CRITERIA FOR LABORATORY ACCREDITATION IN VOLUME METROLOGY

Prepared by: SADCAS Advisory Committee CLAS	**Approved by**: SADCAS CEO	**Approval Date**: 2019-11-12 **Effective Date**: 2019-11-12

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1. PURPOSE AND SCOPE

The purpose of this document is to define the specific environmental, general and technical requirements to be met by accredited laboratories in the field of volume metrology.

This document is applicable to Southern African Development Community Accreditation System (SADCAS) accredited Laboratories. This document does not address the requirements for accreditation for compliance to national legal metrology legislation.

2. ABBREVIATIONS

2.1. **Ex** - Nominal capacity of the volume to be delivered.

2.2. **In** - Nominal capacity of the volume contained.

2.3. **CMC** - Calibration and Measurement Capability.

2.4. **OIML** - International Organization of Legal Metrology.

2.5. **% rh** - % Relative Humidity.

2.6. **SANS** - South African National Standards.

2.7. **UoM -** Uncertainty of Measurement.

2.8. **UUT** - Unit Under Test.

2.9. **ILAC** - International Laboratory Accreditation Cooperation.

2.10. **IAF** - International Accreditation Forum.

3. DEFINITIONS

3.1. Accreditation Criteria

Accreditation criteria represent those requirements including technical requirements with which accredited bodies are required to comply with at all times.

3.2. Accredited Body

Accredited Body means an organization or facility that has been accredited by SADCAS or by a member of the recognition arrangements of the International Laboratory Accreditation Co-operation (ILAC) or the International Accreditation Forum (IAF).

3.3. Diluters

Diluters are used to deliver mixtures of liquids of defined volumetric proportions. [4]

3.4. Dispensers

Dispensers are used for the repetitive delivery (dispensing) of a measured volume of liquid. [4]

3.5. Micro Pipette

A micropipette is a pipette designed to deliver volumes between 0,1 and 10 000 µℓ.

3.6. Nominal Value

Rounded or approximate value of a characterizing quantity of a measuring instrument or measuring system that provides guidance for its appropriate use. For example, 1 000 ml as the nominal quantity value marked on a single-mark volumetric flask. [9]

3.7. Nominal Volume (piston operated volume apparatus)

Volume specified by the manufacturer and used for identification and for indication of the measuring range. [4]

3.8. Nominal Volume (glass volume apparatus)

The volume that the measure is intended to contain or deliver, and that is defined by:

- the brim, where the brim is the datum line; or
- on single value glass measures, the graduation line that indicates the volume; or
- the graduation line that purports to be the nominal volume irrespective of the graduation lines marked above or below such nominal volume line. [4]

3.9. Piston Operated Pipettes

Piston operated pipettes are designed to pick up (or aspirate) and deliver liquids. Piston operated pipettes may be factory preset to deliver a given volume, or may have user selectable volumes within a useful volume range. Piston operated pipettes may be of the positive displacement or air displacement type [4]. This definition would include micropipettes of the piston operated type.

3.10. Pycnometer

A standard vessel used to measure and compare the densities of liquids.

3.11. Reference Temperature

The standard reference temperature, i.e. the temperature at which the article of volumetric laboratory ware is intended to contain, or deliver its nominal volume (nominal capacity) shall be 20 °C. [3]

3.12. Syringes

Syringes used in Laboratories fall into two broad categories, high precision metal and glass instruments, and general purpose, low precision, disposable plastic syringes. [11]

3.13. Unit of Volume

The unit of volume shall be the cubic centimetre (cm^3) or, in special cases the cubic decimetre (dm^3) or cubic millimetre (mm^3) for which the names millilitre (mℓ), litre (ℓ) or microlitre (µℓ) may be used. [3]

4. ENVIRONMENTAL REQUIREMENTS

4.1. When the calibration of pipettes and laboratory glassware is undertaken, the environmental temperature must be maintained between 15 °C and 30 °C provided that the temperature is stable, and is kept constant within ± 0,5 °C during the period of measurement.

4.2. When pipettes are calibrated, the relative humidity shall be maintained above 50 % RH in order to minimize the effects of evaporation. [4]. The relative humidity is not as critical when calibrating laboratory glassware due to the relatively smaller surface area of the liquid exposed to atmosphere when compared to the volume of the liquid.

4.3. Where necessary the laboratory shall maintain appropriate records to demonstrate and confirm both the temperature and temperature stability.

