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Glass delivery measures - Automatic pipettes

Mesures en verre à délivrer - Pipettes automatiques

OIML D 26 Edition 2010 (E)

ORGANISATION INTERNATIONALE DE MÉTROLOGIE LÉGALE

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Updated references

OIML TC 8 updated the following references in this Publication in 2010:

Chapter	Reference	Document	New or complete reference
p.6, References	**ISO 383**	Laboratory glassware - Interchangeable conical ground joints	**ISO 383:1976**
p.6, References	**ISO 384**	Laboratory glassware - Principles of design and construction of volumetric glassware	**ISO 384:1978**
p.6, References	**ISO 1042**	Laboratory glassware - One-mark volumetric flasks	**ISO 1042:1998**
p.6, References	**ISO 4787**	Laboratory glassware - Volumetric glassware - Methods for use and testing of capacity	**ISO 4787:2010**
p.6, References	**ISO 4788**	Laboratory glassware - Graduated measuring cylinders	**ISO 4788:2005**
p.6, References	**OIML R 4**	Volumetric flasks (one mark) in glass	**OIML R 4:1972**
p.6, References	**OIML R 40**	Standard graduated pipettes for verification officers	**OIML R 40:1981**
p.6, References	**OIML R 43**	Standard graduated glass flasks for verification officers	**OIML R 43:1981**
p.6, References	**OIML R 76-1:1992**	Non automatic weighing instruments	**OIML R 76-1:2006**

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Foreword

The International Organization of Legal Metrology (OIML) is a worldwide, intergovernmental organization whose primary aim is to harmonize the regulations and metrological controls applied by the national metrological services, or related organizations, of its Member States.

The two main categories of OIML publications are:

- **International Recommendations (OIML R)**, which are model regulations that establish the metrological characteristics required of certain measuring instruments and which specify methods and equipment for checking their conformity; the OIML Member States shall implement these Recommendations to the greatest possible extent;
- **International Documents (OIML D)**, which are informative in nature and intended to improve the work of the metrological services.

OIML Draft Recommendations and Documents are developed by technical committees or subcommittees which are formed by the Member States. Certain international and regional institutions also participate on a consultation basis.

Cooperative agreements are established between OIML and certain institutions, such as ISO and IEC, with the objective of avoiding contradictory requirements; consequently, manufacturers and users of measuring instruments, test laboratories, etc. may apply simultaneously OIML publications and those of other institutions.

International Recommendations and International Documents are published in French (F) and English (E) and are subject to periodic revision.

This publication - reference OIML D 26, edition 1999 (E) - was developed by the OIML technical committee TC 8 *Measurement of quantities of fluids*. It was approved for final publication by the International Committee of Legal Metrology in 1995.

OIML publications may be obtained from the Organization's headquarters:

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Glass delivery measures Automatic pipettes

1 Scope and field of application

This International Document relates to automatic pipettes which are generally used as national primary standards of volume. It gives advice on the manufacture and installation of such standards together with their calibration, use and maintenance.

Automatic pipettes are used by national laboratories, services of legal metrology and regional laboratory metrological offices for the calibration of volumetric standards of a lower order and for the verification of volumetric instruments - including volumetric glassware (flasks etc.), a field which is covered by various OIML International Recommendations and ISO Standards (see clause 13 for references to International Standards and Recommendations relating to laboratory glassware).

2 Description of automatic pipettes

Glass delivery measures having a nominal capacity not exceeding 250 mL shall generally conform to the design shown in Figure 1; capacities greater than 250 mL shall generally conform to the design shown in Figure 2.

Note: Alternative fittings to those shown in Figures 1 and 2 may be used, for example, ground glass fittings and nylon or Teflon® stopcocks.

Measures shall be in the form of a cylindrical body with hemispherical ends which merge into tubes at each end forming a datum at the top and joining a stopcock with a delivery jet and filling tube at the bottom. Alternatively, measures having nominal capacities exceeding 250 mL may have an ellipsoidal bulb as shown in Figure 2.

