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MANAGEMENT OF VOLUMETRIC GLASSWARE

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Management of Volumetric Glassware

SCOPE

The present document describes the basic principles for the management of volumetric glassware within an OMCL, with regard to calibration and verification.

INTRODUCTION

The glassware used in a laboratory should be capable of achieving the required accuracy for the tests performed, in accordance with ISO/IEC 17025:2005. This glassware includes volumetric flasks, burettes, pipettes or measuring cylinders.

Before being introduced for use, laboratory glassware should be calibrated or checked to demonstrate that it meets the laboratory's requirements and complies with the relevant standard specification. A visual integrity check prior to use and comparison of the certificate with the laboratory requirements are to be carried out at this stage. Intermediate checks, carried out on a random sample of glassware according to a defined procedure, may be needed to maintain confidence in the calibration status of the glassware over the time of use.

Usually, class A and AS volumetric glassware meets the basic accuracy requirements needed in most quantitative analytical work (for tolerances, please refer to references 1, 2 and 3, for instance). It is considered that it is generally not necessary to use individually calibrated glassware with an individual certificate of calibration, since certified glassware usually complies with a certain class and has a certificate of batch calibration. If the quality or uncertainty of a test result obtained while using certified glassware is not suitable or consistent, the laboratory should assess whether the glassware used meets the required tolerance class and/or assess its contribution to the uncertainty of the test. Class A and AS volumetric glassware is supplied with a batch calibration certificate, where the tolerance and the error of the material comply with an international standard. Laboratories can also acquire class A volumetric glassware with individual calibration, as supplied by some brands. The choice of the type of calibration of volumetric glassware (batch or individual) depends on the laboratory's own assessment.

It should be noted that the minimum requirements established by different national accreditation bodies (NAB) for the volumetric glassware used in a laboratory might differ with regard to the validity of the calibration and the frequency of the verification of the suitability of the volumetric glassware after the calibration validity has expired. The initial period of calibration of non-automatic volumetric glassware can range from 2 to 10 years, according to documentation available from ILAC, EA, OIML and several NABs.

GENERAL RULES

The tolerance of the glassware being used should be less than the maximum admissible error for testing. Also, the laboratory should adopt a policy of managing appropriately volumetric glassware, allowing it to identify the needs for calibration and/or verification and, if deemed necessary, to trace back to the targeted glassware.

The laboratory shall ensure that:

- The calibration needs are identified correctly (e.g. need for class A);

- The apparatus that is identified as in need of calibration must be calibrated initially by the vendor or by the OMCL according to a defined and appropriate level prior to use;

- After purchase and prior to the first use, as well as periodically during use, a careful visual inspection of the glassware is made to check for signs of deterioration or attack, such as scratches, blurred, broken or chipped glass, scale or calibration marks not visible. Damaged apparatus must be rejected and discarded. In the event of aggressive use of the equipment, such as the use of corrosive substances or thermal or mechanical shock, visual inspections should be performed more frequently.

Procedures should be defined for the cleaning of glassware, either by washing machine or by hand. Cleaning validation is advisable given the possibility of poor/deficient washing or cross contamination with cleaning agents, such as detergents.

Cleaning procedures, storage, and segregation of volumetric equipment may be critical, particularly for trace analyses where leaching and adsorption can be significant. The laboratory shall ensure that products or processes employed in washing and decontamination do not alter the state of glassware. The laboratory may decide to perform periodic validation of the cleaning of glassware, either by determining relevant chemical parameters in the wash water (such as conductivity and total organic content, TOC), testing cleaning on different surfaces with the worst case product or by a specific detailed procedure (see example in Annex 2), if it is found that checking of blanks is not enough for the needs of the laboratory.

Washing and drying glassware for volumetric purposes should be a temperature that takes into account the chemical nature of the glass. Above 150°C there may be significant changes in the volumetric capacity of borosilicate glass and above 90°C for the soda-lime glass. According to ISO 4787, it is recommended that volumetric instruments should not be heated significantly above 180 °C.

CALIBRATION

Performance of assays and limit tests require the use of calibrated volumetric glassware with acceptance criteria according to at least class A and/or AS. Special care should be taken when volumetric glassware is used for a direct reading of the result, without any comparison with a standard (such as a volumetric burette in an assay by titration).

Prior to use, the compliance of the volumetric glassware to the required class is demonstrated via calibration as follows:

- external calibration, by an accredited entity (batch certificate or individual calibration certificate by supplier/producer, when needed);

- internal calibration, in cases where no batch certificate is available or superior quality requirements have to be fulfilled. In those cases calibration must be performed according to a clearly defined procedure.