4.4. Lighting within the laboratory shall be adequate to facilitate the correct performance of the calibration work undertaken. Cognizance shall be taken of the minimum levels of lighting as may be specified in the local environmental regulations.

4.5. Drafts in the laboratory caused by doors, fans and/or air conditioners should be such that they do not have an adverse effect on the measurement results, where necessary local isolation in the form of draft shields should be utilized.

4.6. Measuring instruments used for the measurement and recording of the ambient temperature, relative humidity and/or barometric pressure in the laboratory shall be calibrated by a calibration laboratory that complies with SADCAS TR 09: Criteria for Performing Calibration and Intermediate Checks on Equipment used in Accredited Facilities.

5. GENERAL REQUIREMENTS

5.1. Laboratories shall have a policy and procedure/s that addresses how their own standards and equipment, and those belonging to customers are to be handled, cleaned and maintained. These policies and procedures shall also address biological and chemical decontamination.

5.2. Laboratories shall be equipped with appropriate calibrated measurement equipment necessary to perform calibration by gravimetric means, and shall include:

- An analytical, micro balance or equivalent weighing device as appropriate;
- Thermometer/s;
- Hygrometer; and
- Barometer.

Table 1 below specifies the minimum resolution for the weighing device, appropriate to the selected volume of the apparatus under test [5]

Table 1 - Minimum Resolution For The Weighing Device, Appropriate To The Selected Volume Of The Apparatus Under Test

Selected volume of the apparatus under test	Minimum resolution of weighing device (mg)
1 µl ≤ V ≤ 10 µl	0.001
10 µl < V ≤ 100 µl	0.01
100 µl < V ≤ 1000 µl	0.1
1 ml < V ≤ 10 ml	0.1
10 ml < V ≤ 200 ml	1

5.3. **R**aw data shall not be recorded in pencil, or erasable ink.

5.4. The scope of accreditation shall list the types of volume measuring and dispensing equipment that the laboratory is competent to calibrate under the parameter 'volume metrology'. This list may include the following:

- Syringes;
- Automated (Glass) Pipettes;
- Piston Pipettes;
- Burettes;
- Flasks;
- Measuring Cylinders;
- Pycnometers;
- Diluters;
- Dispensers;
- Simple measures other than glass;

- Metal Strike measures; and
- Other Metal measures.

The internal methods and the reference methods shall be indicated on the schedule of accreditation.

6. TECHNICAL REQUIREMENTS

6.1. Calibration of Piston Pipettes

6.1.1. Either distilled or deionized water shall be used to calibrate pipettes, and both the water and equipment to be calibrated shall have been allowed to stabilize for a period of not less than 2 hours prior to performing the calibration. [5]

6.1.2. When dispensing volumes of especially less than 50 µℓ, errors due to evaporation of the test liquid during weighing shall be taken into consideration. Where the evaporation has been determined experimentally and corrected for, the uncertainty associated with the correction shall be included in the uncertainty budget. [5]

6.1.3. Where dispensing volumes are greater than 50 µℓ, the uncertainty associated with the evaporation may still be an influencing factor in the associated uncertainty of measurement. Where it is considered that the uncertainty associated with the evaporation is insignificant, the laboratory shall have documented evidence to support this claim.

6.1.4. When calibrating pipettes that deliver a variable selected volume, the pipettes shall be calibrated at a minimum of 3 different volumes, namely:

- The nominal volume;
- 50% of the nominal volume; and
- 10% of the nominal volume or the lower limit as specified by the manufacturer.

Each channel of a multichannel pipette shall be treated as a separate pipette.

6.1.5. When calibrating pipettes using the gravimetric method, a minimum of 10 measurements at each test volume shall be taken [5]

6.1.6. Prior to the commencement, and after completion delivery, the barometric pressure, relative humidity, air temperature and the temperature of the test liquid shall be recorded to the nearest 1 kPa, 10 % and 0.2 °C. [5]

6.1.7. The laboratory shall have a comprehensive procedure describing the calibration process, and the procedure shall address as a minimum:

- Handling procedure for the pipette;
- The addition of water to the weighing vessel;

- Recording of the measurement conditions;
- Pre-wetting of pipette tip;
- The aspiration of the test volume;
- The delivery of the test volume;
- Blow out;
- The weighing procedure;
- Conversion of the mass to volume;
- Evaporation;
- Measurements per volume;
- The uncertainty estimation; and
- Validation.

