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Laboratory glass and plastic ware — Volumetric instruments — Methods for testing of capacity and for use

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 48, *Laboratory equipment*, WG7-Volumetric apparatus made of glass and plastic. ISO/DIS 4787

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This third edition cancels and replaces the second edition (450 4787:2010), which has been technically revised.

The main changes compared to the previous edition are as follows:

- a) plastic ware was included;
- b) new information on meniscus adjustment of convex meniscus;
- c) improved figures for meniscus adjustment;
- d) Table 1 was improved;
- e) new <u>Table 2</u> for minimum requirements for the measurement devices;
- f) new test room ambient conditions;
- g) new information regarding repeatability and uncertainty added in Annex E;
- h) Formula (C.1) was changed to Formula (1).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Laboratory glass and plastic ware — Volumetric instruments — Methods for testing of capacity and for use

1 Scope

This International Standard provides methods for the testing, calibration and use of volumetric instruments made from glass and plastic in order to obtain the best accuracy in use.

NOTE Testing is the process by which the conformity of the individual volumetric instrument with the appropriate standard is determined, culminating in the determination of its error of measurement at one or more points.

The International Standards for the individual volumetric instruments include clauses on the definition of capacity; these clauses describe the method of manipulation in sufficient detail to define the capacity without ambiguity. This International Standard contains supplementary information.

The procedures are applicable to volumetric instruments with nominal capacities in the range of $100\,\mu$ l to $10\,000$ ml. These include single-volume pipettes (see ISO 648), graduated measuring pipettes and dilution pipettes (see ISO 835), burettes (see ISO 385), volumetric flasks (see ISO 1042), and graduated measuring cylinders (see ISO 4788 and ISO 6706).

The procedures are not recommended for testing of volumetric instruments with capacities below 100 µl such as micro-glassware. (standards.iteh.ai)

This International Standard does not deal specifically with pycnometers as specified in ISO 3507. However, the procedures specified below for the determination of volume of glassware can, for the most part, also be followed for the calibration of pycnometers. 78c3-48e8-90a5-

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2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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 1773, Laboratory glassware — Narrow-necked boiling flasks

ISO 3696, Water for analytical laboratory use — Specification and test methods

ISO 4788, Laboratory glassware — Graduated measuring cylinders

ISO/IEC Guide 99:2007, International vocabulary of metrology — Basic and general concepts and associated terms (VIM)

ISO 6706, Plastics laboratory ware — Graduated measuring cylinders

ISO/IEC Guide 98:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/IEC Guide 99 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

4 Summary of method

The general procedure for testing of capacity and for use is based upon a determination of volume of water, either contained in or delivered by the volumetric instrument. This volume of water is based upon knowledge of its mass under consideration of buoyancy and its density (gravimetric method).

5 Volume and reference temperature

5.1 Unit of volume

The unit of volume shall be the millilitre (ml), which is equivalent to one cubic centimetre (cm³).

5.2 Reference temperature

The standard reference temperature, i.e. the temperature at which the volumetric instrument is intended to contain or deliver its volume (capacity) shall be 20 °C. 21)

When the volumetric instrument is required for use in a country which has adopted a standard reference temperature of 27 °C (the alternative recommended in ISO 384 for tropical use), this figure shall be substituted for 20 °C. 8c65b1303885/iso-dis-4787

6 Apparatus and calibration liquid

6.1 Analytical balance or equivalent weighing device.

The balance used for testing shall be chosen according the specified minimum requirements of <u>Table 1</u>, depending on the nominal volume of the volumetric instrument under test.

Table 1 — Minimum requirements for the balance

Nominal capacity	Readability	Repeatability	Expanded uncertainty in use $U(k=2)^a$
V	mg	mg	mg
100 μl ≤ <i>V</i> ≤ 10 ml	0,1	0,2	0,4
10 ml < <i>V</i> ≤ 1 000 ml	1	2	4
V > 1 000 ml	10	10	40

^a Expanded uncertainty in use obtained according to EURAMET CG-18 [2] (which includes applicable definitions) at the value of the nominal volume. If uncertainty in use is not available, then the uncertainty at calibration should be taken.

