

DEPARTMENT OF COMMERCE

CIRCULAR
OF THE
BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 9

**TESTING OF GLASS VOLUMETRIC
APPARATUS**

[8th Edition]

Issued March 31, 1916



WASHINGTON
GOVERNMENT PRINTING OFFICE

1916

CIRCULARS

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TESTING OF GLASS VOLUMETRIC APPARATUS¹

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¹This issue supersedes Circular No. 9, seventh edition, issued Oct. 1, 1914. Beginning July 1, 1916, 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 and capacity pipettes, without subdivisions; burettes and measuring pipettes, with partial or complete subdivisions; specific gravity flasks and Babcock test bottles.

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 centimeter**³ are employed as units of capacity.

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 Scientific Papers No. 92, reprinted from the Bulletin of the Bureau of Standards, vol. 4. Convenient tables for use in testing volumetric apparatus are published in Circular No. 19 of this Bureau.

³ The cubic centimeter is not exactly the one-thousandth part of the liter, but the difference is of no consequence in volumetric analysis; 1 *milliliter* = 1.000027 cubic centimeters. (Trav. et Mém. du Bureau International des Poids et Mesures, XIV, 1910.) In all volumetric work the unit of volume employed is the *milliliter* and not the cubic centimeter, but in this country it is often designated by the letters "cc." This designation has been used in former editions of this circular, but beginning with this issue the more correct designation "ml" will be employed. For the present, apparatus inscribed "cc" will not be rejected for that cause alone, but it is expected that manufacturers will use the designation "ml" exclusively as soon as practicable.

(b) **Standard Temperature.**—Twenty degrees centigrade is regarded by the Bureau as the standard temperature for glass volumetric apparatus, and an extra charge is 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, should adequately resist chemical action, and have small thermal hysteresis. All apparatus should be thoroughly annealed at 400° C for 24 hours and allowed to cool slowly before being graduated.

(d) **Design and Workmanship.**—The cross section must be circular and the shape must permit 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 15° to the horizontal.

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 and parallel to the base 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 clear space between two adjacent marks must not be less than 1 mm. 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.

Two scales will not be permitted on the same piece of apparatus except in the case of the special double-scale cylindrical graduate herewith described. For example, apparatus should not be graduated in both liquid ounces and milliliters. In the case of two units, one of which is an exact multiple of the other, such, for example, as drams and liquid ounces, there is no objection to having the 8-dram, 16-dram, etc., marked, respectively, 1 liquid ounce, 2 liquid ounces, etc., provided that the two series of numbers are placed on opposite sides of the apparatus and the value of each is suitably indicated.

(e) **Inscriptions.**—Every instrument must bear in permanent legible characters the capacity, the temperature 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.

Figs. 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 must be within the following limits:

Diameter (in millimeters)	Capacity of flask (in milliliters) up to and including—											
	25	50	100	200	250	500	1000	2000	3000	4000	5000	6000
Maximum.....	8	10	12	13	15	18	20	25	30	35	40	45
Minimum.....	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 ml or less.....	3	1
More than 100 ml.....	6	2

Flasks may be graduated both *to contain* and *to deliver*, provided the intention of the different marks is clearly indicated and provided the distance between two adjacent marks is not less than 1 mm. 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.**—Cylinders graduated either to contain or to deliver will be accepted for test.

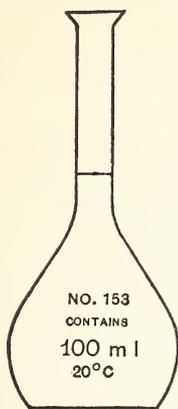


Fig. 1

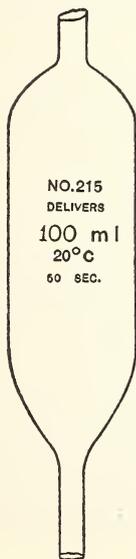


Fig. 2

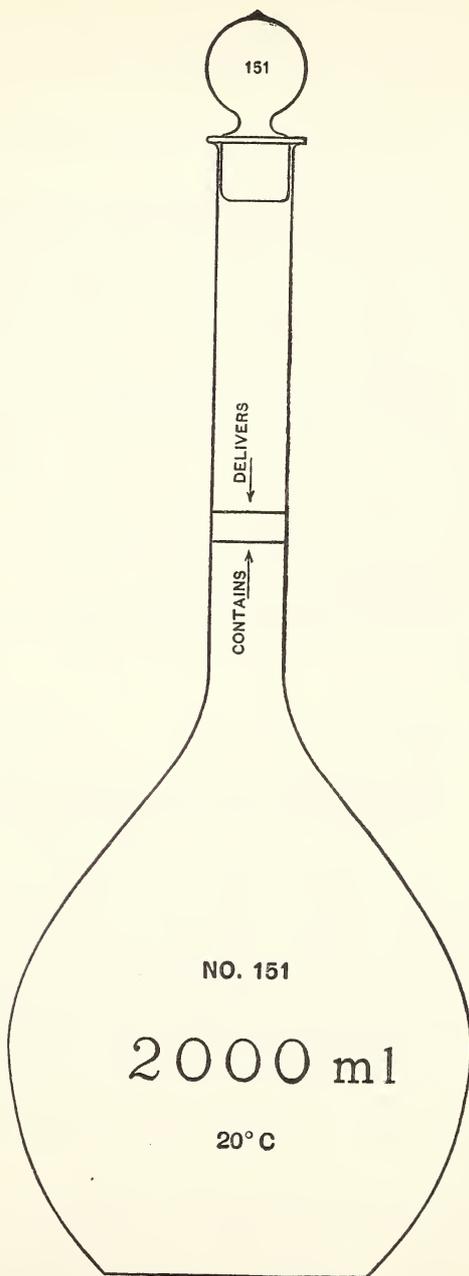


Fig. 3

FIGS. 1-3.—Examples of inscriptions suitable for volumetric apparatus

A double scale graduate, i. e., with a scale reading both up and down the length of the graduate, will not be accepted for test, as it is obvious that the same graduate can not be correct both to contain and to deliver.

