

Australian Standard[®]

**Verification and use of
volumetric apparatus**

**Part 1: General—Volumetric
glassware**

This Australian Standard was prepared by Committee CH/1, Laboratory Glassware and Related Apparatus. It was approved on behalf of the Council of Standards Australia on 31 January 1996 and published on 5 May 1996.

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Australian Government Analytical Laboratories
Environmental Protection Authority, N.S.W.
National Association of Testing Authorities, Australia
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**Part 1: General—Volumetric
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PREFACE

This Standard was prepared by the Standards Australia Committee CH/1 on Laboratory Glassware and Related Apparatus to revise the 1979 edition of AS 2162, *Code of practice for the use of volumetric glassware*, which it now supersedes.

The objective of this Standard is to describe procedures for the verification of volumetric glassware using water or mercury as the working substance, and give recommended practices for the use of such glassware. The Standard was originally published in 1949 to assist persons engaged in the manufacture, verification and use of volumetric glassware.

Attention is drawn to the fact that the attainment of a high level of accuracy depends upon the use of satisfactory techniques as well as that of matching the quality of the glassware to the task on hand. In regard to the latter, it is recommended that the use of Class A glassware be restricted to those operations which necessitate attainment of the highest degree of accuracy.

The units of volume used in this code are the litre (L) and the millilitre (mL). In terms of the SI units the litre is equivalent to 10^{-3}m^3 (1 dm^3) and the millilitre is equivalent to 10^{-6}m^3 (1 cm^3).

This Standard is essentially the same as the previous edition but has been reformatted and includes a new clause dealing with apparatus and material. Clauses considering factors affecting accuracy have also been rewritten.

The terms 'normative' and 'informative' have been used in this Standard to define the application of the appendix to which they apply. A 'normative' appendix is an integral part of a Standard, whereas an 'informative' appendix is only for information and guidance.

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STANDARDS AUSTRALIA

Australian Standard

Verification and use of volumetric apparatus

Part 1: General—Volumetric glassware

1 SCOPE This Standard provides guidance for users wishing to verify or to calibrate their own articles of volumetric glassware by means of water or mercury (when more appropriate) and to enable them to obtain the best accuracy in use.

International Standards and product Standards issued by Standards Australia for individual articles of glassware, include clauses on tolerances and the definition of capacity. These describe in sufficient detail and without ambiguity the method of manipulation to define the capacity. This Standard should be regarded as supplementary to the information contained in glassware product Standards.

2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS

2243 Safety in laboratories

2243.2 Part 2: Chemical aspects

4163 Glassware—Guide to the use of piston operated volumetric apparatus (POVA)

CSIRO

Tech Paper No. 1 The calibration and verification of volumetric measures

3 DEFINITIONS For the purpose of this Standard the definitions below apply.

3.1 Accuracy—A qualitative term describing the degree of closeness with which the indications of an instrument approach the true values of the measurand.

3.2 Calibration—all the operations for the purpose of determining the values of the errors of measuring instruments, material measures, and measurement standards (and, if necessary, to determine other metrological properties, including influence quantities).

3.3 Capacity—the nominal volume of water (or mercury) contained, or delivered by an article of volumetric glassware, at its reference temperature.

3.4 Error of measurement—The discrepancy between the result of the measurement and the value of the measurand.

NOTES:

1 The value of the measurand is a comparison value and, according to the particular case, equal to —

(a) the true value of the quantity (generally unknown);

(b) the accepted true value; or

(c) the arithmetic mean of the results of a series of measurements.

2 The discrepancy can be expressed as the difference between these two values, or as the quotient of that difference and the value of the measurand.

3.5 Measurand—A physical quantity, property or condition which is measured.

NOTE: The term ‘measurand’ is preferred to ‘quantity measured’, ‘measured quantity’ and ‘quantity to be measured’.

3.6 Precision—Precision is a qualitative term to express the resolution or the repeatability of a particular measurement.

NOTE: High precision results in low scatter. Repeated measurements of a measurand, performed with high precision, give a distribution curve with a small standard deviation.

3.7 Verification—the testing of measuring instruments, material measures and measurement standards for conformity with a specification.

