

DEPARTMENT OF COMMERCE AND LABOR

CIRCULAR
OF THE
BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 9

TESTING OF GLASS VOLUMETRIC APPARATUS

[5th Edition]

Issued June 1, 1911



WASHINGTON
GOVERNMENT PRINTING OFFICE

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¹ This issue supersedes Bureau Circular No. 9, fourth edition, issued May 15, 1908. Beginning July 1, 1911, the regulations as contained herein will be enforced in the testing of glass volumetric apparatus.

I. STANDARD SPECIFICATIONS FOR GLASS VOLUMETRIC APPARATUS²

The primary purpose of these specifications is to define the requisite qualifications for precision apparatus.

The bureau aims to encourage excellence in quality by cooperating with makers and users of apparatus, and to this end endeavors to assist manufacturers in establishing standards and perfecting methods. In order that users of standardized apparatus may fully benefit by the facilities of the bureau it is necessary for them when purchasing apparatus to be submitted for test to require that the apparatus shall comply with the specifications of the bureau. By admitting for test only apparatus conforming to these standards the work of testing is confined to apparatus whose utility is sufficient to justify the labor expended in the accurate calibration. Certain of the specifications, such as those regarding quality of glass and process of annealing before calibration, are for their fulfillment dependent largely on the integrity of the maker. Only by supporting conscientious makers in giving consideration, first, to quality, and, second, to cost, can users of standardized apparatus secure a high degree of excellence.

1. TYPES OF APPARATUS WHICH WILL BE REGULARLY ADMITTED FOR TEST

Measuring flasks; measuring cylinders, with or without subdivisions; transfer pipettes—i. e., without subdivisions; burettes and measuring pipettes, with partial or complete subdivisions.

2. GENERAL SPECIFICATIONS

(a) **Units of capacity.**—The liter defined as the volume occupied by a quantity of pure water at 4° C having a mass of 1 kilogram, and the one-thousandth part of the liter, called the milliliter or cubic centimeter³ are employed as units of capacity.

(b) **Standard temperature.**—Twenty degrees Centigrade is regarded by the bureau as the standard temperature for glass volumetric apparatus, and an extra charge will be made for testing apparatus graduated for use at other temperatures.

(c) **Material and annealing.**—The material should be of best quality glass, transparent and free from striæ, which adequately resists chemical action, and has small thermal hysteresis. All apparatus should be thoroughly annealed at 400° C for 24 hours and allowed to cool slowly before being graduated.

² A discussion of these specifications, giving in more detail the significance of the various requirements, together with results of experimental work in relation to volumetric apparatus and a description of the methods employed at the bureau in performing the tests, is published in Technical Paper No. 92, reprinted from the Bulletin of the Bureau of Standards, volume 4.

³ The cubic centimeter is not exactly the one-thousandth part of the liter, but the difference is of no consequence in volumetric analyses; 1 milliliter=1.000027 cubic centimeter. (Trav. et Mém. du Bureau International des Poids et Mesures, XIV, 1910.)

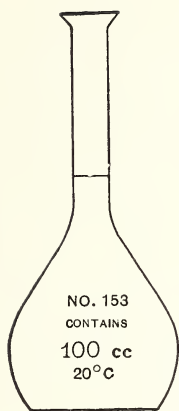


Fig. 1.

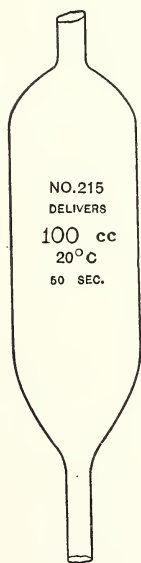


Fig. 2.