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

The relation of the height to the diameter must be such that the graduation marks are not less than 1 mm apart, and also that the graduated height is at least five times the inside diameter. In the case of the 10-ml cylinder subdivided to 0.1 ml and the 25-ml and 30-ml cylinders subdivided to 0.2 ml, it will be found necessary to make the graduated height considerably more than five times the inside diameter in order to give a separation of 1 mm to the graduation marks. On the larger cylinders, subdivided as indicated, the graduation marks will be sufficiently separated if the graduated height is five times the inside diameter.

The ordinary cylindrical graduates as usually subdivided are shown in Fig. 4.

The smallest diameter of the necks of cylinders with stoppers shall not be less than specified below:

Capacity (in milliliters).....	5	10	25	50	100	200	500	1000	2000
Minimum diameter of neck (in millimeters).....	6	7	8	9	10	12	15	18	20

Standard Cylindrical Graduates in United States Customary Units

In view of the difficulty experienced by weights and measures officials in securing satisfactory graduates, it has been thought advisable to prepare detailed specifications for a complete set of cylindrical graduates in United States customary units.

The specifications here presented are intended to cover those graduates most often needed by weights and measures officials in the discharge of their duties, and while for certain special purposes they may not be found satisfactory it is believed that most of the ordinary needs of weights and measures officials will be met by them.

The graduates most often required are the following: One dram (60 minims), subdivided to 2 minims; 2 drams (120 minims), subdivided to 5 minims; 1 liquid ounce, subdivided to $\frac{1}{4}$ dram (15 minims); 2 liquid ounce, subdivided to $\frac{1}{16}$ ounce; 4 liquid ounce, subdivided to $\frac{1}{16}$ or $\frac{1}{8}$ ounce; 8 liquid ounce, subdivided to $\frac{1}{8}$ ounce; 16 liquid ounce, subdivided to $\frac{1}{4}$ ounce; 32 liquid

STANDARD CYLINDRICAL GRADUATES
METRIC UNITS

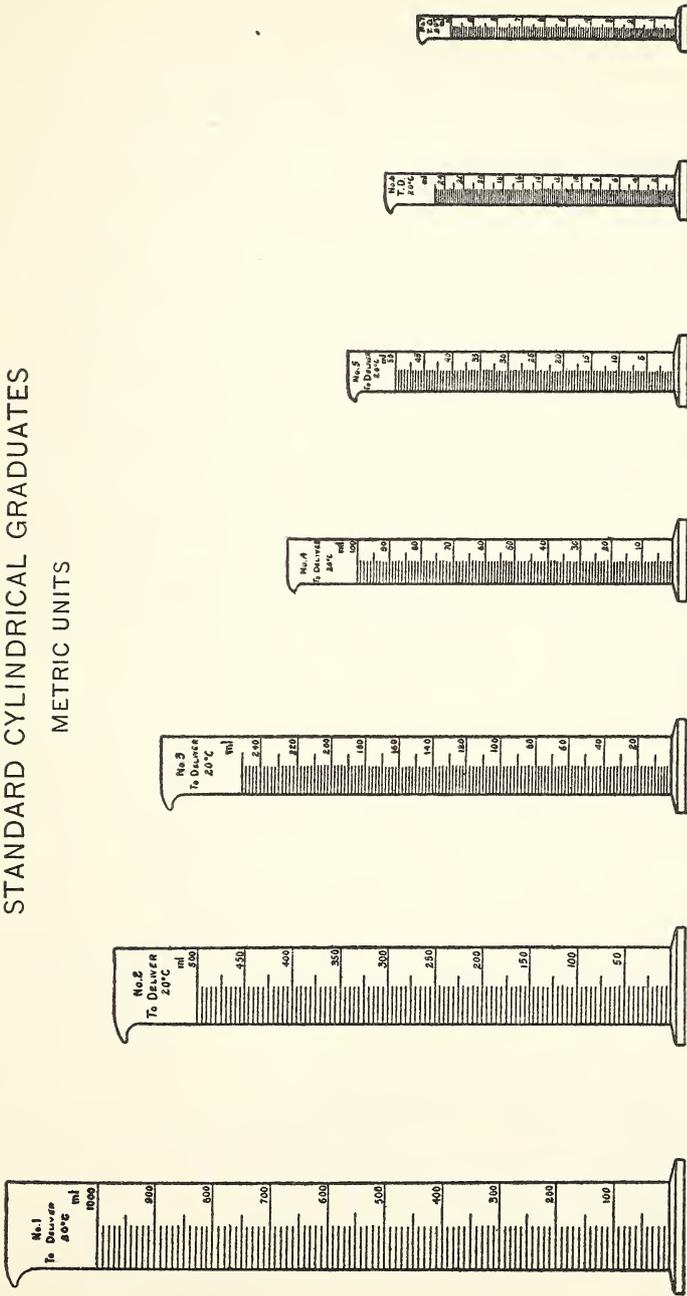


FIG. 4

ounce, subdivided to $\frac{1}{2}$ ounce; 3 cubic inch, subdivided to $\frac{1}{10}$ cubic inch; 10 cubic inch, subdivided to $\frac{2}{10}$ cubic inch; and 35 cubic inch, subdivided to $\frac{1}{2}$ cubic inch.