An overflow chamber shall be fitted to the upper tube in a manner which prevents any leakage of water from the chamber. The outlet of the chamber is positioned so that it is below the level of the datum weir, there being an internally flanged air hole near the top of the chamber diametrically opposite the outlet.

The datum weir and the delivery jet shall have a gradual taper towards the ends so that there is no sudden constriction of the orifice. The end shall be ground square with the axis and slightly beveled on the outside. The stopcock shall be of the double oblique bore type generally conforming to ISO 383 and fitted with a rustproof retaining device or two stopcocks which permit consistent filling and emptying. When fully opened to the delivery position the measure shall be emptied of water in a smooth flow, there being no air trapped in the delivery jet.

The rate of leakage of the stopcock, when tested under a 50 cm head of water with the stopcock free from grease (see also clause 11), shall not exceed 0.006 mL per minute.

3 Delivery time

The delivery time shall be taken as being the period from when the outflow of water begins (with the stopcock in the fully open position) until the descending water surface apparently comes to rest in the bore of the delivery jet.

The delivery time shall be within the upper and lower limits specified in Table 1.

4 Inscriptions

The following inscriptions shall be marked permanently and legibly on the measure:

(a) nominal capacity;
(b) EX 20 °C;
(c) an identification number (also to be marked on the handle of the stopcock).

Additionally, the measure may bear the name of the manufacturer or supplier and a delivery time.

5 Maximum permissible errors

The maximum permissible errors (mpe) in the volume of water delivered by the measure at 20 °C after allowing the drainage time shall not exceed those specified in Table 1.

Table 1
Delivery times and maximum permissible errors

Nominal capacity	Delivery time Minimum	Maximum	mpe ±
5 mL	10 s	20 s	0.06 mL
10 mL	15 s	30 s	0.08 mL
20 mL	15 s	30 s	0.12 mL
25 mL	20 s	40 s	0.12 mL
50 mL	30 s	60 s	0.15 mL
100 mL	30 s	60 s	0.20 mL
200 mL	30 s	60 s	0.4 mL
250 mL	50 s	80 s	0.4 mL
500 mL	60 s	100 s	0.5 mL
1 L	60 s	100 s	1 mL
2 L	80 s	140 s	1 mL
2.5 L	80 s	140 s	1.2 mL
5 L	100 s	150 s	2.5 mL
10 L	120 s	180 s	5 mL

Notes to Table 1:

1) These are the preferred sizes. The choice of other capacities may be made in accordance with national legal requirements.

2) The maximum permissible errors for automatic pipettes are greater than those for flasks as described in OIML R 43. This is because although automatic pipettes allow highly repeatable measurements they are difficult to adjust, therefore they should be calibrated and a correction made. Flasks (OIML R 43) can be made to smaller errors because the position of the line is easy to adjust. Flasks are however more variable in use due to operator effects in judging the meniscus to the line.

6 Reference temperature

The temperature at which the measure is intended to deliver a volume equivalent to its nominal capacity is 20 °C.

7 Manufacture and installation

The measures shall be constructed of good quality clear glass and shall be well annealed and substantially free from visible defects. When in use, the measures must be mounted vertically and securely to permit bottom filling and easy valve stopcock access. A typical installation is shown in Figure 3.

A reservoir of distilled or de-ionized/de-mineralized water shall be sited above the installed measures and in the same room for the purpose of providing temperature stability and gravity filling, and arrangements made to measure the temperature of the water by a thermometer incorporated in the supply line to the measure.

8 Nominal capacities

The preferred nominal capacities are those specified in Table 1.

9 Calibration

9.1 Preparation

The measure should be cleaned with an effective cleansing agent and flushed through to ensure that the inner surfaces are free from dirt and grease. This is important to ensure consistent drainage.

The measure and the water to be used should have been allowed to stabilize thermally in a common environment for about ten hours before calibration is attempted.

