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

Verrerie et matériel en plastique de laboratoire — Instruments volumétriques — Méthodes d'essai de la capacité et d'utilisation

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 48, *Laboratory equipment*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 332, *Laboratory equipment*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

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

a) volumetric plastic ware has been included;

b) new information on meniscus adjustment of convex meniscus has been added; namely, altered procedure "Upper edge of the graduation line is horizontally tangential to the highest point of meniscus" as compared to older procedure "Upper edge of the graduation line is horizontally tangential to the lowest point of the meniscus";

c) improved figures for meniscus adjustment have been provided;

d) Table 1 has been improved;

e) new Table 2 for minimum requirements for the measurement devices has been added;

f) new test room ambient conditions have been added;

g) new information regarding repeatability and uncertainty has been added in Annex E;

h) Formula (C.1) has been 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.

Introduction

The International Standards for the individual volumetric instruments include clauses on the specification of capacity (volume); these clauses describe the method of manipulation in sufficient detail to determine the capacity without ambiguity. This document contains supplementary information.

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

1 Scope

This document 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, resulting in the determination of its error of measurement at one or more points.

This document is applicable to volumetric instruments with nominal capacities in the range of 100 µl to 10 000 ml. These include single-volume pipettes (see ISO 648), graduated pipettes (see ISO 835), burettes (see ISO 385), volumetric flasks (see ISO 1042 and ISO 5215), and graduated measuring cylinders (see ISO 4788 and ISO 6706).

The methods are not intended for testing of volumetric instruments with capacities below 100 µl such as micro-glassware.

This document does not deal specifically with pycnometers as specified in ISO 3507. However, the procedures specified for the determination of volume of glassware can, for the most part, also be followed for the determination of a pycnometer volume. For some types of pycnometers, special handling can be necessary.

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 3507, *Laboratory glassware — Pyknometers*

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

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

ISO 4797, *Laboratory glassware — Boiling flasks with conical ground joints*

ISO 5215[1)], *Laboratory plastic ware — Volumetric flasks*

ISO 6706, *Plastics laboratory ware — Graduated measuring cylinders*

ISO 24450, *Laboratory glassware — Wide-necked boiling flasks*

1) Under preparation. Stage at the time of publication: ISO/DIS 5215:2021.

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

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 terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The general procedure for testing the capacity (volume) 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 (cm^3).

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.

When the volumetric instrument is required for use in a country which has adopted a standard reference temperature of 27 °C (according to ISO 384), this figure shall replace 20 °C in Formula (1).

6 Apparatus and calibration liquid

6.1 Balance

The balance used for testing shall be chosen in accordance with the minimum requirements specified in Table 1, depending on the nominal volume of the volumetric instrument under test.

Table 1 — Minimum requirements for the balance

Nominal capacity (volume) V	**Resolution** mg	**Repeatability** mg	**Expanded uncertainty in use** U (k = 2)[a] mg
100 µl ≤ V ≤ 10 ml	0,1	0,2	0,4
10 ml < V ≤ 1 000 ml	1	2	4
V > 1 000 ml	10	10	40

[a] Expanded uncertainty in use estimated according to Reference [1] (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 specified in Table 2.

Table 2 — Minimum requirements for the measurement devices

Device	Resolution	Expanded uncertainty of measurement U (k = 2)
Thermometer for liquids	0,1 °C	0,2 °C
Thermometer for room air	0,1 °C	0,2 °C
Hygrometer	1 % relative humidity	5 % relative humidity
Barometer	0,1 kPa	1 kPa
Timing device	1 s	Not applicable

6.3 Calibration liquid

As calibration liquid during testing, distilled or de-ionized water complying with ISO 3696, Grade 3 or better should be used. The water temperature shall be within ± 0,5 °C of room air temperature.

6.4 Receiving vessel

The receiving vessel shall be a conical flask, manufactured from glass, e.g. in accordance with ISO 1773, ISO 4797, or ISO 24450, if possible, with ground joint. 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

The same sources of error are, naturally, inherent to calibration, testing and use. In calibration, every attempt is made to reduce these errors to a minimum; in use, 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.

7.2 Temperature

7.2.1 Temperature of the volumetric instrument

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 5.2).

