



SLOVENSKI STANDARD

SIST ISO 4787:1995

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Laboratorijska steklovina - Volumetrijska steklovina - Metode za uporabo in preskušanje kapacitete

Laboratory glassware -- Volumetric glassware -- Methods for use and testing of capacity

Verrerie de laboratoire -- Verrerie volumétrique -- Méthodes d'utilisation et de vérification de la capacité

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71.040.20	Laboratorijska posoda in aparati	Laboratory ware and related apparatus

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International Standard



4787

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Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity

Verrerie de laboratoire — Verrerie volumétrique — Méthodes d'utilisation et de vérification de la capacité

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4787 was prepared by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*.

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Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity

1 Scope and field of application

This International Standard provides methods for the testing of volumetric glassware in order to obtain the best accuracy in use.

The International Standards for the individual articles include clauses on the definition of capacity, which describe the method of manipulation in sufficient detail to define the capacity without ambiguity. This International Standard is supplementary to the information contained in these definitions.

The procedures are applicable to small-capacity ware, usually defined as items with capacities in the range of 0,1 to 2 000 ml. These include transfer and one-mark pipettes without subdivisions; graduated measuring pipettes and dilution pipettes, with partial or complete subdivisions; burettes; volumetric flasks; graduated measuring cylinders. The procedures are not recommended for testing of apparatus with capacities below 0,1 ml, such as microglassware, for example.

NOTES

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

2 This International Standard does not deal specifically with pyknometers as specified in ISO 3507. However, the procedures specified below for the determination of volume of glassware can, for the greater part, also be followed for the calibration of pyknometers.

2 References

ISO 384, *Laboratory glassware — Principles of design and construction of volumetric glassware.*

ISO 385/1, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 385/2, *Laboratory glassware — Burettes — Part 2: Burettes for which no waiting time is specified.*

ISO 385/3, *Laboratory glassware — Burettes — Part 3: Burettes for which a waiting time of 30 s is specified.*

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 835/1, *Laboratory glassware — Graduated pipettes — Part 1: General requirements.*

ISO 835/2, *Laboratory glassware — Graduated pipettes — Part 2: Pipettes for which no waiting time is specified.*

ISO 835/3, *Laboratory glassware — Graduated pipettes — Part 3: Pipettes for which a waiting time of 15 s is specified.*

ISO 835/4, *Laboratory glassware — Graduated pipettes — Part 4: Blow-out pipettes.*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3507, *Pyknometers.*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders.*

3 Summary of method

The general procedure is based upon a determination of volume of water either contained in or delivered by the vessel.

This volume of water is based upon knowledge of its mass and its tabulated density.

4 Definitions

For the purpose of this International Standard, the following definitions apply (see also ISO 384).

4.1 Unit of volume

The unit of volume shall be the cubic centimetre (cm³) or, in special cases, the cubic decimetre (dm³) or cubic millimetre (mm³) for which the names millilitre (ml), litre (l) or microlitre (μl) may be used.

NOTE — The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm³) [and, similarly, the litre (l) for the cubic decimetre (dm³) and the microlitre (μl) for the cubic millimetre (mm³)], in accordance with a decision of the twelfth Conférence Générale des Poids et Mesures. The term millilitre is acceptable, in general, for references in International Standards to capacities of volumetric glassware and it is used, in particular, in the present text.

4.2 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.

NOTE — When it is necessary in tropical countries to work at an ambient temperature considerably above 20 °C, and these countries do not wish to use the standard reference temperature of 20 °C, it is recommended that they adopt a temperature of 27 °C.

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5 Apparatus and materials

5.1 Balance

A laboratory balance is required with sufficient capacity to weigh the loaded vessel. The discrimination of the balance will be a limiting factor in the accuracy of the measurements. Either a single-pan, self-indicating instrument or an equal-arm balance of adequate discrimination and capacity may be used. The balance shall have a discrimination not greater than 1/10 of the limits of error of the instrument to be tested. In either case, the instrument shall be calibrated with adequate accuracy (see 9.3). The balance shall have dimensions to accept the size of the vessels which need to be weighed.