6.2 Measurement devices

The minimum requirements for each relevant measurement device are described in Table 2.

Not applicable

Parameter	Readability	Expanded uncertainty of meas- urement	
		(k=2)	
Thermometer for liquids	0,1 °C	0,2 °C	
Thermometer for room air	0,1 °C	0,2 °C	
Hygrometer	1 % rel. humidity	5 % rel. humidity	
Barometer	0.1 kPa	1 kPa	

Table 2 — Minimum requirements for the measurement devices

6.3 Calibration liquid, distilled or deionized water complying with ISO 3696, Grade 3 should be used for testing. The water temperature shall be within ±0,5 °C of ambient air temperature.

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6.4 Receiving vessel, conical flask, if possible, with ground joint, manufactured from glass, e.g. in accordance with ISO 1773, ISO 4797, or ISO 24450. The receiving vessel shall have a capacity adequate to the amount of water delivered by the volumetric instrument.

7 Factors affecting the accuracy of volumetric instruments

7.1 General

Timing device

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The same sources of error are, naturally, inherent both in calibration and use. In the former, every attempt is made to reduce the errors to a infiniting in the latter, the care needed is dependent upon the degree of accuracy required. When the greatest possible accuracy is desired, the volumetric instrument should be used as closely as possible to the way it has been calibrated.

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7.2 Temperature

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7.2.1 Temperature of the volumetric instrument

- **7.2.1.1** The capacity of the volumetric instruments varies with change of temperature. The particular temperature at which a volumetric instrument is intended to contain or deliver its nominal capacity is the "reference temperature" of the instrument (see <u>5.2</u>).
- **7.2.1.2** A volumetric instrument which was calibrated at 20 °C, but used at a reference temperature of 27 °C, would show an extra error of only 0,007 % if it is made of borosilicate glass having a coefficient of cubical thermal expansion of 9.9×10^{-6} °C⁻¹ and of 0,02 % if it is made of soda-lime glass having a coefficient of cubical thermal expansion of 27×10^{-6} °C⁻¹, respectively. These errors are smaller than the limits of error for most volumetric instruments. When performing calibrations, it is important to refer to the reference temperature.

7.2.2 Temperature of calibration liquid

The temperature of the water used for the calibration shall be measured to ± 0.1 °C, with a maximum variation of ± 1 °C during the test. Corrections for differences in temperature, prevailing during testing or use, from the reference temperature shall be applied in accordance with <u>Formula (1)</u>, <u>9.5</u> and <u>Annex C</u>. The liquid temperature should be measured in the vessel where the instruments are filled from or directly inside the instruments if technically possible.

7.3 Cleanliness of surface

The volume contained in, or delivered by, of a volumetric instrument depends on the cleanliness of the internal surface. Lack of cleanliness of glass surface results in errors through a poorly shaped meniscus involving two defects:

- incomplete wetting of the glass surface, i.e. the liquid surface meets the glass at an arbitrary angle instead of forming a curve such that it meets the glass tangentially;
- a generally increased radius of curvature, due to contamination of the liquid surface reducing the surface tension.

Volumetric instruments made of polyolefins, such as Polypropylene (PP) and Polymethylpentene (PMP), or fluoroplastics, such as Perfluoroalkoxy-Copolymer (PFA), have water-repellent surfaces which results in a poorly shaped convex or even flat meniscus (see <u>8.2</u> and 8.3).

The ascending or descending liquid meniscus shall not change shape (i.e. it shall not crinkle at its edges). To ascertain whether a piece of apparatus is satisfactorily clean, it shall be observed during filling and dispensing. Additionally, an experienced operator can recognize the shape of an uncontaminated meniscus, in relation to its diameter.