4 APPARATUS AND MATERIALS

4.1 Balance A laboratory balance of sufficient capacity is required to weigh the loaded vessel which is to be calibrated or verified. The balance shall have dimensions that accept the size of vessel which is to be weighed.

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 a tenth of the tolerance of the vessel to be tested. In either case, the instrument shall be calibrated with adequate accuracy.

The balance shall be calibrated against certified masses using an appropriate procedure.

NOTE: Suitable calibration procedures may be found in PROWSE, D.B., *The calibration of balances*, Melbourne: CSIRO, Australia, 1985.

4.2 Thermometer A thermometer is required which will be capable of reading to $\pm 0.1^\circ\text{C}$ of the test temperature.

4.3 Barometer A barometer is required which will provide atmospheric pressure measurements to ± 0.1 kPa.

NOTE: It is not necessary to measure the barometric pressure, unless abnormal barometric conditions are prevailing and it is desired to attain a precision of better than 1 part in 2×10^4 . Table E6 gives the correction to be applied when weighings are made at an altitude of greater than 150 m.

4.4 Water Distilled or deionized water suitable for laboratory purposes should be used.

NOTE: The quality of the water should be checked for the presence of contaminants.

4.5 Pipette filler A suitable suction device used when filling pipettes.

5 FACTORS AFFECTING ACCURACY

5.1 General The most important factor affecting the accuracy of an item of volumetric glassware is the internal diameter in the plane of the graduation line. However, the diameter in the part bearing the graduation line or scale cannot be reduced below a reasonable value because—

- (a) in volumetric flasks, cleaning would become difficult;
- (b) in bulb pipettes, a very narrow suction tube would make accurate setting difficult on account of the high velocity of the meniscus in the tube; and
- (c) burettes and graduated measuring cylinders would become too long.

When the greatest possible accuracy is desired in use, the article shall be used as nearly as possible in the manner in which it was verified.

5.2 Temperature

5.2.1 Effects Changes in temperature are of importance both in the verification and use of volumetric glassware.

Vessels should preferably not be heated to temperatures above 60°C as they may undergo permanent changes in volume. Where prolonged or repeated heating above 100°C is necessary, the glassware shall be frequently recalibrated.

5.2.2 Temperature of vessel Since the capacity of a glass vessel varies with change of temperature, any given vessel can be correct at only one temperature; the particular temperature at which a vessel is intended to contain or deliver its nominal capacity is the ‘reference temperature’ of the vessel, which in Australia, is 20°C.

The effect of change in temperature on the capacity of a glass vessel is relatively small, amounting to about 3 parts per 100 000 of the capacity for a change of 1°C for vessels of soda glass, and about 1 part per 100 000 for those of borosilicate glass. Even for changes in temperature as great as 7°C, the effects are smaller than the precision of measurement attainable for most articles of volumetric glassware. It is, nevertheless, important to specify a reference temperature to provide a sound basis of verification. The temperature of the vessel concerned shall be recorded. For verification it is also necessary to take into account the type of glass used in the construction of the vessel, so that correct allowance can be made for its change of capacity, between the test temperature and the reference temperature.

5.2.3 Temperature of liquid The coefficient of cubical expansion of the water or mercury used for verification, and also that of any liquid used in the analysis for which the glassware is intended, is much greater than that of the glass. In the temperature range concerned, the dilatation of both water and mercury is such that the change in volume due to a change in temperature of 1°C is very close to 2 parts in 10 000 and for organic liquids may be as high as 1 part in 1000. During the *verification* of glassware the temperature of the liquid used shall be accurately measured, and appropriate corrections applied.

5.3 Cleanness of glass surface The volume contained, or delivered, by a glass vessel depends appreciably on the thoroughness with which the vessel has been cleaned. Any foreign matter left on the wall after cleaning tends to rise with, and contaminate the surface of, the liquid whose volume is to be measured. This leads to a decrease in the surface tension at the liquid surface and a consequent change in shape and volume of the meniscus. In addition, erroneous readings may be obtained with vessels graduated ‘to deliver’ if, as a result of imperfect cleaning, the film of liquid left on the wall is not uniformly distributed.