5.2 Thermometer

A thermometer is required to measure the temperature of the water. Its limits of error shall be 0,1 °C (see 9.5).

5.3 Barometer

A barometer capable of providing atmospheric pressure measurements consistent with appropriate tolerances is required.

NOTE — The barometer should preferably have limits of error of 1 mbar.¹⁾

5.4 Water

Distilled or deionized water, suitable for general laboratory purposes, shall be used.

6 Factors affecting the accuracy of volumetric laboratory ware

6.1 General

The same sources of error are, naturally, inherent both in testing and use. In the former, every attempt is made to reduce these errors to a minimum; in the latter, the care needed is dependent upon the degree of accuracy required; when the greatest possible accuracy is desired, the article should be used as nearly as possible in the manner in which it is tested.

6.2 Temperature

6.2.1 Temperature of the vessel

The capacity of a glass vessel varies with change of temperature; the particular temperature at which a vessel is intended to contain or deliver its nominal capacity is the "reference temperature" of the vessel (see 4.2).

NOTE — The coefficient of cubical thermal expansion of glass from which volumetric glassware is manufactured falls in the approximate range 10×10^{-6} to $30 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$. A vessel made of soda-lime glass having a coefficient of cubical thermal expansion of $30 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, which was adjusted at 20 °C but used at 27 °C would, at the temperature of use, show an extra error of only 0,02 %, which is smaller than the limits of error for most articles of volumetric glassware. It follows, therefore, that the reference temperature is of minor importance in practical use of the glass vessel, but in order to provide a sound basis for adjustment (see B.1.4) it is important to specify a reference temperature and the vessel should be equilibrated at that temperature before testing.

6.2.2 Temperature of liquid

The temperature of the water used for the testing of volumetric glassware shall be accurately measured to within $\pm 0,1 \text{ } ^\circ\text{C}$. Corrections for differences in temperature from the reference temperature shall be applied in accordance with annex B.

When using volumetric glassware ensure that all solutions used in connection with each other are close to a common temperature when their volumes are measured.

6.3 Cleanliness of glass surface

The volume contained in or delivered by a glass vessel depends on the cleanliness of the internal glass surface of the vessel. Lack of cleanliness can give rise to error through a badly shaped meniscus involving two defects:

- incomplete wetting of the glass surface, i.e. the liquid surface meets the glass at an appreciable 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.

In vessels used for delivery, lack of cleanliness can cause additional errors due to the film of liquid on the walls being irregularly distributed or incomplete.

In use, as distinct from testing, chemical contamination can introduce an error even though it has no influence on the accuracy of volume measurement.

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

Therefore, where vessels are fitted with ground stoppers, special attention shall be paid to cleaning the ground zone.

A satisfactory method of cleaning is described in annex A. To ascertain whether a piece of glass apparatus is satisfactorily clean, it shall be observed during filling. A delivery vessel should preferably be filled from below the liquid surface (i.e. through the stopcock in the case of a burette or through the jet of a pipette). The rising liquid meniscus shall not change shape

1) 1 mbar = 100 Pa

(i.e. it shall not crinkle at its edges). After over-filling and withdrawing a little liquid (through the jet in the case of a delivery vessel, by means of a drawn-down glass tube in the case of a content vessel), the surface of the glass above shall remain uniformly wetted and the meniscus shall not crinkle at its edges. Additionally, an experienced operator can recognize the shape of an uncontaminated meniscus, in relation to its diameter.

7 Setting of the meniscus (see the figure)

Most items of volumetric glassware employ the principle of setting or reading a meniscus (the interface between air and the liquid the volume of which is being measured) against a reference line or scale.

