 kPa)	± 10^{-5}
Air temperature	± 2.5 °C	± 10^{-5}
Relative humidity	± 10 %	± 10^{-6}
Density of weights	± 0.6 g/ml	± 10^{-5}
[a] example : a relative volumetric error of 10^{-4} to the measured volume of 100 ml would be 0.01 ml		

2.11.1.3 When the temperature at which the volumetric instrument is used (t2) differs from the reference temperature (t1), the volume of the volumetric instrument at (t2) can be calculated from the following equation:

$$V_{t2}=V_{t1}[1+\gamma\,(t_2-t_1)]$$

where γ is the coefficient of cubical thermal expansion (see 4.11.2). For information on the effect of temperature differences.

2.11.2 Method –II

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

Alternatively, the following equation can be used for easy calculations:

$$V_{27}= (I_L - I_E) \times Z$$

Where, V is volume in µl, $(I_L - I_E)$ is weight in mg, Z is conversion factor (mg/µl)

Note: The conversion factor used to calculate the volume of the distilled water from its mass is called the Z factor (Z conversion factor) . The volumetric value can be obtained by multiplying the measured mass value with the Z factor. Tables B-6, B-7 and B-8 of IS/ISO 4787:2010 give the factor Z conversion values for different types of glass at common air pressure versus temperature in these tables, the combines effects of the density of the water, the thermal expansion of glass and the air buoyancy have been taken into account. The used balance weight is ρ_B =8.0 g/ml.

2.11.2.1 The Z conversion values is derived from the following equation

$$Z = (\rho_W - \rho_A)^{-1} \times (1- \rho_A / \rho_B) \times [1-\gamma(t-27)]$$

If, the general equation 8.11.1 is used for calculation of volume, the table B-3, B-4, B-5 list the necessary values for ρ_A, ρ_B and γ.

2.11.2.2 If the test temperature is different from the temperature of adjustment (which is 20°C, see ISO 8655-2, ISO 8655-3, ISO 8655-4, ISO 8655-5) and if the thermal expansion correction factor Y of the piston operate volumetric apparatus is known,

the equation $\boxed{\mathbf{V_i = m_i . Z}}$ may be replaced by equation $\boxed{\mathbf{V_i = m_i . Z.Y}}$

2.11.3 Method-III

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

2.11.3.1 Equation to calculate the mass of water (M_w) contained in the measure upto the mark is as given below:

$$M_W = (1 - \rho_a / \rho_s) \times [(M_1 - M_2) + (R_2 - R_1)] / (1 - \rho_a / \rho_w)$$

Where,

ρ_a - density of air during the measurement and calculated using BIPM formula or from the table using measured value of temperature, pressure and relative humidity of air

ρ_s - density of the material of the standard weight

ρ_w - density of water at temperature t_w = average temperature of the dispensed water [can be taken from water density table]

(M_1-M_2) - Weighed mass value of the water contained in the measure upto the mark

(R_2-R_1) - Difference in balance reading

2.11.3.2 Equation to calculate volume is as given below:

$$V_m = M_W / \rho_w = (1 - \rho_a / \rho_s) \times [(M_1 - M_2) + (R_2 - R_1)] / (\rho_w - \rho_a)$$

2.11.3.3 Equation to convert the volume to the reference temperature:

$$V_{27} = V_m \times [1 - \gamma(t_w - 27)]$$

where γ is the coefficient of cubical thermal expansion of the material of the measure.

2.11.3.4 Evaporation

Especially for small volumes below 50 µl, errors due to evaporation of the test liquid during weighing shall be taken in to consideration. Apart from the design of the weighing vessel, the test cycle time is important.

In order to keep the error due to evaporation as small as possible, the following additional items can be considered, if volumes below 50 µl are tested:

- a balance with appropriate accessories such as an evaporation trap could be used ;or

- the test liquid to be weighed could be delivered in to a capillary tube, although this method does not replicate the normal method of use and the user should verify for himself that correlation exists.

Regardless of these items, the error due to evaporation during the measuring series can be determined experimentally (see 7.2.8 of the standard ISO 8655-6) and compensated mathematically (see 8.1 of the standard ISO 8655-6). The uncertainty of this compensation should be added to the uncertainty of measurement.

2.12 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.

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.

2.13 Evaluation of CMC

2.13.1 Refer NABL 143 for CMC evaluation.

2.13.2 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).

2.13.3 All of the uncertainty components given above at 2.12. are applicable for CMC also as the uncertainty due to UUT is negligible in this case.

2.14 Sample Scope

An illustrative example of 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	Volume - Glassware, Burette, Pipette,	Weighing balance with resolution 1 mg , Standard weights and distilled water	10 - 1000 ml @ 27 °C	1 ml	0.8 ml	1 ml	"Gravimetric Method based on ISO 4787".	
	Micro-pipette	Weighing Balance with resolution 0.001 mg	1µl - 10 µl @ 27 °C	0.1 µl	0.09 µl	0.1 µl	ISO 8655 part 6	

* *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

2.15 Key Points

Following are the requirements not limited to:

2.15.1 The laboratory has to demonstrate the CMC values of 1/3rd of the accuracy of the specified by the manufacturer of volumetric apparatus & micro pipettes.

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

National Accreditation Board for Testing and Calibration Laboratories
NABL House
Plot No. 45, Sector- 44,
Gurgaon – 122002, Haryana
Tel.: +91-124 4679700
Fax: +91-124 4679799
Website: www.nabl-india.org