When performing calibrations, it is important to refer to the reference temperature in the report. For example if a volumetric instrument made of borosilicate glass having a coefficient of cubic thermal expansion of 9,9 × 10^{-6} $°C^{-1}$ is calibrated at 20 °C but used at a temperature of 27 °C it would show an extra error of 0,007 %. A volumetric instrument made of soda-lime glass having a coefficient of cubic thermal expansion of 27 × 10^{-6} $°C^{-1}$ would show an extra error of 0,02 %, for a 7 °C temperature change.

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 Formula (1) (see 9.5) and Annex C. 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, 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 8.2).

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. Where volumetric instruments are fitted with ground stoppers, special attention shall be paid to cleaning the ground zone.

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

Recommended cleaning procedures are included in Annex A (for glass) and Annex B (for plastic). Other cleaning procedures can be used as well.

Fluoride containing cleaning agents should be strictly avoided in the case of glassware.

7.4 Conditions of used volumetric instruments

The surface of volumetric instruments 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.

Volumetric glassware should not be heated to a temperature considerably above 180 °C. Although the strain point of glasses used for volumetric purposes is in the range of 500 °C, alterations of volume may occur at temperatures considerably below the strain point.

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 decreases if the jet is broken (shorter delivery time) or increases 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 used.

8 Setting the meniscus

8.1 General

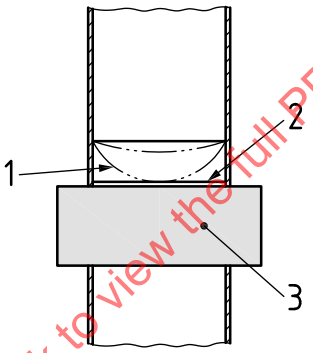
Most volumetric instruments employ the principle of setting or reading a meniscus (the interface between air and the liquid) against a graduation line or ring mark. Wherever practicable, the meniscus should descend to the position of setting.

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).

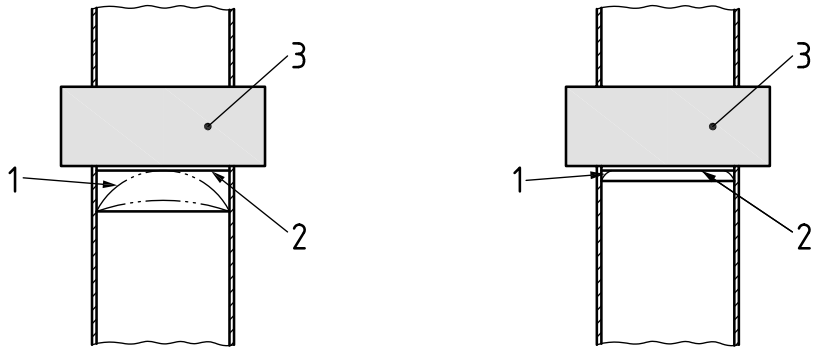


Key

1 meniscus
2 graduation line
3 dark coloured paper

Figure 1 — Setting of concave meniscus

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).

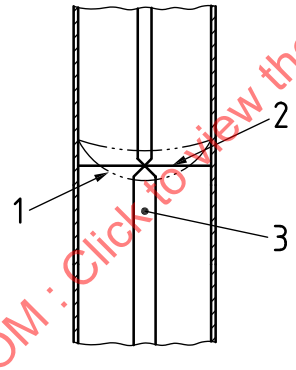


Key

1 meniscus
2 graduation line
3 dark coloured paper

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

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 Schellbach ribbon

Figure 3 — Setting of meniscus with Schellbach ribbon

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 that 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 30 % and 80 % and a temperature of (20 ± 3) °C or (27 ± 3) °C with a maximum variation of ± 1 °C during the test.

NOTE 1 Humidity below 40 % can facilitate the occurrence of static charges that render the weighing process very difficult. At humidity above 60 %, corrosion of some balances can occur [1].

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 °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 2 The equilibration time is usually about 2 h and can be considerably longer.

9.3 Filling and delivery

9.3.1 Volumetric flasks and measuring cylinders

Volumetric flasks made of glass 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 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 in accordance with the specifications given 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 that 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, ISO 648 and ISO 835.

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 in the corresponding International Standard (ISO 648 or ISO 835) for the particular pipette.

A waiting time, if specified, shall be observed before making the final setting of the meniscus for delivery of a given volume. If the setting after delivery is done at a lower graduation line, the liquid flow shall be nearly stopped a few millimetres above the graduation line. After observation of the waiting time, the final setting shall be completed quickly.