6.1.8. The calibration certificate shall include:

- The identification of the pipette including the supplier (or manufacturer's) name, the type or model number, the serial number (unique or other inventory number), nominal volume or volume range;
- The reference temperature;
- Identification of the pipette tip (or description);
- The environmental conditions (barometric pressure, relative humidity, and temperature); and
- Reference to the procedure used.

6.1.9. Where devices are made from composite materials (e.g. piston pipettes) it is difficult to state a specific value for the thermal coefficient of expansion, and therefore the calibration should be conducted at a temperature as close to the temperature at which the pipette is to be used, and this temperature shall be specified on the calibration certificate. An approximate value of the cubic coefficient of expansion should then be used when estimating the uncertainty of measurement. See Appendix C.

6.1.10. The following should be considered as a minimum when calculating the uncertainty of measurement:

- The mass (balance or mass pieces dependent upon method);
- The evaporation and/or correction for the evaporation;
- The water temperature;
- The air density determined from the air temperature, pressure and relative humidity;
- Instrument characteristics such as coefficient of thermal expansion;
- The measurement repeatability; and
- The conversion from mass to volume, including when Z correction factors are used.

Any deviation from the above shall be technically justified, and supported with documented evidence. [8]

6.2. Calibration of Laboratory Glassware

6.2.1. In addition to the equipment identified in 5.2, the laboratory shall have a stopwatch necessary for the evaluation of the delivery time.

6.2.2. The standard reference temperature, i.e. the temperature at which the volumetric glassware is intended to contain or deliver its nominal volume is 20 °C [3]. Should the laboratory calibrate the glassware at a reference temperature other that 20 °C, this shall be clearly stated on the Calibration certificate or report.

6.2.3. Prior to calibration, the glassware shall be visually inspected to determine if there are any visible defects, graduation lines shall be clean, permanent and of uniform thickness, and shall lie in planes at right angles to the longitudinal axis of the glassware. There shall be no irregularity in the spacing of graduation lines. [6] If any defect in the glass or graduation lines is observed this shall be reported on the calibration certificate or report.

6.2.4. The measurements shall be performed with distilled or deionized water suitable for general laboratory use. [3]

6.2.5. The temperature of the liquid shall be measured and corrections in temperature from the reference temperature shall be applied.

6.2.6. Laboratory glassware shall be suitably cleaned prior to calibration.

6.2.7. The laboratories procedure shall address the following:

- Cleaning of the glassware;
- Weighing of the glassware;
- Filling of the glassware;
- Details of how the meniscus is to be set and read;
- Delivery time;
- Corrections to be applied for buoyancy, temperature, thermal expansion, and water density;
- The conversion from mass to volume;
- The uncertainty estimation; and
- Validation.

6.2.8. The calibration certificate shall include:

- The identification and description of the glassware, the supplier (or manufacturers) name, the nominal volume or capacity, the serial number, (or other unique identification);
- The reference temperature;
- An indication if the glassware is intended to contain (In) or deliver (Ex) the indicated volume;
- The environmental conditions (Barometric Pressure, Relative Humidity, and Temperature); and
- The cubic coefficient of expansion used. '(Appendix C)

6.3. Calibration of Automated Pipettes

6.3.1. In addition to the equipment identified in 5.2 the laboratory shall have a stand capable of supporting the pipette in a vertical position.

6.3.2. Automatic pipettes are normally used for the calibration of volumetric instruments – including volumetric standards of a lower order. [7]

6.3.3. The automatic pipette should be inscribed with:

- The nominal capacity;
- Ex 20ºC;
- An identification number;
- The name of the manufacturer or supplier; and
- The delivery time.

6.3.4. The calibration procedure shall address:

- Thermal stability of liquid;
- Cleaning of the automatic pipette;
- Pre-wetting of internal surfaces;
- The delivery time;
- Weighing of the volumetric sample;
- Collection of drips;
- Number of measurements to be performed;
- Corrections to be applied, including water density, buoyancy, and thermal expansion;
- The uncertainty estimation; and
- Validation of the method.

6.4. Sources of uncertainty in volume determination using gravimetric method are the following:

- Mass (balance or mass pieces);
- Evaporation or correction for the evaporation;
- Water temperature;
- Air density;
- Measurement repeatability;
- Meniscus reading;
- Cubical thermal expansion coefficient;
- Water density; and
- Mass piece density.