The measure should then be filled from the stabilized source until the weir overflows with water and emptied to wet all the internal surfaces. Following drainage, the inner surfaces of the measure should be free from water breaks.

The delivery time should fall between the upper and lower limits as specified in Table 1 and can be established at this point. This delivery time must be recorded and used in each subsequent operation.

9.2 Procedure (ISO 4787, Annex B)

After preparation as above, the pipette is operated to deliver a volume of water for weighing and subsequent determination of the delivered volume. A drainage time convention should be used: e.g. allow 15 seconds after the main flow has ceased and collect the drip (if any) residing on the discharge jet by lightly touching on the inner surface of the receiving vessel.

The weighing instrument to be used must be sited reasonably close to the measure so that the weighing of the delivered water can be made as soon as is practicable after delivery. This weighing must take place in the same environment as the measure is located. The temperature of the water should be taken immediately after weighing. This temperature should not differ by more than 0.2 °C from the temperature of the water from the reservoir. It is recommended that at

least five measurements be made in order to determine the repeatability of the measure. Calculations of the delivered volume are made in accordance with ISO 4787.

Note: The weighing instrument used in this procedure shall be of suitable capacity and have an accuracy at least equal to that of a nonautomatic weighing instrument of Class II (see OIML R 76-1).

9.3 Corrections (ISO 4787)

Corrections to the measurement should be made for:

- the density of the water (as affected by temperature);
- the expansion of the measure (if not calibrated at 20 °C);
- the buoyancy difference between water and the mass standards used in the comparison or those used to calibrate the weighing instrument.

9.4 Uncertainties

It should be possible to calibrate measures to an expanded uncertainty (calculated with k = 2) of one third of the appropriate mpe in Table 1. Above one litre capacity this will give an uncertainty of 0.01 %; not more than half of this figure should be caused by random components of uncertainty.

9.5 Frequency

Measures of one litre and above should be re-calibrated at intervals of between 1–5 years depending on their stability.

Measures below one litre should be re-calibrated at intervals of between 2–10 years.

10 Method of use

Automatic pipettes are used as EX (delivery) measures to deliver liquid into IN (contents) measures such as secondary or working standards (volumetric flasks) which are in turn used to verify liquid measuring equipment.

The method of use is similar to the procedure for calibration and the pipette must always be operated at least once before use to prime it, and to eliminate systematic error on the first run caused by the retention of liquid in the delivery jet.

11 Maintenance

Automatic pipettes require very little maintenance. They should be cleaned at intervals especially before use; it may be advantageous to keep them filled with distilled or de-ionized water even when not in use and keep them in the dark to reduce the growth of algae. The stopcock should be lightly greased (when the pipette is *not* in use) and moved from time to time to prevent seizure or sticking.

12 Administrative aspects

Automatic pipettes, being primary standards of volume, should be certified by the national metrological authority (or one approved/accredited by the authority) which has the necessary expertise. The certificate issued by the approving authority should state:

- the nominal volume;
- the serial number;
- the period of validity;
- the method of test including the delivery time, drainage time, number of measurements, and coefficient of expansion.

The statement of results should include:

- the arithmetic mean of the volume delivered at the reference temperature (normally 20 °C);
- the range of the results of the number of measurements made;
- the expanded uncertainty of the mean volume calculated with k = 2.

13 References

ISO 383	Laboratory glassware - Interchangeable conical ground joints.
ISO 384	Laboratory glassware - Principles of design and construction of volumetric glassware.
ISO 1042	Laboratory glassware - One-mark volumetric flasks.
ISO 4787	Laboratory glassware - Volumetric glassware - Methods for use and testing of capacity.
ISO 4788	Laboratory glassware - Graduated measuring cylinders.
OIML R 4	Volumetric flasks (one mark) in glass
OIML R 40	Standard graduated pipettes for verification officers
OIML R 43	Standard graduated glass flasks for verification officers
OIML R 76-1	Nonautomatic weighing instruments