In the past this last-named graduate has sometimes been further subdivided to $\frac{1}{4}$, $\frac{1}{2}$, and 1 pint, either liquid or dry, and sometimes both. In that case the number and closeness of the graduations are likely to lead to confusion and error in the use of the graduate. Its use should be avoided and in the future it will not be accepted by the Bureau for test. Since it is considered important by most inspectors to have a single graduate which may be used for testing both liquid and dry measures, it has been thought advisable to provide for such a graduate. For that purpose a cylinder may be made up with divisions of only the $\frac{1}{4}$, $\frac{1}{2}$, and 1 liquid pint, and $\frac{1}{4}$, $\frac{1}{2}$, and 1 dry pint, leaving the remainder of the cylinder entirely blank, except for the necessary inscription. The major divisions of the cylinder will then not be obscured and confused by the small subdivisions. Such a cylinder may be used to test both liquid and dry measures by using with it the usual auxiliary tolerance graduate. For this purpose the 2-ounce or 4-ounce graduate, subdivided to $\frac{1}{2}$ dram, and the 3 cubic inch subdivided to $\frac{1}{10}$ cubic inch will be found convenient.

It might be said in this connection that when a large graduate is used in the test of measures by pouring from the graduate into the measure a small auxiliary graduate should always be used for determining the excess or deficiency of the measure, as the subdivisions of the large graduate are not sufficiently fine for the purpose. Also the first few divisions at the base of a graduate are difficult to read and are not always accurate.

BASIS OF GRADUATION.—All graduates herein described and for which specifications are herein given should be graduated in accordance with the following relations:

8 liquid drams = 1 liquid ounce.

32 liquid ounces = 1 liquid quart.

4 liquid quarts = 1 U. S. liquid gallon = 231 cubic inches.

1 liquid quart = 57.75 cubic inches.

1 dry quart = 67.20 cubic inches.

For the purpose of bridging over to the metric system the relation is

1 liquid ounce = 29.5729 milliliters.

Graduates may be calibrated for use either to contain or to deliver, but on each graduate the method of use must be stated. (See Inscription.)

Graduates that are to be used dry to receive liquids and measure them in the graduate should be calibrated to contain.

Graduates that are to be used to pour water from the graduate into other measures, and those which are to be used wet to contain water from other measures should be calibrated to deliver. For example, a graduate that is to be used in testing milk bottles, either by pouring water from the graduate into the bottles or from the bottles into the graduate without drying the graduate between bottles, should be calibrated to deliver. After having been wet the graduate will, on successive fillings and emptyings, deliver the same quantity that is poured into it.

In ordering graduates the purchaser should consider the use to which they are to be put and should specify accordingly whether they should be calibrated to contain or to deliver.

SPECIFICATIONS.—The same general specifications apply to these graduates as to other volumetric apparatus described in this circular.

The relation of the height to the diameter of the cylinder should be such that the highest graduation will come within the following limits:

Total nominal capacity	Distance of highest graduation from top	
	Minimum	Maximum
	cm	cm
60 minims	2	4
120 minims	2	4
1 liquid ounce	2	5
2 liquid ounces	3	5
4 liquid ounces	3	6
8 liquid ounces	4	8
16 liquid ounces	5	10
32 liquid ounces	5	10
3 cubic inches	3	5
10 cubic inches	4	8
35 cubic inches	5	10

The graduations should be of uniform width, continuous, and finely but distinctly etched or engraved, and must be perpendicular to the axis of the cylinder.

All graduations must extend at least halfway around and at least every tenth graduation entirely around the cylinder.

The space between two adjacent graduations must never be less than 1 mm, and in general should be from 2 to 5 mm, depending upon the size of the graduate.

Inscription.—Each graduate must bear in permanent and legible characters the capacity, the temperature at which it is to be used, the method of use—that is, whether to contain or to deliver—and an identification number. It is desirable, also, that the name of the manufacturer be placed on each graduate.

The numbers indicating the capacity of the graduate at its different points should be placed immediately above the marks to which they refer.

Subdivisions and Length of Lines.—The 60-minim graduate, subdivided to 2 minims, should have each 2-minim graduation extend at least halfway around the cylinder. Each 10-minim graduation should extend entirely around and should be numbered.

The 120-minim graduate, subdivided to 5 minims, should have each 5-minim graduation extend at least halfway around, each 10 minims about three-fifths around, and each 20 minims entirely around. Each 20-minim graduation should be numbered.

The 1 liquid-ounce (8 drams) graduate, subdivided to $\frac{1}{4}$ dram (15 minims), should have each $\frac{1}{4}$ -dram graduation extend at least halfway around, each $\frac{1}{2}$ dram about three-fifths around, and each dram entirely around the cylinder. Each dram graduation should be numbered.