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. In the case of a mercury meniscus, however, the highest point of the meniscus shall be set to the lower edge of the graduation line. When the article is used with opaque wetting liquids, the horizontal line of sight shall be taken through the upper edge of the meniscus, and, where necessary, an appropriate correction shall be applied. (See the figure.)

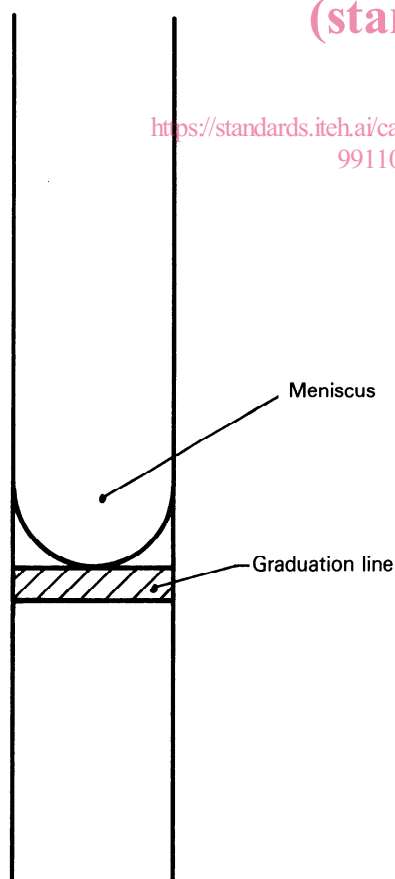


Figure – Setting of the meniscus

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 paper round the vessel not more than 1 mm below the level of the setting 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 vessel simultaneously. On apparatus provided with 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 Delivery time

For articles 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 walls of the vessel. The volume of this film depends on the time taken to deliver the liquid, and the volume delivered decreases with decreasing delivery time. It follows, therefore, that such a vessel can deliver a particular volume for one value only of the delivery time. The shorter the specified delivery time, the greater is the variation in the volume delivered due to small variations in delivery time which inevitably occur. Provided that the delivery time is never less than a certain value, the volume of the residual film is sufficiently small and uniform to ensure that departures from the nominal delivery time which occur in practice have a negligible effect on the volume delivered and that the drainage occurring after delivery is negligibly small.

The same effect may be achieved by splitting the time into a significantly shorter delivery time and a definite waiting time. It follows that the jet shall not be interfered with. Any alteration of the jet in order to increase the speed of delivery will cause the scale reading to be in error, which cannot be estimated, as well as decrease the consistency of reading.

In view of the above, delivery times are specified in the International Standards on volumetric glassware adjusted for delivery, using water as the liquid. The delivery ranges should be specified so that no reasonable differences in volume will appear if the actual delivery time varies in that range caused, for example by traces of dust. As a safeguard, nevertheless, the delivery time may be marked on burettes and pipettes made to Class A tolerances to enable the user to check whether the jet has become blocked or damaged, by measuring the delivery time. Such inscription is required, in some countries, by legal metrology authority.

9 Testing procedure

9.1 General

The vessel to be tested shall be cleaned and kept filled with pure water until shortly before required (see annex A). Vessels adjusted to contain shall then be dried, for example by rinsing with ethanol and using a current of warm air. Vessels adjusted to deliver shall be appropriately cleaned. Disposable pipettes need not be cleaned before testing.

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9.2 Temperature conditions

All tests shall be carried out in a room, the temperature of which is constant to at least 1 °C/h.

Make sure that the vessel or weighing bottle and the water are at room temperature.

9.3 Tare

The vessel to be tested, or a weighing bottle if a vessel intended for delivery is to be tested, shall be appropriately weighed, i.e. to an accuracy better than 10 % of the tolerance laid down.