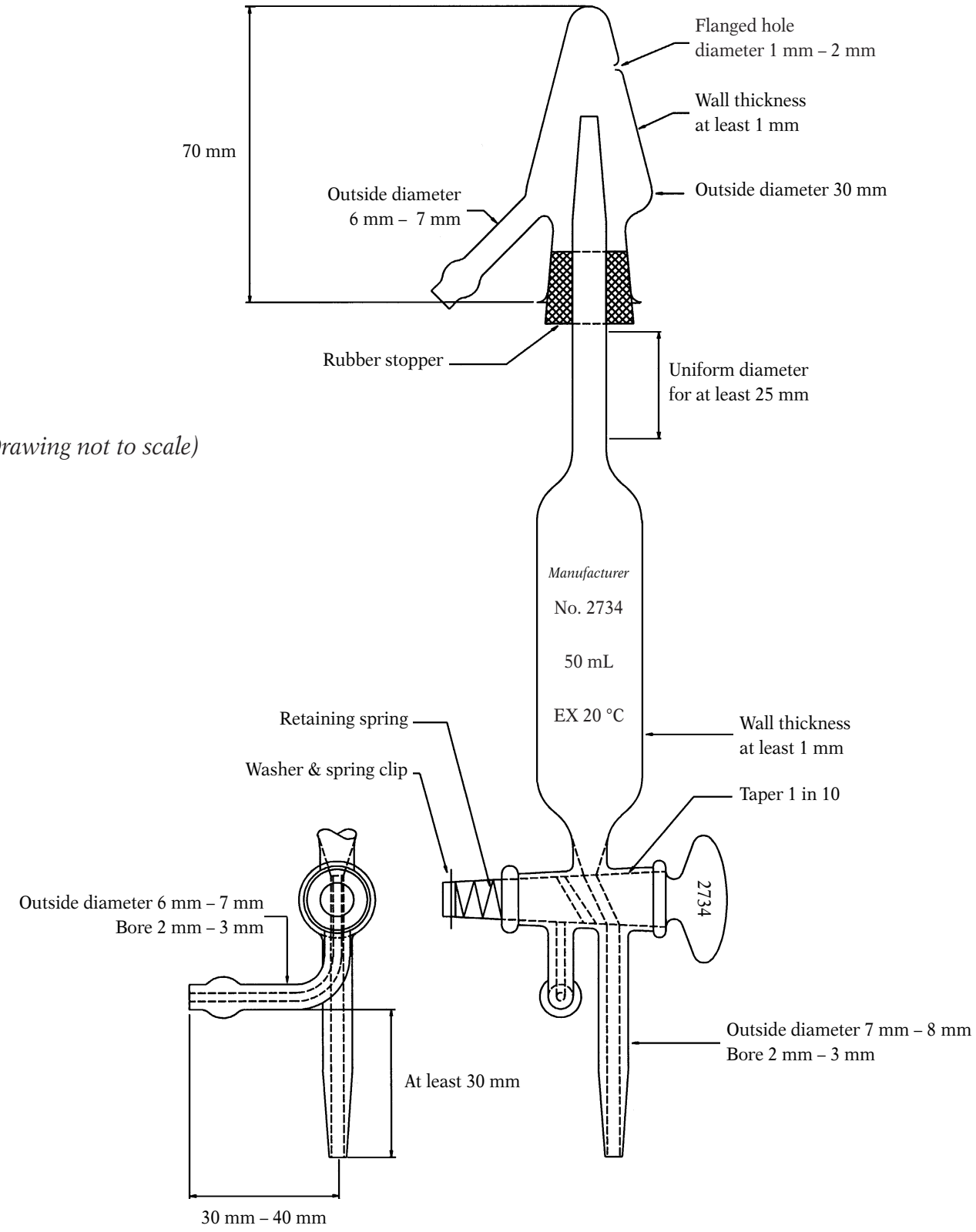

Figure 1 Glass delivery measure, nominal volume not exceeding 250 mL