The 2-ounce graduate, subdivided to $\frac{1}{16}$ ounce, should have each $\frac{1}{16}$ -ounce graduation extend at least halfway around, each $\frac{1}{8}$ -ounce graduation about three-fifths around, and each $\frac{1}{4}$ -ounce graduation entirely around the cylinder. Each $\frac{1}{2}$ -ounce graduation should be numbered.

The 4-ounce graduate, if subdivided to $\frac{1}{16}$ ounce, should have the same arrangement and length of graduations as the 2-ounce, except that only each $\frac{1}{2}$ -ounce graduation should extend entirely around and only each ounce should be numbered.

If subdivided only to $\frac{1}{8}$ ounce, then each $\frac{1}{8}$ -ounce graduation should extend at least halfway around, and each $\frac{1}{2}$ -ounce and 1-ounce entirely around, and each 1-ounce graduation should be numbered.

The 8-ounce graduate, subdivided to $\frac{1}{8}$ ounce, should have each $\frac{1}{8}$ ounce extend at least halfway around, each $\frac{1}{2}$ -ounce graduation about three-fifths around, and each 1-ounce graduation entirely around and numbered.

STANDARD CYLINDRICAL GRADUATES
U. S. LIQUID OUNCES

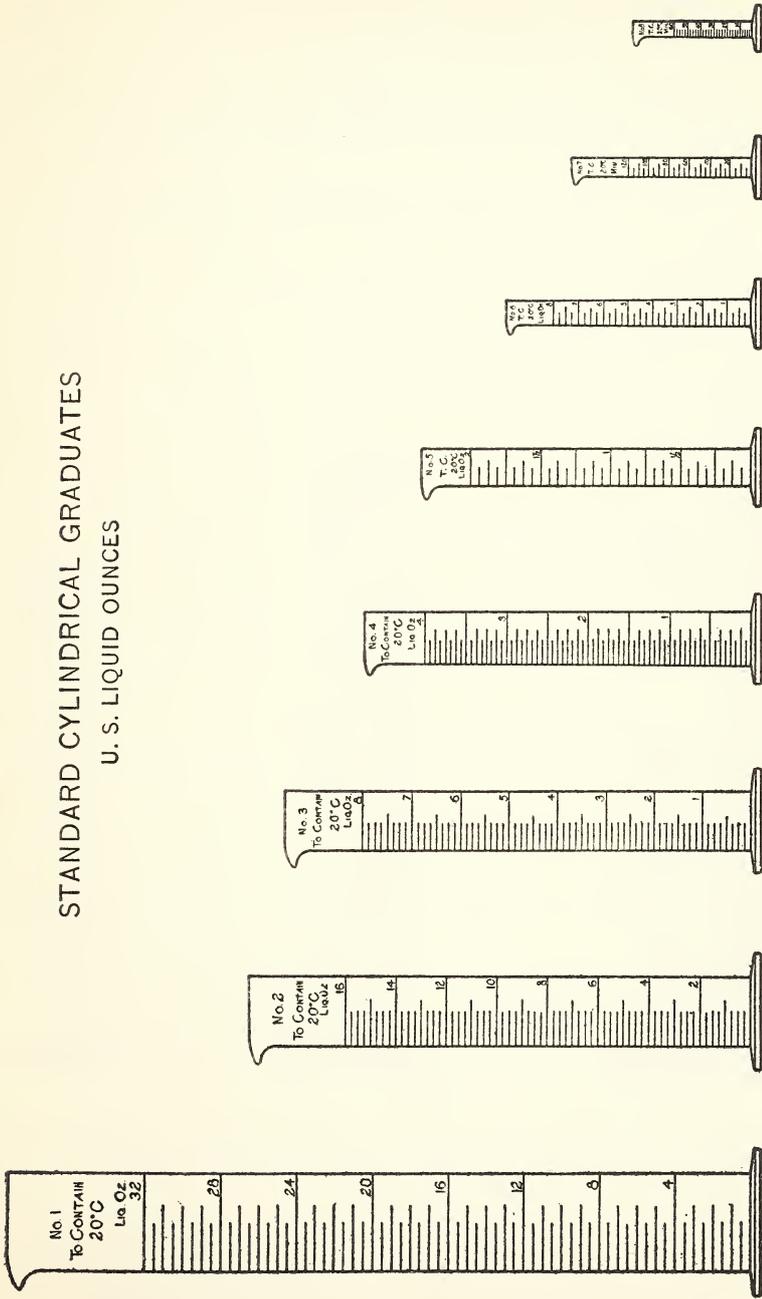


FIG. 5

The 16-ounce graduate, subdivided to $\frac{1}{4}$ ounce, should have each $\frac{1}{4}$ ounce extend at least halfway around, each 1-ounce about three-fifths around, and each 2-ounce entirely around and numbered.