9.4 Filling

A vessel adjusted to contain shall either be filled to a distance of a few millimetres above the graduation line to be tested; the final setting to the line shall be made by withdrawing the surplus water by means of a glass tube drawn down to a jet or, in the case of pipettes adjusted to contain, by means of filter paper. Alternatively, the walls of the vessel shall be completely wetted for a considerable distance above the graduation line to be tested. The vessel shall be filled to a few millimetres below the graduation line by running water down the wetted wall of the neck. Two minutes drainage time shall be observed; the final setting shall then be made by discharging the required water against the wall about 1 cm above the graduation line and rotating the vessel to re-wet the wall uniformly.

Vessels adjusted to deliver shall be clamped in a vertical position and filled to a few millimetres above the graduation line to be tested; any liquid remaining on the outside of the jet shall be removed. The setting 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 an inclined glass surface into contact with the tip of the jet. Delivery into the tared weighing bottle shall then be made with the flow unrestricted. Other precautions which are necessary to obtain the correct delivered volume vary from vessel to vessel and are described in the appropriate standards in the clause defining capacity.

9.5 Weighing

The filled vessel or weighing bottle shall be weighed to the same accuracy as in 9.3 and the temperature of the water shall be measured using a thermometer graduated and accurate to 0,1 °C, either situated in the water supply line or inserted in the filled vessel after weighing.

Two weighings are required, namely I_L , referring to the loaded vessel, and I_E , referring to the empty vessel. Normally, I_E and I_L are observed under the same conditions, hence a precise zero adjustment of the balance is not necessary. Either a single-pan balance or a double-pan balance may be used. In the latter case, a vessel similar to the one being weighed is placed on the opposite pan, during both weighings, to serve as a tare. Both of the required weighings shall be carried out in as short a time-interval as convenient to ensure that they have been made under similar conditions. The temperature of the air in the balance housing and the barometric pressure shall be recorded for use in the subsequent calculations.

The manufacturer's instructions shall be followed in making the requisite measurements. Weighings shall be made with care and made expeditiously to minimize evaporation losses which would constitute a source of error. The balance used shall be in good working order. The vessels that are weighed shall be clean and shall be handled carefully to avoid contamination. They may be wiped with a clean cotton cloth as required. Handling with clean cotton gloves is considered to be good practice.

9.6 Evaluation

The difference of the results of the first and second weighings is the apparent mass of the water contained in or delivered by the tested vessel.

NOTE — The apparent mass, thus obtained, is the mass not corrected for air buoyancy.

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

- the density of water at the temperature of test;
- the thermal expansion of the glass between the temperature of test and the reference temperature;
- the effect of air buoyancy on the water and on the weights used.

Instructions for calculating the volume of the vessel at the reference temperature of 20 °C, in which these factors have been taken into account, are given in annex B.

10 Use

10.1 General

Where the greatest attainable accuracy is required, a vessel shall be manipulated in a manner as similar as possible to that employed during testing, and corrections for scale error shall be used. The vessel shall be cleaned before use (see annex A). If, during test, deviations from indicated volumes were noticed, the appropriate corrections shall be applied.

10.2 Flasks (see ISO 1042)

A flask need not be dried after cleaning and rinsing with distilled water, if it is to be used for making up an aqueous standard solution.

The procedure for setting of the meniscus on the line shall reproduce the conditions of test and is illustrated by the following example in the case of dilute aqueous solution. Introduce the material to be dissolved with sufficient water to dissolve it by shaking, assisted by no more than moderate warming, if necessary. Then add water to bring the liquid surface to within a few centimetres of the graduation line. Plug, mix, then rinse by gathering the water in the flask to bring the liquid surface to within 1 cm of the graduation line. Leave the flask to stand without its stopper for 2 min to allow liquid in the neck to drain. If it is necessary to wait a further time for the solution to regain room temperature, then the stopper may be replaced. Then set

the bottom of the meniscus on the line by running the necessary water down the neck from a point less than 1 cm above the graduation line.

Finally, shake the flask thoroughly, after the stopper has been replaced, and check for the correct level of solution in the flask.