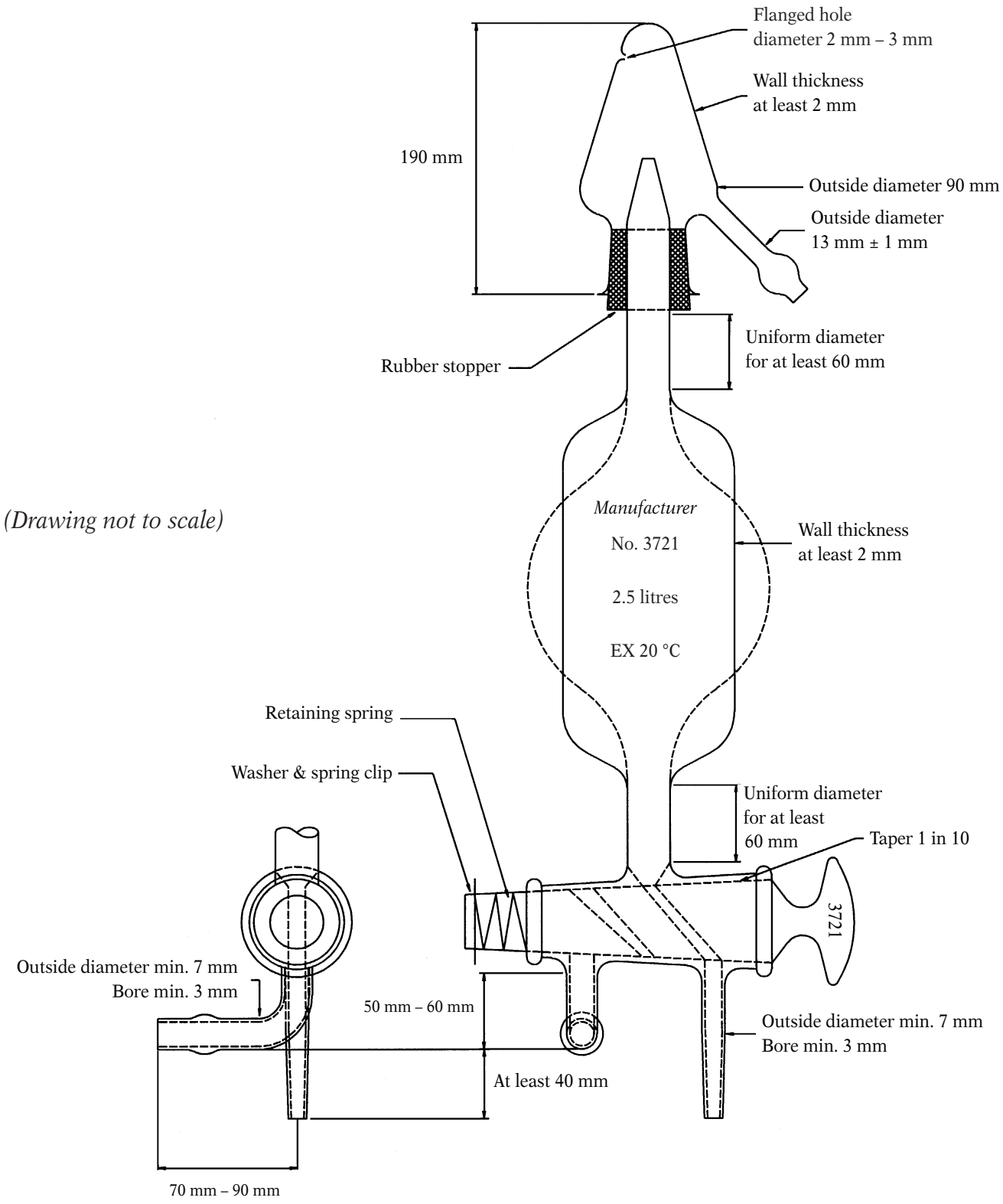

Figure 2 Glass delivery measure, nominal volume greater than 250 mL