The 32-ounce graduate, subdivided to $\frac{1}{2}$ ounce, should have each $\frac{1}{2}$ -ounce extend at least halfway around, each 1-ounce about

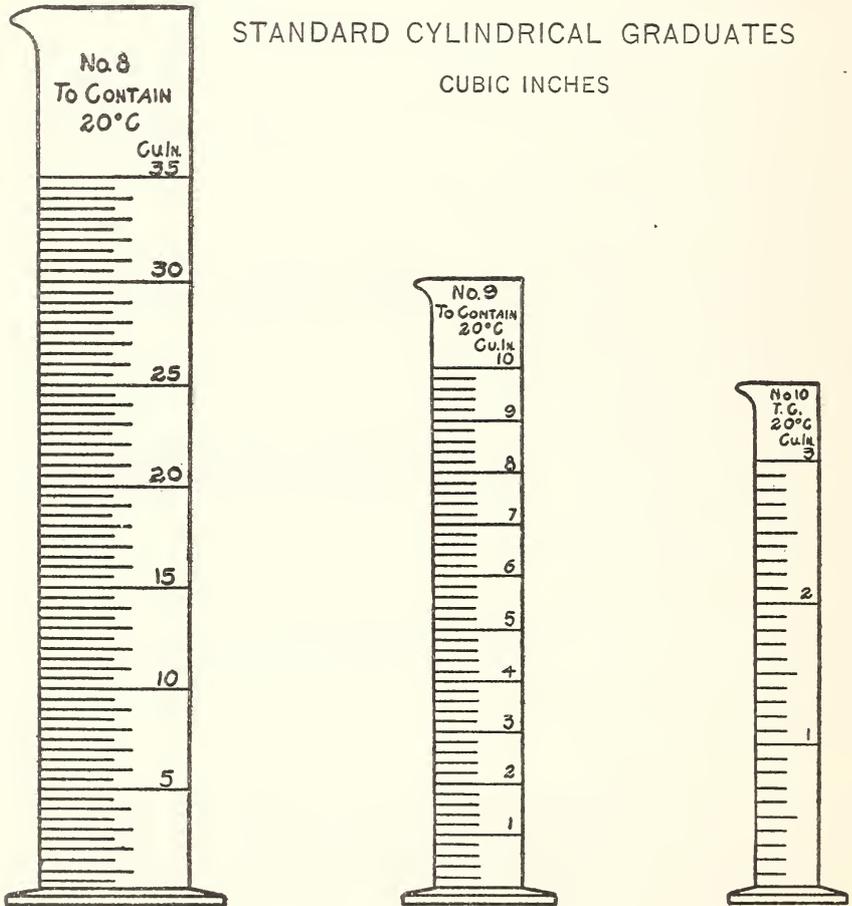


FIG. 6

three-fifths around, and each 4-ounce entirely around and numbered.

The 3 cubic inch graduate, subdivided to $\frac{1}{10}$ cubic inch, should have each $\frac{1}{10}$ cubic inch graduation extend at least halfway around, each $\frac{5}{10}$ cubic inch graduation three-fifths around, and each 1 cubic inch graduation entirely around and numbered.

The 10 cubic inch graduate, subdivided to $\frac{2}{10}$ cubic inch, should have each $\frac{2}{10}$ cubic inch graduation extend at least halfway around, and each 1 cubic inch graduation entirely around and numbered.

The 35 cubic inch graduate, subdivided to $\frac{1}{2}$ cubic inch, should have each $\frac{1}{2}$ cubic inch extend at least halfway around, each 1 cubic inch about three-fifths around, and each 5 cubic inch entirely around and numbered.

The tolerances to be allowed on cylinders are shown under "Tolerances," page 25.

The special double-scale graduate with subdivisions of $\frac{1}{4}$, $\frac{1}{2}$, and 1 liquid pint, and $\frac{1}{4}$, $\frac{1}{2}$, and 1 dry pint should have all graduations extend entirely around and be numbered, the numbers for the liquid pint and for the dry pint being placed on opposite sides of the cylinder.

(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 milliliters) 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 1 minute and not less than the following for the respective sizes:

Capacity (in milliliters) 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 stopcocks.