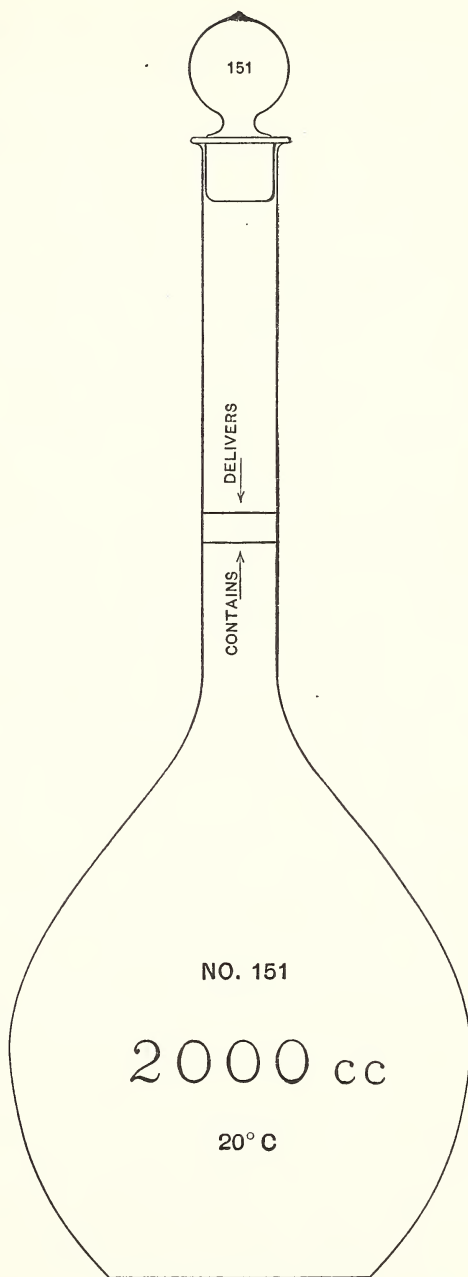


Fig. 3.

(d) **Design and workmanship.**—The cross section must be circular and the shape must permit of complete emptying and drainage.

Instruments having a base or foot must stand solidly on a level surface, and the base must be of such size that the instruments will stand on a plane inclined at 15° . Stoppers and stopcocks must be so ground as to work easily and prevent leakage.

The parts on which graduations are placed must be cylindrical for at least 1 cm on each side of every mark, but elsewhere may be enlarged to secure the desired capacities in convenient lengths.

The graduations should be of uniform width, continuous and finely but distinctly etched, and must be perpendicular to the axis of the apparatus. All graduations must extend at least halfway around, and on subdivided apparatus every tenth mark, and on undivided apparatus all marks must extend completely around the circumference.

The space between two adjacent marks must not be less than 1 millimeter. The spacing of marks on completely subdivided apparatus must show no evident irregularities, and sufficient divisions must be numbered to readily indicate the intended capacity of any interval. Apparatus which is manifestly fragile or otherwise defective in construction will not be accepted.

(e) **Inscriptions.**—Every instrument must bear in permanent legible characters the capacity, the temperature in Centigrade degrees at which it is to be used, the method of use—i. e., whether to contain or to deliver; and on instruments which deliver through an outflow nozzle the time required to empty the total nominal capacity with unrestricted outflow must be likewise indicated.

Every instrument should bear the name or trade-mark of the maker. Every instrument must bear a permanent identification number, and detachable parts, such as stoppers, stopcocks, etc., belonging thereto must bear the same number.

Figures 1, 2, and 3 (two-fifths actual size) illustrate several arrangements of designating marks which are considered suitable.

3. SPECIAL REQUIREMENTS

(a) **Flasks.**—At the capacity mark or marks on a flask the inside diameter should be within the following limits:

Capacity of flask (in cc) up to and including.	25	50	100	200	250	500	1000	2000	3000	4000	5000	6000
Maximum diameter (in mm).	8	10	12	13	15	18	20	25	30	35	40	45
Minimum diameter (in mm).	6	6	8	9	10	12	14	18	20	22	25	30

The neck of a flask must not be contracted above the graduation mark.