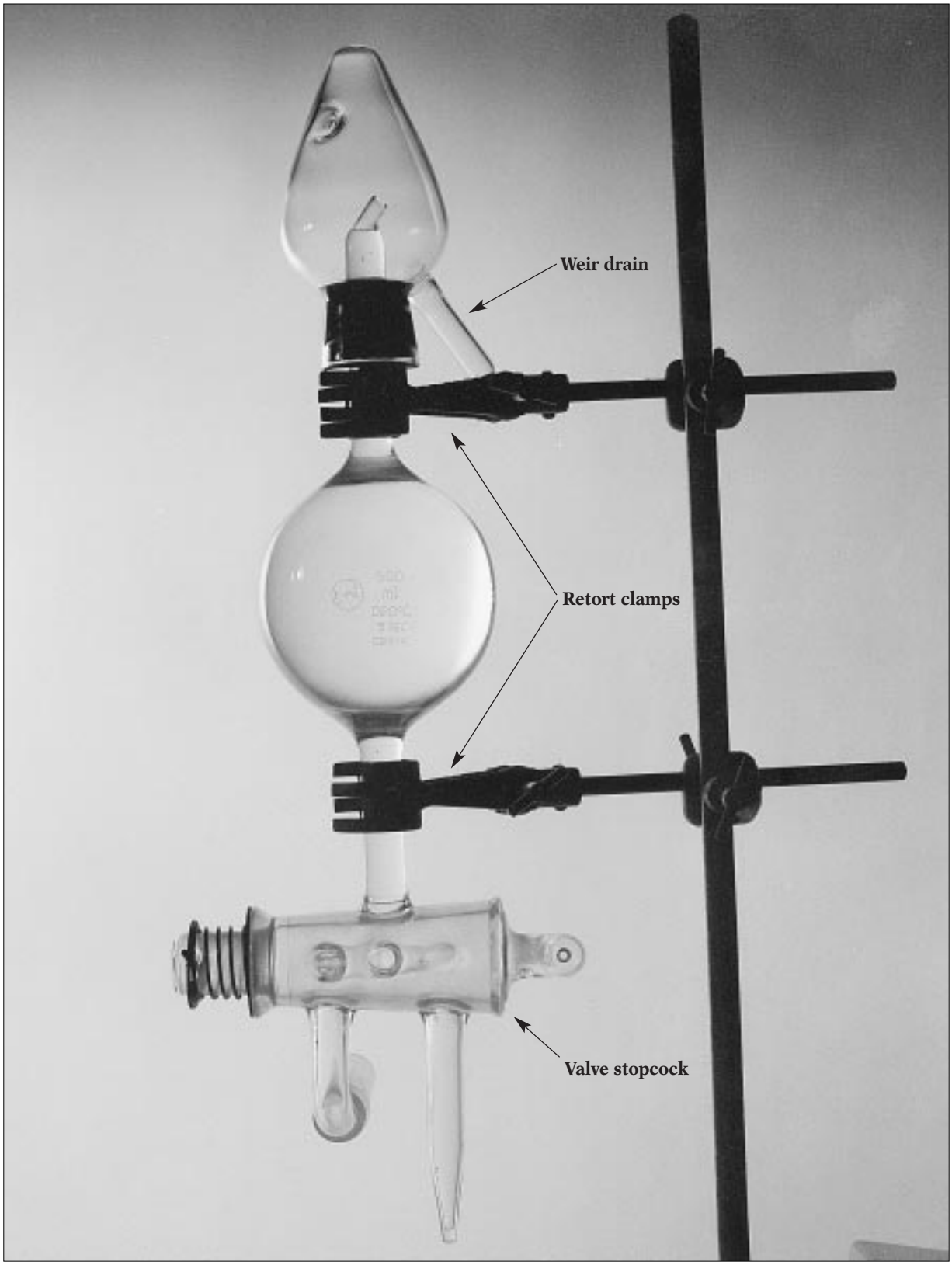

Figure 3 Glass delivery measure

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