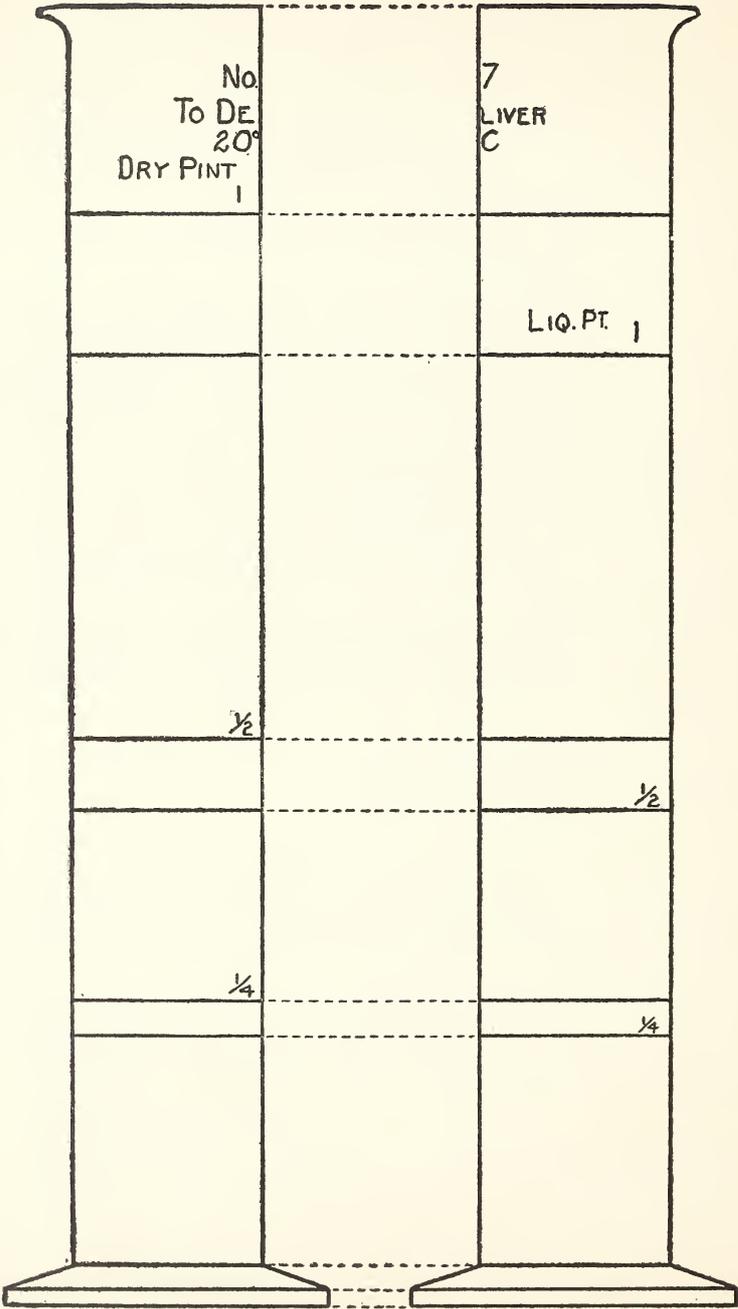


FIG. 7.—Special double-scale graduate, U. S. liquid pint and dry pint

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 3 minutes nor less than the following for the respective lengths:

Length graduated	Time of outflow not less than—	Length graduated	Time of outflow not less than—
cm	sec	cm	sec
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.

On 50-ml and 100-ml burettes the highest graduation mark should not be less than 3 cm nor more than 10 cm from the upper end of the burette. On burettes having a capacity of 25 ml or less this distance should not be less than 2 cm nor more than 6 cm.

(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 Fig. 8.

4. SPECIAL APPARATUS

(a) **Giles Flasks.**—As considerable difficulty has been experienced by manufacturers in supplying Giles flasks to meet the foregoing specifications, there are given below proper dimensions for Giles flasks of various common sizes.

At the capacity mark the inside diameter of the neck shall be within the following limits:

Capacity (in milliliters)	Inside diameter of neck	
	Minimum	Maximum
25-27.5.....	6	9
50-55.....	6	11
100-110.....	8	13
200-220.....	9	15
500-550.....	12	18
1000-1100.....	14	20
2000-2200.....	18	25

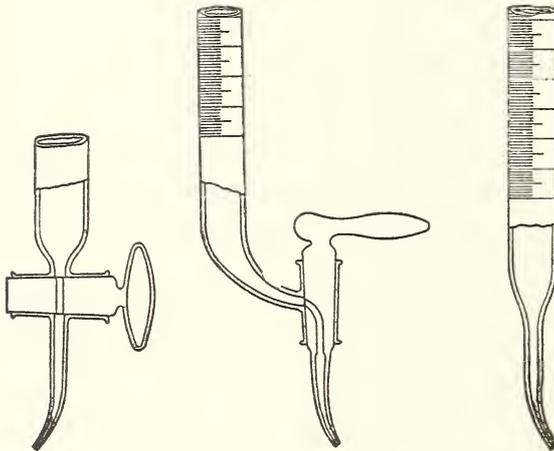


FIG. 8 (two-fifths actual size)

The neck shall be cylindrical on each side of every graduation mark for at least the following distances:

Capacity (in milliliters)	Above upper graduation mark	Between upper graduation mark and bulb	Between lower graduation mark and bulb	Below lower graduation mark
25-27.5.....	3	0.8	1	1
50-55.....	3	.8	1	1
100-110.....	3	.8	1	1
200-220.....	4	.8	1.5	2
500-550.....	4	1.0	1.5	2
1000-1100.....	5	1.0	1.5	2
2000-2200.....	5	1.0	1.5	2

On account of the bulb in the neck of a Giles flask it is more unstable or top-heavy than a flask of the ordinary type. For that reason it has been thought advisable to allow a somewhat shorter minimum length for the cylindrical portion of the neck on each side of the graduation mark than is allowed on ordinary flasks. In other respects the same general specifications apply to the Giles flasks as to other volumetric apparatus.

(b) **Specific-Gravity Flasks.**⁴—**MATERIAL AND ANNEALING.**—The material from which the flasks are made shall be glass of the best quality, transparent, and free from striæ. It shall adequately resist chemical action and have small thermal hysteresis. The flasks shall be thoroughly annealed at 400° C to 500° C for 24 hours and allowed to cool slowly before being graduated. They shall be of sufficient thickness to insure reasonable resistance to breakage.

DESIGN.—The cross section of the flask shall be circular and the shape and dimensions shall conform to the diagram shown in Fig. 9. This design is intended to insure complete drainage of the flask on emptying and stability of standing on a level surface, as well as accuracy and precision of reading. The neck of the flask shall be cylindrical for at least 1 cm above and below every graduation mark. There shall be a space of at least 1 cm between the highest graduation mark and the lowest point of the grinding for the glass stopper.