9.3.3 Pipettes adjusted to contain

Fill the pipette by suction slowly and carefully, stopping when the meniscus reaches the graduation line. Carefully wipe off any residual water from the end of the pipette.

The filled pipette shall be weighted in the balance using the appropriated means in order to measure its contained volume.

9.3.4 Burettes adjusted to deliver

Burettes adjusted to deliver in accordance with the specifications given in ISO 385 shall be clamped in a vertical position and filled to a few millimetres above the graduation line to be tested. The stopcock and jet shall be free from air bubbles. 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 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.

Delivery into the tared receiving vessel shall then be made with the flow unrestricted until the meniscus has come to a few millimetres above the graduation line to be tested, while the stopcock is fully open and the jet is not in contact with the receiving vessel. After the final setting of the meniscus, any drop of liquid adhering to the jet is removed by bringing an inclined glass surface into contact with the tip of the jet at an angle of about 30°, finally drawing it over a distance of about 10 mm.

Class A and AS burettes manufactured from standard drawn tubing should be tested at five points on the scale. Burettes manufactured from “precision bore” tubing can be tested only at three points on the scale. For more details see ISO 385.

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

Determine the delivery time by the unrestricted outflow of the liquid from the zero mark to the lowest graduation mark with the stopcock fully open and the jet not being in contact with the surface of the receiving vessel. The delivery time thus determined should be within the limits specified for the particular burette.

A waiting time, if specified, shall be observed before making the final setting of the meniscus for delivery of a given volume. If the setting after delivery is done at a lower graduation line, the liquid flow shall be nearly stopped a few millimetres above the graduation line. After observation of the waiting time, the final setting shall be completed quickly.

9.3.5 Pycnometers

The filling of a pycnometer shall be performed in accordance with the specifications given in ISO 3507 and the manufacturer's instructions.

9.4 Weighing

The volumetric instrument or the receiving vessel (see 6.4) shall be tared and weighed using a balance in accordance with 6.1 and the temperature of the water shall be measured to ± 0,1 °C.

Alternatively, two weighings can be performed (empty and loaded vessel). The corresponding balance indications are I_L, referring to the loaded vessel, and I_E, referring to the empty vessel. Usually, I_E and I_L are observed under the same conditions, hence a precise zero adjustment of the balance is not necessary. Both of the required weighings shall be carried out in a short time interval as convenient to ensure that they have been made at the same temperature. This air temperature, the humidity and the barometric pressure shall be recorded for use in the subsequent calculations.

The manufacturer's instructions shall be followed in making the required measurements. Weighings shall be made with care and made expeditiously to minimize evaporation losses which would constitute a source of error.

To perform repeated tests in flasks and cylinders two options may be used:

a) Option 1 — The flask or cylinder is dried after each run. Obtaining an initial dry weight will allow the operator to determine when the flask or cylinder is sufficiently dry.

b) Option 2 — After the first run, for the subsequent runs, a sufficient amount of liquid is removed, the temperature of the water inside the flask or cylinder is measured, the inner walls of the volumetric instrument above the graduation line to be calibrated are dried and finally the meniscus is set again. The initial value of the dried flask or cylinder (tare) is used in all repetitions.

9.5 Volume and uncertainty calculation

In order to obtain the volume contained in, or delivered by, the volumetric instrument under test at the reference temperature from the mass of water, the following factors shall be taken into account:

a) the density of water at the temperature of test;

b) the density of the air;

c) the thermal expansion of the glass or the plastic material used;

d) the effect of air buoyancy on the water and on the weights used.

The calculation of the volume at the reference temperature of 20 °C, V_{20} (at a reference temperature of 27 °C, V_{27}), from the mass of the water, contained or delivered, is given by Formula (1):

$$V_{20} = (I_L - I_E) \times \frac{1}{(\rho_W - \rho_A)} \times \left(1 - \frac{\rho_A}{\rho_B}\right) \times [1 - \gamma(t - 20)] \quad (1)$$

where

I_L is the balance indication of the vessel loaded with water, in grams;

I_E is the balance indication of the 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 grams per millilitre, obtained from Table C.3 or Formula (C.4) at the temperature, humidity 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 grams per millilitre, or, when using an electronic balance without weights, the (reference) density of the weights with which it has been adjusted;

NOTE 1 Weights conforming to Reference [2] 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.