7. REFERENCES

1. EURAMET cg-19, Version 3.0 (09/2018) - Guidelines on the determination of uncertainty in gravimetric volume calibration.

2. ISO/IEC 17025:2017 - General requirements for the competence of testing and calibration laboratories.
3. ISO 4787:2010 Laboratory glassware – Volumetric instruments – Methods for testing capacity and for use.
4. ISO 8655-1:2002 - Piston operated volumetric apparatus – Part 1: Terminology, general requirements and user recommendations.
5. ISO 8655-6: 2002 - Piston operated volumetric devices – Part 6: Gravimetric methods for the determination of measurement error.
6. ISO 4788-2005 - Laboratory Glassware – Graduated Measuring Cylinders.
7. OIML D26-1999 - Glass Delivery Measures – Automatic Pipettes.
8. ISO/TR 20461 - Technical Report – Determination of uncertainty for volume measurements made using the gravimetric method.
9. ISO/IEC Guide 99 (JCGM 200:2012) - International vocabulary of metrology — basic and general concepts and associated terms (VIM), issued by BIPM, IEC, IFCC, ILAC, ISO, IUPAC, IUPAP and OIML.
10. SANS 1698:2007 - Verification standards for the verification of volume-measuring instruments, including commercial standards of volume. Edition 1.2 (ISBN 978-0-626-19459-8)
11. UKAS LAB 15 Traceability - Volumetric Apparatus Ed 2 March 2009

APPENDIX A - AMENDMENT RECORD

Revision Status	Change			Approved by	Effective Date
	Page	Clause/ Sub-clause	Description of Change		
Issue 1	-	-	-	SADCAS CEO	2019-11-12

APPENDIX B: SAMPLE OF SCHEDULE OF ACCREDITATION

S C H E D U L E OF A C C R E D I T A T I O N

VOLUME METROLOGY

Laboratory Accreditation Number: CAL-9 …… (ISO/IEC 17025:2017)

Permanent Address of Laboratory …. …. …. …. **Postal Address** …. …. …. **Tel** : …. **Cell** : …. **Fax** : …. **Email** : ….	**Technical Signatories** : …. …. …. …. …. **Nominated Representative** : …. **Issue No** : …. **Date of Issue** : …. **Expiry Date** : ….

ITEM	MEASURED QUANTITY OR TYPE OF GAUGE OR INSTRUMENT	METHOD	RANGE OF MEASURED QUANTITY	CALIBRATION AND MEASUREMENT CAPABILITY EXPRESSED AS AN UNCERTAINTY (±)
1	Micropipettes / Syringes	Internal: e.g. *P-NMI-xyz* Reference: e.g. *EURAMET cg-19*	1 µℓ to 10 µℓ	0,3 µℓ
			10 µℓ to 100 µℓ	0,9 µℓ
			100 µℓ to 200 µℓ	0,8 µℓ
			200 µℓ to 500 µℓ	2,8 µℓ
2	Glassware	Internal: …. Reference: …..	10 mℓ to 5 ℓ	0,05 %

Original date of accreditation: ….

The CMC, expressed as an expanded uncertainty of measurement, is stated as the standard uncertainty of measurement multiplied by a coverage factor k = 2, corresponding to a confidence level of approximately 95%.

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SADCAS Technical Manager

APPENDIX C: CUBIC COEFFICIENTS OF EXPANSION

Cubic coefficients of expansion of various materials used in the manufacture of volume measuring and dispensing devices

Material	**Cubic coefficient of thermal expansion**	**Reference Source**
Plastic material	0,000 300 $°C^{-1}$ *	NPL Guide 69
Fused Silica (Quartz)	0,000 001 6 $°C^{-1}$	ISO 4787-1984
Borosilicate Glass (Pyrex)	0,000 010 $°C^{-1}$	ISO 4787-1984
Soda-Lime Glass	0,000 025 $°C^{-1}$	ISO 4789-1984
Mild Steel	0,000 033 $°C^{-1}$	SANS 1698-2005
Stainless Steel	0,000 051 $°C^{-1}$	SANS 1698-2005
Copper, brass	0,000 053 $°C^{-1}$	SANS 1698-2005
Aluminum	0,000 069 $°C^{-1}$	SANS 1698-2005

* The cubic coefficient of expansion of 'plastic' materials can vary greatly due to their actual composition.