CAPACITY.—The flask should contain approximately 250 ml when filled to the zero graduation mark.

GRADUATIONS.—The neck shall be graduated from 0 to 1 ml and from 18 ml to 24 ml into 0.1-ml divisions. The 0.1-ml graduations should be continued two below the 0 and two above the 1 ml graduation. The graduations shall be of uniform width, finely but distinctly etched, and shall be perpendicular to the axis of the flask. The 0.1-ml graduations shall be at least 1 mm apart. This will require an internal diameter of the neck not greater than 11.3 mm. The 1-ml graduations shall extend completely around the neck of the flask and shall be numbered to indicate the capacity. The 0.1-ml graduations shall extend at least halfway around the neck and the 0.5-ml graduations shall have a length about midway between the other two. The graduation marks shall have no apparent irregularities of spacing.

⁴ From Circular No. 33 of the Bureau of Standards. In Circular No. 33 the abbreviation "cc" is used instead of "ml" for designating the unit of volume.

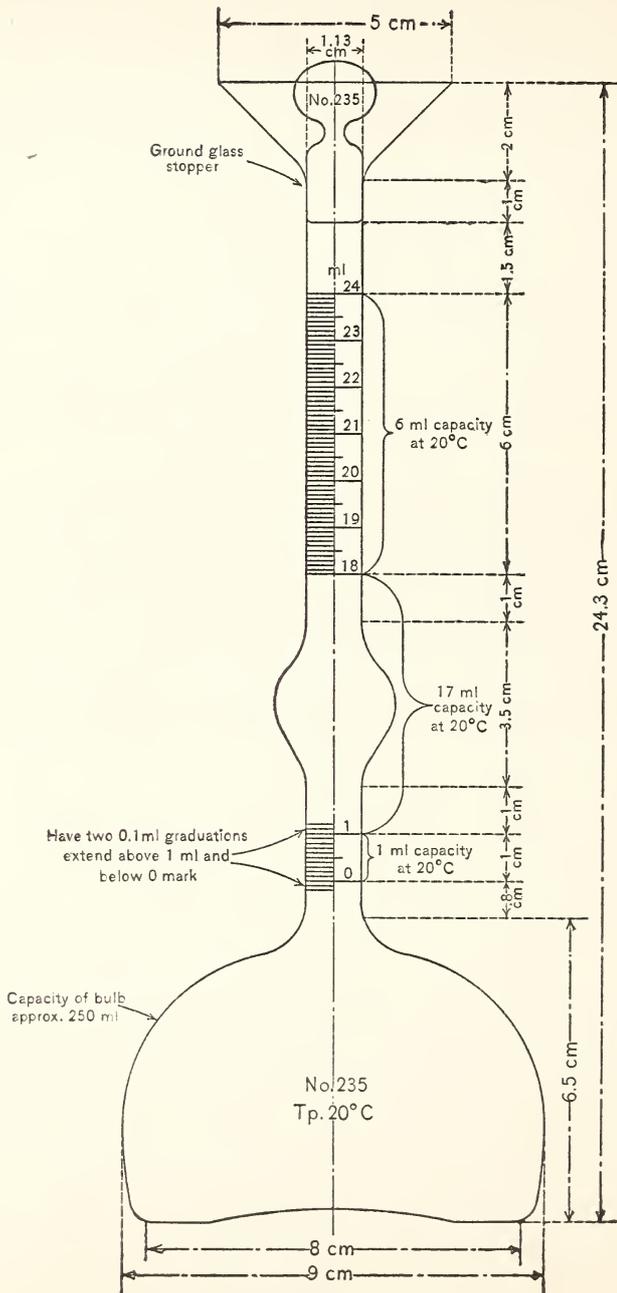


FIG. 9.—Specific gravity flask

STANDARD TEMPERATURE.—The flasks shall be standard at 20° C. The indicated specific gravities will then be at 20° referred to water at 4° as unity—that is, density at 20° in grams per milliliter.

INSCRIPTIONS.—Each flask shall bear a permanent identification number and the stopper shall bear the same number. The standard temperature shall be indicated and the unit of capacity shall be shown by the letters “ml” placed above the highest graduation mark.

TOLERANCE.—The error of any indicated capacity shall not be greater than 0.05 ml.

INTERPRETATION OF THE SPECIFICATION.—The foregoing specification is intended to represent the most desirable form of specific-gravity flask for use in testing cements. Variations of a few millimeters in such dimensions as total height of flask, diameter of base, etc., are to be expected and will not be considered sufficient cause for rejection. The requirements in regard to tolerance, inscriptions, length, spacing, and uniformity of graduations will, however, be rigidly enforced.

(c) **Babcock Test Bottles.**—The following specifications for standard Babcock glassware were proposed and adopted at a meeting held in Washington, D. C., on March 28, 1911, by a committee representing the dairy division of the United States Department of Agriculture, the Official Dairy Instructors' Association, the United States Bureau of Standards, and the manufacturers of Babcock glassware. They have since that time been adopted by the Official Dairy Instructors' Association and by the dairy departments of several States, and are now being very generally employed by manufacturers and users of Babcock glassware.