ρ_W is the density of water at t (in degrees Celsius), in grams per millilitre, calculated with the "Tanaka" Formula, Reference [3] [see Table C.4 or Formula (C.5)];

γ is the coefficient of cubic thermal expansion of the material of which the volumetric instrument tested is made, in reciprocal degrees Celsius (see Table D.1);

t is the temperature of the water used in the test, in degrees Celsius.

The balance indication after tare or the difference of the results of the first and second weighing is the mass of the water contained in, or delivered by, the volumetric instrument under test.

NOTE 2 The mass, thus obtained, is the mass uncorrected for air buoyancy.

To facilitate the calculation of the instrument's volume V_{20}, a factor Z may be introduced in Formula (1). More details can be found in Annex C.

A major source of error associated with this measurement is in the adjustment of the meniscus, which will depend on operator care and is related to the cross-section of the tubing where the meniscus is located.

If uncertainty is estimated the repeatability shall be included.

Guidance on the uncertainty estimation of the volume value obtained by application of Formula (1) and information on repeatability of measurements are given in Annex E and Reference [4].

10 Procedure for use

10.1 General

Where the greatest attainable accuracy is required, volumetric instruments shall be handled in a manner as similar as possible to that employed during calibration as described in Clause 9. For further details, see the relevant clause "Definition of capacity" or "Basis of adjustment" in the appropriate International Standards.

Always clean volumetric instruments before use (see 7.3) and check the jet for possible damage and unrestricted outflow of liquid with volumetric instruments adjusted to deliver.

According to 7.5, the delivered volume of liquid with instruments adjusted to deliver depends on the delivery time (specified in the appropriate standards) and physical properties of the liquid. The accuracy in use can vary when viscosity and/or surface tension are different from that of water. Dilute aqueous solutions, such as are ordinarily employed in volumetric analysis, can often be used without significant error.

Liquids which are too opaque for the bottom of the meniscus to be visible may be read on the "upper edge" of the meniscus, with rather less accuracy and precision than is possible when viewing the lowest point of the meniscus.

The temperature of use is also important. Whereas the expansion of the volumetric instrument itself is negligible, the expansion of liquid shall be considered. Care shall be taken when preparing a solution (e.g. a standard solution) by adding known volumes of two or more liquids measured by volumetric instruments. Preferably the temperatures of the individual liquids should be as close as possible to each other and also to the temperature of the volumetric instrument containing the solution to be prepared. If these temperatures are significantly different from each other, corrections should be applied to account for thermal expansion effects of the liquids.

Mixing of liquids and preparation of solutions can cause thermal changes and non-ideal behaviour. These effects should be taken into account when measuring volume.

10.2 **Volumetric flasks** (in accordance with ISO 1042 or ISO 5215)

The procedure of setting the meniscus with respect to a given ring mark shall reproduce the conditions of calibration and is illustrated by the following example in the case of a dilute aqueous solution.

— Introduce the solid material and add sufficient water to dissolve it by carefully swaying the flask without contaminating the surface above the graduation line (if necessary, this process can be assisted by no more than moderate warming.).

— Then, while still swaying the flask to mix its content, add more water to bring the liquid surface to within a few centimetres below the graduation line.

— Close the flask and shake it upside down to mix the contents, then carefully remove and rinse the stopper, gathering the water in the flask to bring the liquid surface to within 1 cm below the graduation line.

— Leave the flask to stand without its stopper for 2 min to allow the liquid in the neck to drain. If necessary, wait for the solution to regain room temperature. During the waiting time, the rinsed and dried stopper may be replaced.

— Then set the meniscus on the graduation line by running the necessary water down the neck from a point less than 10 mm above the graduation line (see 8.2)

— Finally, close the volumetric flask and shake it by multiple inversions for thorough mixing.

10.3 **Measuring cylinders** (in accordance with ISO 4788 or ISO 6706)

To set the meniscus precisely (see 8.2), fill the measuring cylinder with the relevant liquid to a few millimetres above the nominal capacity line or selected graduation line. Wait 2 min to allow liquid in the cylinder to drain. Then set the bottom or the top of the meniscus, depending on whether is a concave, convex or even flat one, on the graduation line by withdrawing the surplus of liquid by means of a tube drawn out to a jet.