Lack of cleanliness causes additional errors with volumetric instruments used for delivery due to the film of liquid on the walls being irregularly distributed or incomplete, e.g. forming drops on the glass surface. Furthermore, chemical residues can introduce an error in the analytical result by contamination. Therefore, where volumetric instruments are fitted with ground stoppers, special attention shall be paid to cleaning the ground zone. ARD PREVIEW

NOTE Small residues of acid, for example, could impair the concentration of the alkaline solution with which the volumetric instrument is filled.

Satisfactory methods of cleaning are described in Annex A and Annex B.

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7.4 Conditions of used volumetric in struments/iso-dis-4787

The surface shall be free from obvious damage, the graduations and inscriptions shall be clearly readable and especially with instruments adjusted to deliver the jet shall be free from damage and allow an unrestricted outflow of liquid.

7.5 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 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 a 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.

In view of the above, delivery times and waiting times have been specified in the International Standards on volumetric instruments; these times shall be observed.

8 Setting the meniscus

8.1 General

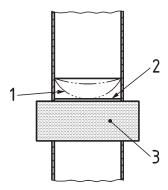
Most volumetric instruments employ the principle of setting or reading a meniscus (the interface between air and the liquid) against a graduation line or ring mark. Wherever practicable, the meniscus should descend to the position of setting.

The tubing of the volumetric instrument shall be in a vertical position. The eye of the operator shall be in the same horizontal plane as the meniscus or the graduation line (graduation mark).

8.2 Setting the meniscus

8.2.1 Meniscus of transparent liquids

In case of a concave meniscus, the meniscus shall be set so that the plane of the upper edge of the graduation line is horizontally tangential to the lowest point of the meniscus, the line of sight being in the same plane (see Figure 1).



Key

- 1 Meniscus
- 2 Graduation line
- 3 dark coloured paper

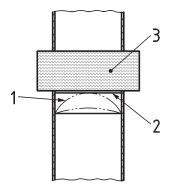
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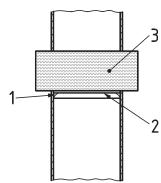
(standards.iteh.ai)

Figure 1 — Setting of concave meniscus

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In case of a convex or even flat meniscus, known for water-repellent, non-wetting surfaces of polyolefins, such as PP and PMP, or fluoroplastics, such as PFA, the meniscus shall be set so that the plane of the upper edge of the graduation line is horizontally tangential to the highest point of the meniscus, the line of sight being in the same plane (see Figure 2).





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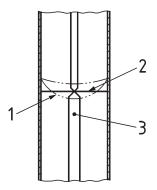
- 1 Meniscus
- 2 Graduation line
- 3 dark coloured paper

Figure 2 — Setting of convex meniscus (left) or even flat (right)

In case that an instrument showing a convex or even flat meniscus was calibrated according to the procedure noted in the previous version of ISO 4787 (upper edge of the graduation line is horizontally tangential to the lowest point of the meniscus) differences by setting meniscus with the procedure

mentioned above (upper edge of the graduation line is horizontally tangential to the highest point of the meniscus) shall be corrected. These differences can lead to very significant values with respect to the error limit.

On volumetric instruments fitted with a Schellbach ribbon, the meniscus shall be set using the constriction produced by the interaction between the meniscus and the Schellbach ribbon. Setting is done when the tip of the constriction points to the graduation line (see Figure 3).



Key

- 1 Meniscus
- 2 Graduation line
- 3 dark coloured paper

iTeh STANDARD PREVIEW Figure 3 — Setting of meniscus with Schellbach ribbon (standards.iteh.ai)

The lighting should be arranged so that the meniscus appears dark and distinct in outline. For this purpose, it should be viewed against a white background and shaded from undesirable illumination. This can be achieved, for example, by securing a strip of black or blue paper directly below the level of the graduation line or ring mark or by using a short section of thick black rubber tubing cut open at one side and of such size as to clasp the tube firmly. Parallax is avoided when the graduation lines are of sufficient length to be seen at the front and back of the volumetric instrument simultaneously.

On volumetric instruments which have graduation lines on the front only, parallax can be made negligible when making a setting on the top edge of the line by using the black shading strip, taking care that the top edge of this is in a horizontal plane. In this case, the eye shall be placed so that the front and back portions of the top edge appear to be coincident.