The frequency of calibration of volumetric instruments depends on their characteristics, the type and frequency of their use and previous calibrations history. The laboratory should decide the suitable frequency for calibration of all its volumetric glassware, taking into account the use.

If no previous reference exists, initial intervals of annual calibration could be adopted, and adjusted afterwards, depending on the use and history of the glassware. An intermediate verification may be performed, if needed.

If the initial certificate expires or the volumetric glassware has been exposed to environmental or analytical conditions that could jeopardise calibration, a verification of the suitability of the equipment may be performed, if found to be necessary. The laboratory can also decide to perform internal or external calibration, depending on the use.

For volumetric glassware which produces a direct result, such as burettes used in volumetric titrations, re-calibration of the equipment should be carried out within the schedule established by the laboratory or, alternatively, new calibrated volumetric glassware should be introduced, taking into account the laboratory requirements and needs.

VERIFICATION

Procedures for verification of volumetric glassware are to be implemented by the laboratory. In all cases a careful visual inspection of the glassware is made annually to check for scratches, blurred glass, broken glass, chipped glass and poorly visible scale or calibration marks. Initially, a yearly analytical assessment of glassware could be performed on a representative sample and number of items of glassware in use. Based on the results of the verification, the frequency can be prolonged: in cases where all test results are in compliance with the acceptance criteria in consecutive years, a re-verification may be performed after five years.

Procedures for verification of volumetric glassware are described in several documents, issued either by ISO, EA, OIML or NIST. In general, all of them describe a gravimetric verification procedure for the volumetric capacity. The amount of water that the vessel contains, or delivers at a measured temperature, is accurately weighed, and the volume calculated in cubic centimetres at standard temperature and pressure. All of these documents state that special care must be taken to ensure that the glassware is clean and, in particular, grease free.

REFERENCES

1. ISO 385:2005 “Laboratory glassware – Burettes”
2. ISO 648:2008 “Laboratory glassware -- Single-volume pipettes”
3. ISO 1042:1998 “Laboratory glassware -- One-mark volumetric flasks”
4. ISO 4787:2010 “Laboratory glassware -- Volumetric instruments -- Methods for testing of capacity and for use”
5. OIML R 43 Edition 1981 (E) “Standard graduated glass flasks for verification officers”
6. OIML D 26 Edition 2010 (E) “Glass delivery measures - Automatic pipettes”
7. ILAC P10:01/2013 “ILAC Policy on Traceability of Measurement Results”
8. UKAS LAB15 EDITION 2 (June 2009) “Traceability: Volumetric Apparatus”
9. ISO 17025:2005 “General requirements for the competence of testing and calibration laboratories”

ANNEXES

In this section, examples are given in order to help laboratories with the implementation of some of the tasks which have been described in the core document, namely:

- how to perform the verification of glassware in Annex 1, in accordance to an OIML standard;
- how to perform the validation of the cleaning of glassware in Annex 2.

ANNEX 1

OIML (reference 5) describes the following procedure for the verification of glassware:

"OIML R 43 Edition 1981 (E) "Standard graduated glass flasks for verification officers
(…)
APPENDIX A: VERIFICATION OF STANDARD GRADUATED GLASS FLASKS"

A.1. Test liquid.
A. 1.1. Water: distilled or deionized of high purity, in accordance with the following requirements when tested immediately before use: it must be free from dissolved gases, and heavy metals, in particular copper, as shown by the dithizone test, the specific conductivity must not exceed 1 × 104 S × m-1 at 20 °C, and it must be neutral to methyl red.

A.2. Instruments.
A.2.1. Scales: of suitable maximum capacity, having an accuracy at least equal to that of nonautomatic weighing machines of the high accuracy class (or possibly special accuracy class).
3.2.2. Thermometer: with appropriate measurement range, suitable for the measurement of temperature with error not exceeding ± 0.1 °C.