The capacity mark on any flask must not be nearer the end of the cylindrical portion of the neck than specified below:

Capacity	Distance from upper end	Distance from lower end
	cm	cm
100 cc or less	3	1
More than 100 cc	6	2

Flasks of 1 liter or more may be graduated both to contain and to deliver, provided the intention of the different marks is clearly indicated. In flasks graduated to more than one capacity, the error in the volume between two consecutive marks shall not exceed one-half of the permissible error in the volume indicated by the first mark.

(b) **Cylinders.**—Only cylinders graduated to contain will be accepted for test.

The inside diameter of cylinders must not be more than one-fifth the graduated length.

(c) **Transfer pipettes.**—Pipettes for delivering a single volume are designated "transfer" pipettes.

The suction tube of each transfer pipette must be at least 16 cm long, and the delivery tube must not be less than 3 cm nor more than 25 cm long.

The inside diameter of any transfer pipette at the capacity mark must not be less than 2 mm and must not exceed the following limits:

Capacity of pipettes (in cc) up to and including	25	50	200
Diameter (in mm)	4	5	6

The outside diameter of the suction and delivery tubes of transfer pipettes, exclusive of the tip, must not be less than 5 mm.

The capacity mark on transfer pipettes must not be more than 6 cm from the bulb.

The outlet of any transfer pipette must be of such size that the free outflow shall last not more than one minute and not less than the following for the respective sizes:

Capacity (in cc) up to and including	5	10	50	100	200
Outflow time (in seconds)	15	20	30	40	50

(d) **Burettes and measuring pipettes.**—Only those burettes emptying through a nozzle permanently attached at the bottom are accepted for test.

Side tubes are not permitted on burettes unless provided with stop-cocks.

So-called "Shellbach" burettes—that is, those having a milk-glass background with a colored center line—will not be accepted for test.

The distance between the extreme graduations must not exceed 70 cm on burettes nor 35 cm on measuring pipettes.

The rate of outflow of burettes and measuring pipettes must be restricted by the size of the tip, and for any graduated interval the time of free outflow must not be more than three minutes nor less than the following for the respective lengths:

Length graduated	Time of outflow	Length graduated	Time of outflow
<i>Centimeters</i>	<i>Seconds</i>	<i>Centimeters</i>	<i>Seconds</i>
15	30	45	80
20	35	50	90
25	40	55	105
30	50	60	120
35	60	65	140
40	70	70	160

The upper end of any measuring pipette must be not less than 10 cm from the uppermost mark and the lower end not less than 4 cm from the lowest mark.

(e) **Burette and pipette tips.**—Burette and pipette tips should be made with a gradual taper of from 2 cm to 3 cm, the taper at the extreme end being slight.

A sudden contraction at the orifice is not permitted, and the tip must be well finished.

In order to facilitate the removal of drops and to avoid splashing when the instrument is vertical, the tip should be bent slightly.

The approved form of tips for burettes, measuring pipettes, and transfer pipettes is shown in figure 4.

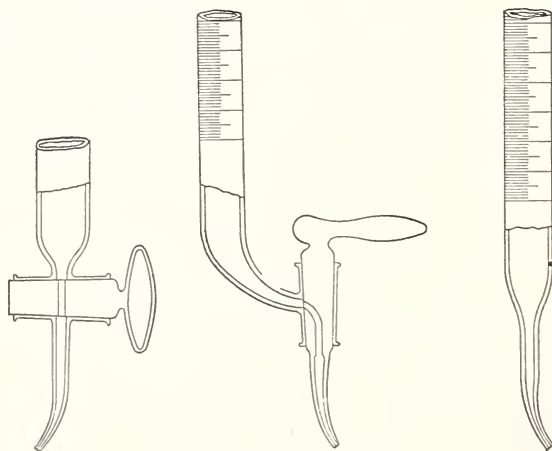


Fig. 4 (two-fifths actual size).

4. SPECIAL RULES FOR MANIPULATION

These rules indicate the essential points in the manipulation of volumetric apparatus which must be observed in order that the conditions necessary to obtain accurate measurements may be reproduced.

(a) **Test liquid.**—Apparatus will be tested with water and the capacity determined will, therefore, be the volume of water contained or delivered by an instrument at its standard temperature.