To ascertain whether a piece of glass apparatus is satisfactorily clean, it shall be observed during filling. A vessel graduated to deliver should preferably be filled from below the meniscus. The rising liquid meniscus shall not change shape, e.g. it shall not crinkle at its edges. After overfilling and withdrawing a little liquid (through the jet if a delivery vessel, and by means of a drawn-down glass tube if a content vessel) the surface of the glass above shall remain uniformly wetted and the meniscus shall not crinkle at its edges but shall merge gradually onto the wall of the vessel. With experience an observer is able to recognize the shape of a contaminated meniscus in relation to its diameter.

When volumetric glassware is being used, as distinct from being verified, chemical contamination can introduce an error even though it has no influence on the accuracy of volume measurement. Therefore, vessels should be thoroughly decontaminated before use, paying special attention to ground glass joints where fitted.

NOTE: Satisfactory methods of cleaning and decontamination are described in Appendix A.

5.4 Setting of meniscus The meniscus, whether water or mercury is used, is said to be set to a line when the edge of the line which is nearer the meniscus forms a horizontal tangent to the convex side of the meniscus. Thus in the case of a water meniscus, the *lowest* point of the meniscus is set to the *upper* edge of the line, whereas with a mercury meniscus the *highest* point of the meniscus is set to the *lower* edge of the line.

Opaque liquids which obscure the lower point of the meniscus may be read on the ‘upper edge’ of the meniscus. This method is less precise than when the setting is made on the *lowest* point of the meniscus. In the case of flasks and pipettes it is necessary to apply a correction for the height of the meniscus. Flasks or pipettes may also be calibrated by setting to the top of the water meniscus.

To make a precise setting of the meniscus, the lighting should be so arranged that the meniscus appears dark and distinct in outline; it should therefore be viewed against a white background and shaded from undesirable illumination. This effect can be achieved by securing a suitable strip of black paper around the vessel, not more than 1 mm below the level of setting for a concave meniscus, nor more than 1 mm above for a convex meniscus. The paper shall have the edge nearest the meniscus cut clean and straight and may be held in place by means of a paper clip as shown in Figure 1. The width (W) of the paper strip shall be at least equal to the diameter of the vessel, at the point where the meniscus is located.