10.4 **Burettes** (in accordance with ISO 385)

After rinsing with the liquid or reagent to be used, prime the stopcock and fill the burette, clamped in a vertical position, a few millimetres above the zero graduation line. Wait 2 min for drainage before setting the meniscus at the zero line. Now, titration can be performed until the end point is reached. The meniscus reading at the relevant graduation line gives the volume that has been delivered.

In practice, a burette is generally not employed in the same way as it is tested. Typically, in use, the approach to the finally desired delivery point is made dropwise, to avoid over delivery, and frequently takes a period of time that is similar to, or even greater than, any specified waiting time observed during testing. Therefore, it follows that in use, the waiting time, if specified, need generally not to be observed.

10.5 Pipettes

WARNING — Use an appropriate pipetting aid for filling to avoid any danger to the operator. Always hold the pipette at the top while inserting in the aspiration adapter because pipettes can break and cause injury. Pipetting aids which allow the unrestricted outflow of the liquid should be used.

10.5.1 Pipettes adjusted to deliver (see ISO 648 and ISO 835, or other pipettes, e.g. plastic ones)

After rinsing with the liquid or reagent to be used, fill the pipette by suction to a few millimetres above the selected graduation line. Remove any liquid remaining on the outside of the jet.

The final setting of the meniscus shall then be made by dispensing the surplus liquid through the jet. Remove any drops of liquid adhering to the jet by bringing an inclined ground glass vessel into contact with the tip of the jet. Delivery shall then be made with the tip of the jet in contact with the inner surface of the inclined receiving vessel.

If the setting after delivery is done at a lower graduation line, the liquid flow shall be nearly stopped a few millimetres above the graduation line. After observing a waiting time, if specified, complete the final setting quickly.

A waiting time, if specified, shall be observed before making the final setting for delivery of a given volume.

10.5.2 Pipettes adjusted to contain

If the pipette is not dry, rinse the pipette with the reagent to be used to a few millimetres below the desired graduation line. Fill the pipette by suction to the graduation line. Remove any liquid remaining on the outside of the jet. For complete discharge, rinse the pipette several times with the diluting medium.

10.6 Pycnometers

Pycnometers shall be used in accordance with the specifications given in ISO 3507 and the manufacturer's instructions.

Annex A
(informative)

Cleaning of volumetric glassware

A.1 The volume contained in or delivered by volumetric glassware depends on thorough cleaning of the entire internal surface in order to ensure uniform wetting and formation of a well-shaped meniscus.

A.2 Glassware can be cleaned manually, in an immersion bath or in a laboratory washing machine. To reduce volume changes through glass erosion and destruction of graduations, gentle cleaning should be performed immediately after use with detergents of low alkalinity at temperatures below 70 °C with short contact time. The cleanliness of the inner glass surface should be ascertained as specified in 7.3.

A.3 If the inner glass walls are not sufficiently clean after the above treatment, the volumetric instrument should be filled with a mixture of equal parts of a 30 g/l solution of potassium permanganate ($KMnO_4$) and 1 mol/l solution of sodium hydroxide (NaOH). After about 2 h, a residue of MnO_2 may be removed by means of dilute hydrochloric acid or oxalic acid.

The volumetric instrument should then be rinsed with distilled water and it should again be ascertained that the walls are sufficiently clean. If they are not, the procedure should be repeated. If this treatment is not successful, specific cleaning methods described in laboratory handbooks should be applied. The method shall not change the volume of the instrument.

Annex B
(informative)

Cleaning of volumetric plasticware

B.1 The volume contained in or delivered by volumetric plasticware depends on thorough cleaning of the entire internal surface in order to ensure uniform wetting and formation of a well-shaped meniscus.

B.2 Plasticware can be cleaned manually in an immersion bath or in a laboratory washing machine. Polyolefins, such as PP and PMP, as well as the fluoroplastic PFA have water-repellent surfaces that are very durable and easy to clean. To reduce volume changes through erosion and destruction of graduations, gentle cleaning should be performed immediately after use with detergents of low alkalinity at temperatures below 60 °C with short contact time.

Annex C
(normative)

Calculation formulae and tables

C.1 General calculation

C.1.1 The calculation of the volume at the reference temperature of 20 °C, V_{20} (at a reference temperature of 27 °C, V_{27}), from the mass of the water, contained or delivered, is given by Formula (1) (see 9.5).