10.3 Cylinders (see ISO 4788)

After cleaning and drying, fill the cylinder with the relevant liquid to a few millimetres above the nominal capacity line or selected graduation line. Then withdraw the surplus of liquid by means of a glass tube drawn down to a jet.

10.4 Burettes (see ISO 385/1, ISO 385/2 and ISO 385/3)

After cleaning and rinsing with distilled water, rinse a burette (including the stopcock and jet) with the reagent to be used.

Clamp a plain glass test tube, large enough to hold a thermometer, near the burette if the burette is of such a size that it is not large enough to insert a thermometer in the top for observing the temperature of the liquid.

Fill the burette, clamped in a vertical position, without wetting the walls above the zero graduation line for more than a few millimetres. If the walls do become wetted, allow adequate time for drainage before setting the zero line. The stopcock and jet shall be free from air bubbles and shall be filled prior to setting the meniscus by running some liquid out through the jet.

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. The best accuracy is attained when scale corrections are used and delivery takes place with the stopcock fully open and the jet not being in contact with the receiving vessel or with the liquid surface in that vessel, as in test. For titration therefore, it is desirable to know roughly what volume of reagent is going to be required to reach the end-point; this can be achieved by carrying out a preliminary titration if sufficient sample is available. If this is not possible, the error incurred will in general be less than 0,5 t ml, where the capacity tolerance is $\pm t$ ml, provided that the titration time does not exceed the natural delivery time by more than 60 s. A waiting time, if specified, shall be observed before making the final setting for delivery of a given volume. A waiting time should normally not be observed when performing a titration, since establishing the end-point of the titration will in general take more time than the specified waiting time.

The above remarks apply to the use of a burette with transparent liquids with a viscosity not very different from water. Very viscous liquids cannot accurately or easily be used in burettes because of the quantity left on the walls and the slow rate of flow. Dilute aqueous solutions, however, such as are ordinarily employed in volumetric analysis, can be used without significant error; for example 1 mol/l solutions introduce errors smaller than Class A tolerances and 0,1 mol/l solutions introduce correspondingly smaller errors. The ac-

curacy also deteriorates when using non-aqueous liquids, since their surface tension may differ considerably from that of water.

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 precision than is possible when viewing the lowest point of the meniscus.

10.5 Pipettes

10.5.1 Pipettes adjusted to deliver (see ISO 648 and ISO 835).

After cleaning and rinsing with distilled water, rinse a pipette with the reagent to be used.

Fill the pipette by suction to a few millimetres above the zero line or selected graduation line.

WARNING — If the pipette is to be filled with any potentially dangerous liquid, it is essential that an appropriate pipetting aid be used in order to avoid danger to the operator. This rule applies to poisonous and corrosive liquids, and to all biological fluids because of the potential risk of infection. It is recommended that pipetting aids which allow the unrestricted outflow of the liquid should be used.

To obtain the correct delivered volume, handle the pipette in the manner described under "definition of capacity" in the appropriate International Standard.

Observe any waiting time specified before removing the pipette from contact with the receiving vessel.

The waiting time of 3 s in the case of pipettes for delivery down to the jet is not critical and does not require timing; remove the pipette from contact with the receiving vessel as soon as it is certain that the meniscus has come to rest.

The drop remaining in the jet shall not be expelled except in the case of "blow-out" type pipettes in which the last drop forms a part of the volume to be delivered (see ISO 835/4). As with burettes, very viscous liquids cannot accurately or easily be used in pipettes. No significant error is introduced using dilute aqueous solutions such as are ordinarily used in volumetric analysis.

10.5.2 Pipettes adjusted to contain

After cleaning and rinsing with distilled water, dry or rinse out a pipette with the reagent to be used. Fill the pipette by suction (see the note in 10.5.1) to as close as possible above the total capacity line or selected graduation line.

To obtain the correct contained volume, handle the pipette in the manner described under "definition of capacity" in the appropriate International Standard.