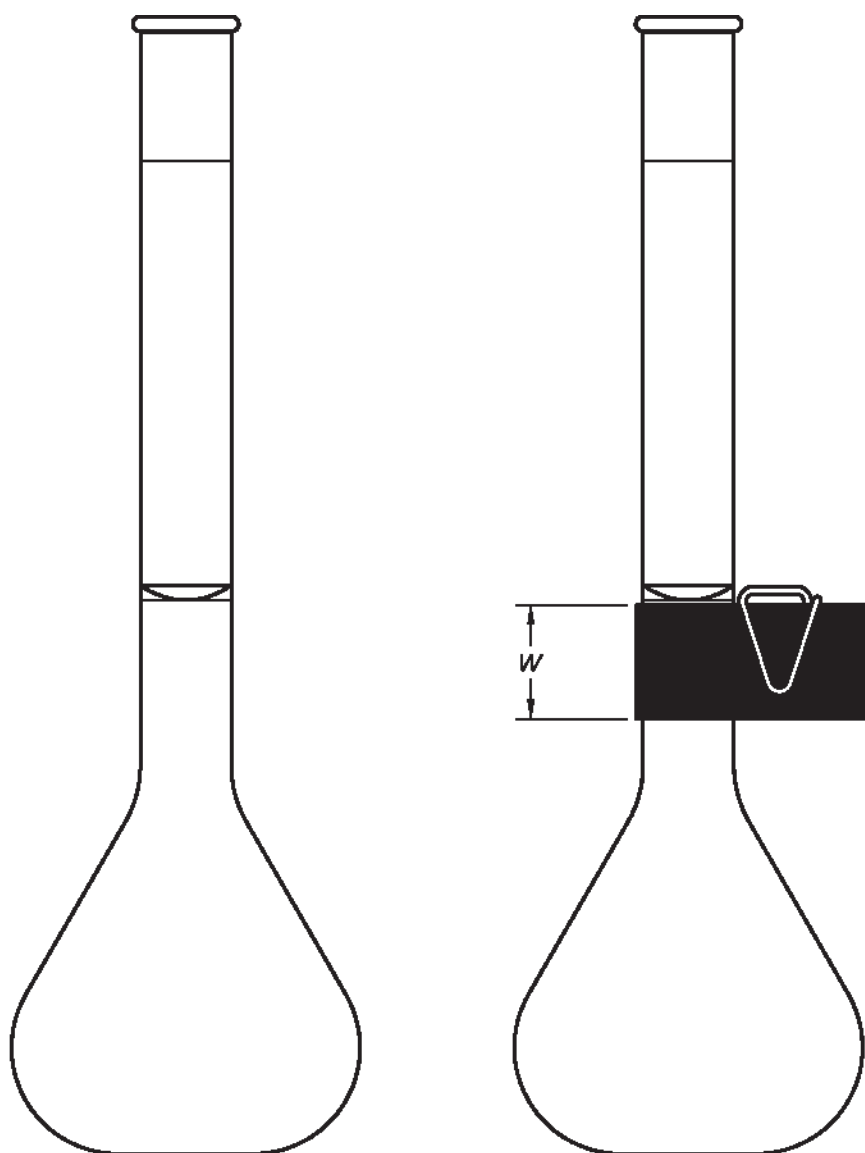


FIGURE 1 USE OF BLACK PAPER FOR SHADING THE MENISCUS

In addition to having good general illumination, the working space should be provided with means for obtaining enhanced indirect back illumination. An arrangement suitable for most purposes is shown in Figure 2(a). If long scales, such as those on burettes, are being read, better illumination is obtained when the fluorescent lamps are mounted vertically (see Figure 2(b)).

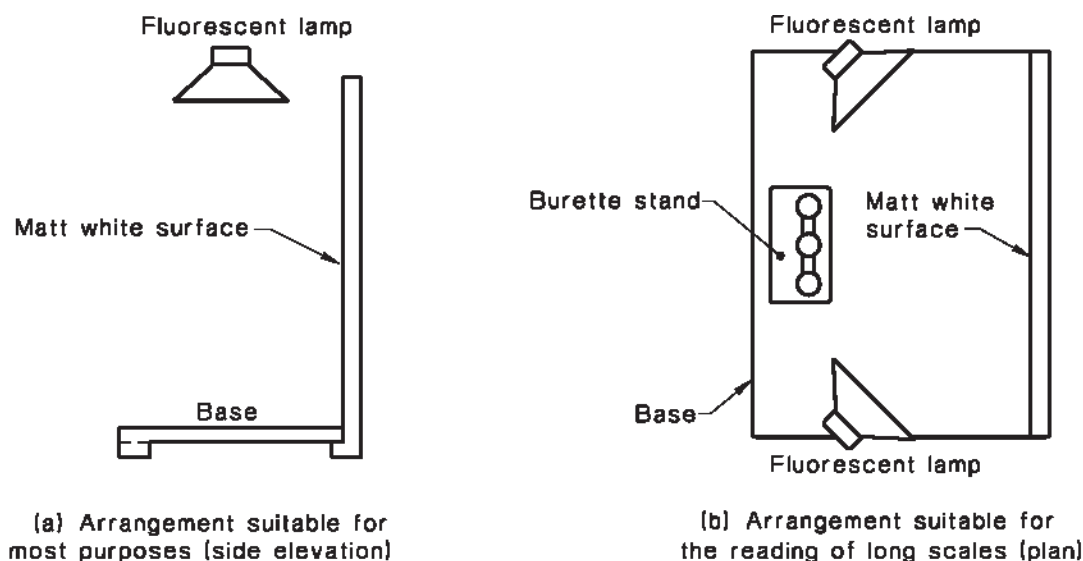


FIGURE 2 RECOMMENDED ARTIFICIAL ILLUMINATION

Parallax error is avoided when the graduation lines are of sufficient length to be seen at the front and back of the vessel simultaneously. When vessels are provided with graduation lines on the front only, parallax error can be made negligible when making a setting on the top edge of the line by using the shading strip referred to above, care being taken that the top edge of the strip is in the 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.

When mercury is being used, a rising meniscus is preferred, but in all cases before reading a mercury meniscus the apparatus shall be lightly tapped, with a pencil or similar object in the vicinity of the line being observed, in order to obtain a stable and well-formed meniscus.

5.5 Time of delivery 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 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, e.g. 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 the legal metrology authority.