A.3. Method.
A.3.1. Determination of the « contained » capacity (capacity « In »).
A.3.1.1.
— clean and dry the flask,
— weigh the flask empty,
— allow the temperature of flask and water used for testing to equalize,
— record the water temperature,
— place the flask on a flat horizontal surface, and fill it with water to a point a few millimetres below the mark indicating the nominal capacity « In »,
— add more water slowly, to bring the meniscus carefully to the scale mark concerned,
— check that the outside surface of the flask, and its internal surface above water level are dry, and that neither bubbles or foam are present in the water,
— weigh the flask and its contents.
A.3.1.2.
From the difference between the results of weighing the flask when full and the flask when empty, and allowing for correction of the displaced air, the mass of the quantity of water corresponding to nominal « contained » capacity is obtained.
Knowing the temperature of the water in the flask, and using tables for the density of water as a function of the temperature, the volume of the water contained in the flask may be determined.
From this volume and the coefficient of cubic expansion of the glass, the conventional true capacity of the flask is determined for the scale mark corresponding to nominal « contained » capacity, at the chosen reference temperature (20 °C or 27 °C).
A.3.1.3.
Repeat the procedure for filling and weighing the filled flask as in point A.3.1.1., and calculate as in point A.3.1.2., for four other scale marks, including both the highest and lowest scale marks.
A.3.1.4.
The error on the « In » capacity for any scale mark, is equal to the difference between the « In » capacity indicated by a mark, and the conventional true capacity corresponding to this mark, determined by the method described in points A.3.1.1. to A.3.1.3.
The error on the « In » capacity between any two scale marks, is equal to the difference between:
— the difference between the capacities indicated by the marks and,

— the difference between the conventional true capacities determined by the method given in points A.3.1.1. to A.3.1.3.

A.3.2. Determination of the « delivered » capacity (capacity « Ex »)

A.3.2.1.

— clean the flask,
— equalize the temperature of the flask and the water used for testing,
— record the water temperature,
— fill the flask with water to a few millimetres below the scale mark indicating the nominal capacity « Ex »,
— empty the flask, and allow to drain for 2 to 3 minutes,
— refill the flask as above, adding the water slowly so as to bring the meniscus carefully to the scale mark concerned,
— check that the outside surface of the flask, and its internal surface above water level are dry, and that neither bubbles or foam are present in the water,
— weigh the flask and its contents,
— empty the contents into a container,
— hold the flask in an inverted position to drain for 30 seconds,
— remove the last drop adhering to the lip of the neck, by bringing the lip into contact with the internal wall of the container,
— weigh the empty flask.

A.3.2.2. From the difference between the results of weighing the flask when full and when empty, and allowing for correction of the displaced air, the mass of the quantity of water corresponding to the «delivered » capacity is obtained.

Knowing the temperature of the water in the flask, and using tables for the density of water as a function of the temperature, the volume of the water delivered can be determined.

From this volume and the coefficient of cubic expansion of the glass, the conventional true capacity of the flask for the scale mark corresponding to « Ex » nominal capacity, at the chosen reference temperature (20 °C or 27 °C) may be determined.

A.3.2.3. Repeat the procedure for filling, draining and weighing as in point A.3.2.1., and calculate as in point A.3.2.2., for four other scale marks, including both the highest and lowest scale marks.

A.3.2.4. The error on the « Ex » capacity for any scale mark, is the difference between the « Ex » capacity indicated by a mark, and the conventional true capacity corresponding to this mark, determined by the method given in points A.3.2.1. to A.3.2.3.

The error on the « Ex » capacity between any two scale marks, is equal to the difference between:

— the difference between the capacities indicated by these marks, and
— the difference between the conventional true capacities determined by the method given in points A.3.2.1. to A.3.2.3.”[5]

ANNEX 2

Example of validation of a cleaning procedure for volumetric glassware using a glassware washing machine (source: CY – Workshop for auditors October 2015)

1. The procedure can be used during the IQ-OQ of the new glassware washing machine and for PQ (e.g. every 6 months);
2. The laboratory chooses an available "difficult to clean" API;
3. From the volumetric flasks described in the testing method of the selected API, two of the lower and two of the maximum nominal volume flasks are selected (e.g. 2 x 5ml and 2 x 100ml volumetric flasks);
4. A reference solution of the API is prepared as described in the testing method;
5. Each of the selected volumetric flasks is filled to about 10% of its nominal volume with this reference solution, closed and shaken so the inner walls of the flasks will be covered by the liquid. They are emptied and allowed to dry;
6. The 4 flasks are then washed alone in the washing machine using the defined washing instructions (Empty Load);
7. The same procedure is performed with the maximum number of volumetric flasks that can be washed in the washing machine (Full Load);
8. All the flasks are filled with the solvent solution used for the preparation of the Reference Standard Solution and analysed with the HPLC testing method of the API used (the method is validated and the validation parameters, e.g. LOD, LOQ etc. are used);
9. No traces of the API above the LOD of the method should be detected.