(b) **Method of reading.**—In all apparatus where the volume is limited by a meniscus the reading or setting is made on the lowest point of the meniscus. In order that the lowest point may be observed it is necessary to place a shade of some dark material immediately below the meniscus, which renders the profile of the meniscus dark and clearly visible against a light background. A convenient device for this purpose is a collar-shaped section of thick black rubber tubing, cut open at one side and of such size as to clasp the tube firmly.

(c) **Cleanliness of apparatus.**—Apparatus must be sufficiently clean to permit uniform wetting of the surface.

(d) **Flasks and cylinders.**—In filling flasks and cylinders the entire interior of the vessel will be wetted, but allowed a sufficient time to drain before reading.

Flasks and cylinders when used to deliver should be emptied by gradually inclining them until when the continuous stream has ceased they are nearly vertical. After half a minute in this position the mouth is brought in contact with the wet surface of the receiving vessel to remove the adhering drop.

(e) **Pipettes and burettes.**—In filling pipettes and burettes excess liquid adhering to the tip should be removed when completing the filling.

In emptying burettes they should be held in a vertical position, and after the continuous unrestricted outflow ceases the tip should be touched with the wet surface of the receiving vessel to complete the emptying. In the case of transfer pipettes they should be held in a vertical position and the outflow should be unrestricted until the surface of the water reaches the upper end of the delivery tube; the tip should then be touched to the wet surface of the receiving vessel and kept in contact with it until the emptying is complete.

Stopcocks, when used, should be completely open during emptying.

Burettes should be filled nearly to the top, and the setting to the zero mark made by slowly emptying.

While under normal usage the measurements ordinarily are from the zero mark, other initial points may be used on burettes of standard form without serious error.

5. TOLERANCES

(a) Flasks.—

Capacity less than and including	Limit of error	
	If to contain	If to deliver
cc	cc	cc
25	0.03	0.05
50	.05	.10
100	.08	.15
200	.10	.20
300	.12	.25
500	.15	.30
1000	.30	.50
2000	.50	1.00
3000	.75	1.50
4000	1.00	2.0
5000	1.2	2.4
6000	1.5	3.0

(b) Transfer pipettes.—

Capacity less than and including	Limit of error
cc	cc
2	0.006
5	.01
10	.02
30	.03
50	.05
100	.08
200	.12

(c) Burettes and measuring pipettes.—

Capacity of total graduated portion less than and including	Limit of error of total or partial capacity	
	Burettes	Measuring pipettes
cc	cc	cc
2	-----	0.01
5	0.01	.02
10	.02	.03
30	.03	.05
50	.05	.08
100	.10	.15

(d) Cylinders.—

Capacity of total graduated portion less than and including	Limit of error of total or partial capacity
cc	cc
30	0.06
50	.10
100	.30
200	.50
500	1.20
1000	2.00
2000	4.00

(e) **Delivery time.**—The delivery time marked on any instrument must be within the limits prescribed in the specifications, and the error permitted in the marked delivery time is as follows:

Delivery time less than and including	Limit of error
Sec	Sec
15	3
20	4
30	6
50	8
100	15
200	20

II. TESTS PERFORMED BY THE BUREAU

1. NATURE OF TESTS

Apparatus submitted for test is first examined as to its conformity with the specifications concerning design and marks, including test of outflow time where this is limited.

Apparatus having subdivisions is examined as to the apparent regularity of spacing.

If the apparatus complies with the specifications in other respects, a test is made of its capacity.

This test may be either to ascertain whether the capacity is correct within the prescribed limits of error or to determine the correction for use in precise measurements.

2. PRECISION STAMP

If the result of examination and test of flasks, cylinders, and transfer pipettes indicates a satisfactory conformity to the specifications, the official precision stamp, consisting of the letters "U. S.," and the year date, surrounded by a circle, is etched as shown below:



3. CERTIFICATES OF CAPACITY

Burettes and measuring pipettes will be tested for at least five intervals, and if found to conform to the specified requirements will be assigned a B. S. certificate number as shown below:

B. S. No. 1763

1911

A certificate will be furnished, giving the volumes delivered by the intervals tested.