8.2.2 Meniscus of opaque liquids

When the volumetric instrument is used with opaque wetting liquids forming a concave meniscus, the horizontal line of sight shall be taken through the upper edge of the meniscus, and, where necessary, an appropriate correction shall be applied.

In case of a convex or even flat meniscus, the meniscus shall be set so that the plane of the upper edge of the graduation line is horizontally tangential to the highest point of the meniscus, the line of sight being in the same plane, and, where necessary, an appropriate correction shall be applied.

9 Calibration procedure

9.1 General

Volumetric instruments other than disposable pipettes shall be thoroughly cleaned shortly before calibration (see 7.3). Volumetric instruments adjusted to contain shall be dried after cleaning.

For volumetric instruments adjusted to deliver, it is important that receiving vessels manufactured from glass are used. Capillary effects influencing the delivery time and the delivered volume depend considerably on the material on which the liquid runs down. In addition, the electrostatic charges of glass are minimal; this is important for the weighing procedure.

9.2 Test room

The test shall be carried out in a draught-free room with a stable environment. The test room shall have a relative humidity (RH) between 35 % and 80 % and a temperature of (20 \pm 3) °C or (27 \pm 3) with a maximum variation of \pm 1 °C during the test.

Environmental conditions with humidity below 35 % should be strictly avoided since they facilitate the occurrence of static charges which render the weighing process very difficult.

Prior to the test, the apparatus to be tested, all test equipment, and water shall have stood in the test room for a sufficient time to reach equilibrium with the test room conditions, the temperature variation of the room during this time should not be more than $1\,^{\circ}\text{C}$ per hour. Test water should be covered to avoid evaporation cooling. Temperatures (room and calibration liquid), atmospheric pressure and humidity shall be recorded.

NOTE It is unlikely that this equilibration time will be less than 2 h and could be considerably longer.

9.3 Filling and delivery

9.3.1 Volumetric flasks and measuring cylinders PREVIEW

Volumetric flasks in accordance with ISO 1042, volumetric flasks made of plastic in accordance with ISO 5215 and measuring cylinders in accordance with ISO 4788 or ISO 6706 shall be dried after cleaning. They shall be filled by means of a plastic tube with tip to a distance of a few millimetres above the ring mark or the graduation line to be tested, so that the walls of the volumetric instrument considerably above the ring mark are not wetted. The final setting of the meniscus to the ring mark or graduation line shall be made by withdrawing the surplus water by means of a plastic tube drawn out to a jet. The movement of the meniscus when setting shall be downwards. If a little refilling is necessary or if the reading is delayed to the adjustment of the meniscus, careful swaying is necessary to refresh the meniscus shape.

9.3.2 Pipettes adjusted to deliver

Pipettes adjusted to deliver according to the specifications in ISO 648 and ISO 835, or other pipettes, e.g. plastic ones, shall be clamped in a vertical position and filled through the jet to a few millimetres above the graduation line to be tested; any liquid remaining on the outside of the jet shall be removed. The final setting of the meniscus shall then be made by running out the surplus water through the jet. Any drop of liquid adhering to the jet shall be removed, for example by bringing a ground glass surface into contact with the tip of the jet at an angle of about 30°. Draw this ground glass surface downwards through a distance of about 10 mm to remove residual water. Delivery into the tared receiving vessel shall then be made with the flow unrestricted while the tip of the jet is in contact with the inner ground surface of the receiving vessel, finally drawing it over a distance of about 10 mm, with the receiving vessel held inclined at an angle of about 30°.

Other precautions which are necessary to obtain the correct delivered volume vary with different types of instruments and are described in the clause defining capacity in the appropriate International Standards.

Determine the delivery time while the tip of the jet is in contact with the inner surface of the receiving vessel, above the level of any collected liquid, but without movement of one against the other throughout the delivery period. The delivery time thus determined should be within the limits specified for the particular pipette.