C.1.2 In order to give an estimate of the extent to which the various parameters originated from the weighing procedure influence the result, some parametric tolerances, with the corresponding error in the volume determined, are given in Table C.1.

Table C.1 — Examples for volumetric errors

Parameter	Parametric tolerance	Volumetric error relative to the volume[a]
Water temperature	±0,5 °C	$\pm 10^{-4}$
Air pressure	±8 mbar (0,8 kPa)	$\pm 10^{-5}$
Air temperature	±2,5 °C	$\pm 10^{-5}$
Relative humidity	±10 %	$\pm 10^{-6}$
Density of weights	±0,6 g/ml	$\pm 10^{-5}$

[a] Example: A relative volumetric error of $\pm 10^{-4}$ to the measured volume of 100 ml would be 0,01 ml.

C.1.3 The largest source of experimental error associated with the determination of volume is in the setting of the meniscus which depends on operator care, the cleaning of the instrument and is related to the cross-section of the tubing where the meniscus is located. Some typical values are given in Table C.2. It is preferable that the user determines the particular experimental error. In case this is not possible the values from Table C.2 can be used.

Table C.2 — Indicative error related to the setting of meniscus

Error in meniscus position mm	Volume error at neck diameter µl			
	5 mm	10 mm	20 mm	30 mm
0,05	1	4	16	35
0,1	2	8	31	71
0,5	10	39	157	353
1	20	78	314	707
2	39	157	628	1 414

C.1.4 When the temperature at which the volumetric instrument is used (t_2) differs from the reference temperature (t_1), the volume of the volumetric instrument at t_2 can be calculated from Formula (C.1):

$$Vt_2 = Vt_1 [1 + \gamma(t_2 - t_1)] \tag{C.1}$$

where γ is the coefficient of cubic thermal expansion of the material of the volumetric instrument (see Table D.1). For information on the effect of temperature differences, see 7.2.1.

C.2 Tables for calculation

C.2.1 To facilitate an easy calculation of the instrument's volume V_{20} at a reference temperature of 20 °C from the mass obtained by using a balance, a factor Z can be introduced in Formula (1):

$$V_{20} = (I_L - I_E) \times Z \tag{C.2}$$

Table C.5, Table C.6 and Table C.7 give factor Z conversion values for different types of glass at common air pressure versus temperature. In these tables, the combined effects of the density of the water, the cubic thermal expansion of the glass and the air buoyancy have been taken into account. The density of weights used for the balance adjustment is ρ_B = 8,0 g/ml.

The factor Z conversion values have been derived from Formula (1) as follows:

$$Z = \frac{1}{(\rho_W - \rho_A)} \times \left(1 - \frac{\rho_A}{\rho_B}\right) \times [1 - \gamma(t - 20)] \tag{C.3}$$

C.2.2 For the calculation of the volume according to Formula (1) (see 9.5), Table C.3, Table C.4 and Table D.1 list the necessary values for ρ_A, ρ_W and γ.

The density of air in Table C.3 is given for a relative humidity of 50 % and a content of 0,04 % by volume carbon dioxide. In practice, usual deviations from these conditions, e.g. a relative humidity in the range of 30 % to 80 %, will introduce negligible error without significance for the purposes of this document.

The simplified Formula (C.4) [5] for the air density can be used under the constraints given below:

$$\rho_A = \frac{0{,}348\ 48p - 0{,}009 h_r \exp(0{,}061t)}{t + 273{,}15} \times \frac{1}{1\ 000} \tag{C.4}$$

where

ρ_A is the air density, in g/ml;

t is the ambient temperature, in °C;

p is the barometric pressure, in hPa;

h_r is the relative air humidity, in %.

Under the following conditions: barometric pressure between 600 hPa and 1 100 hPa, ambient temperature between 15 °C and 27 °C and relative humidity between 20 % and 80 %, the relative uncertainty of the air density calculated using Formula (C.4) is 2,4 × 10^{-4} [1].

The density of water in Table C.4 is based on Tanaka et al., see Reference [3].

$$\rho_W = a_5 \left[1 - \frac{(t + a_1)^2 (t + a_2)}{a_3 (t + a_4)}\right] \tag{C.5}$$