When desired, certificates of capacities of flasks, cylinders, and transfer pipettes will also be furnished.

4. SPECIAL TESTS

Apparatus of approved design intended for special purposes, not conforming with the specifications will be received for test only by previous arrangement, when accompanied by complete description of the intended use.

The bureau reserves the right to reject any apparatus on points affecting its accuracy or utility not covered by the regulations.

III. SCHEDULE OF FEES FOR TESTING VOLUMETRIC APPARATUS

SCHEDULE 23

1. FLASKS AND TRANSFER PIPETTES

- (a) For testing and stamping, each capacity tested \$o. 25
 (b) For testing, stamping, and certificate of capacity, each capacity tested 35

2. BURETTES AND MEASURING PIPETTES

- (c) For testing and certifying capacity of five intervals I. 25
 (d) For testing and certifying capacity of additional intervals, each additional interval 25

3. CYLINDERS

- (e) For testing and stamping, each capacity tested 25
 (f) For certificate of capacity at five points I. 25

4. TEMPERATURES OTHER THAN 20° C

- (g) For testing apparatus intended for temperatures other than 20° C between 15° C and 30° C, additional charges for each piece 20

5. UNITS OF CAPACITY OTHER THAN CUBIC CENTIMETERS

- (h) For testing apparatus of indicated capacity other than in cubic centimeters, additional charges for each piece 20

6. DISQUALIFIED APPARATUS

- (i) For preliminary examination of apparatus disqualified for test, charges for each piece . . . 10

IV. DIRECTIONS FOR SUBMITTING APPARATUS FOR TEST

Application for test.—The request for test should be made in writing and should include a complete list of the apparatus and a statement of the nature of the test desired. Unless certificates are requested, flasks, transfer pipettes, and cylinders are examined and tested to determine whether in conformity with specified requirements, and if satisfactory are given the precision stamp.

Unless otherwise requested, burettes and measuring pipettes and subdivided cylinders will be examined and the capacity of five intervals tested. If in satisfactory conformity with the requirements, the results of the test are certified. If more than five intervals are to be tested, the request must so state.

Patrons should always examine apparatus carefully before submitting it for test, to ascertain if it complies with specifications, thus avoiding the loss of transportation on apparatus not entitled to verification.

Purchasers of apparatus to be submitted to the bureau for test should so specify to the dealer in order to avoid unnecessary delays and misunderstandings.

Identification marks.—Instruments and the packages in which they are shipped should both be plainly marked to facilitate identification, preferably with the name of the manufacturer or shipper, and a special reference

number should be given to each article, which should be referred to in the correspondence concerning the test. After receipt at the bureau the B. S. test number should also be used.

Shipping directions.—Instruments should be securely packed in cases or packages which may be used in returning them to the owner. In all cases, transportation charges are payable by the party desiring the test, and should be prepaid. Apparatus must be accompanied by an itemized shipping invoice.

Breakage.—No risk of breakage will be assumed by the bureau. All possible care will be taken in handling the apparatus submitted for test, but a certain amount of breakage is unavoidable and must be borne by the owner.

Address.—Articles should be addressed, "Bureau of Standards, Department of Commerce and Labor, Washington, D. C.;" delays incident to other forms of addresses will thus be avoided. Articles delivered in person or by messenger should be left at the shipping office of the bureau, and should be accompanied by a written request for the verification.

Remittances.—Fees should be remitted by money order drawn to the order of the "Bureau of Standards," and should be sent with the request for test whenever practicable. Delays in forwarding fees involve corresponding delays in the return of articles tested, as the articles are held until the fees due thereon have been paid.

All communications should be addressed, "Bureau of Standards, Department of Commerce and Labor, Washington, D. C."

S. W. STRATTON,
Director.

Approved:
BENJ. S. CABLE,
Acting Secretary.