SPECIFICATIONS.—The standard Babcock test bottles for milk and cream shall be the following:

- (1) Eight per cent, 18-gram, 6-inch milk-test bottle.
- (2) Fifty per cent, 9-gram, 6-inch cream-test bottle.
- (3) Fifty per cent, 9-gram, 9-inch cream-test bottle.

(1) *Eight Per Cent, 18-gram, So-called 6-inch Milk-test Bottle.*—**Graduation:** The total per cent graduation shall be 8. The graduated portion of the neck shall have a length of not less than 63.5

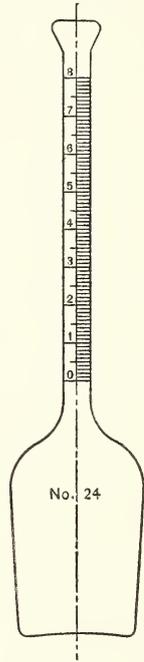


FIG. 10

mm (2.5 inches.)⁵ The graduation shall represent whole per cent, five-tenths per cent, and tenths per cent. The tenths per cent graduation shall be not less than 3 mm in length; the five-tenths per cent graduations shall be 1 mm longer than the tenths per cent graduations, projecting 1 mm to the left; the whole per cent graduations shall extend one-half way around the neck to the right, projecting 2 mm to the left of the tenths per cent graduations. Each per cent graduation shall be numbered, the number being placed on the left of the scale. The error at any point of the scale shall not exceed one-tenth of 1 per cent.

Neck: The neck shall be cylindrical for at least 9 mm below the lowest and above the highest graduation mark. The top of the neck shall be flared to a diameter of not less than 10 mm.

Bulb: The capacity of the bulb up to the junction of the neck shall not be less than 45 ml.⁶ The shape of the bulb may be either cylindrical or conical, with the smallest diameter at the bottom. If cylindrical, the outside diameter shall be between 34 and 36 mm; if conical, the outside diameter of the base shall be between 31 and 33 mm and the maximum diameter between 35 and 37 mm.

The charge of the bottle shall be 18 grams.

The total height of the bottle shall be between 150 and 165 mm ($5\frac{7}{8}$ and $6\frac{1}{2}$ inches).

Each bottle shall bear a *permanent identification number*.

(2) *Fifty Per Cent 9-gram, So-called 6-inch Cream-test Bottle.*—**Graduation:** The total per cent graduation shall be 50. The graduated portion of the neck shall have a length of not less than 63.5 mm (2.5 inches). The graduation shall represent 5 per cent, 1 per cent, and 0.5 per cent. The 0.5 per cent graduations shall be at least 3 mm in length; the 1 per cent graduations shall be 2 mm longer than the 0.5 per cent graduations, projecting 2 mm to the left; the 5 per cent graduation shall extend halfway around the neck to the right and project 4 mm to the left of the 0.5 per cent graduations. Each 5 per cent graduation shall be numbered the number being placed on the left of the scale. The error at any point of the scale shall not exceed five-tenths of 1 per cent.

Neck: (Same as standard milk-test bottle.) The neck shall be cylindrical for at least 9 mm below the lowest and above the high-

⁵ The English equivalents here given are only approximate.

⁶ In the original specifications the abbreviation "cc" was used instead of "ml" for designating the unit of volume.

est graduation mark. The top of the neck shall be flared to a diameter of not less than 10 mm.

Bulb: (Same as standard milk-test bottle.) The capacity of the bulb up to the junction of the neck shall not be less than 45 ml. The shape of the bulb may be either cylindrical or conical, with the smallest diameter at the bottom. If cylindrical, the outside diameter shall be between 34 and 36 mm; if conical, the outside diameter of the base shall be between 31 and 33 mm and the maximum diameter between 35 and 37 mm.

The charge of the bottle shall be 9 grams. All bottles shall bear on top of the neck above the graduations in plainly legible characters a mark denoting the weight of the charge to be used (9 grams).

The total height of the bottle shall be between 150 and 165 mm ($5\frac{7}{8}$ and 6.5 inches). (Same as standard milk-test bottles.)

Each bottle shall bear a permanent identification number.

(3) *Fifty Per Cent 9-gram, So-called 9-inch Cream-test Bottles.*—The same specifications in every detail as specified for the 50 per cent 9-gram 6-inch cream-test bottle shall apply to the 9-inch bottle, with the exception, however, that the total height of this bottle shall be between 210 and 225 mm ($8\frac{1}{4}$ and $8\frac{7}{8}$ inches), and the graduated portion of the neck shall have a length of not less than 125 mm.

THE STANDARD BABCOCK PIPETTE.—

Total length of pipette not more than.....mm..	7 330
Outside diameter of suction tube.....do....	6-8
Length of suction tube.....do....	130
Outside diameter of delivery tube.....do....	4 5-5.5
Length of delivery tube.....do....	100-120
Distance of graduation mark above bulb.....do....	30-60
Tolerance.....ml..	0.05
Nozzle straight.	

Delivery 17.6 ml of water at 20° C in 5 to 8 seconds.

SKIM-MILK-TEST BOTTLES.—The committee did not adopt a standard skim-milk-test bottle, for the reason that experiments have shown that it can give only approximate results.

Babcock test bottles will be calibrated on the basis that on an 18-gram bottle each 1 per cent interval has a capacity of 0.2 ml at 20° C, and that on a 9-gram bottle each 1 per cent interval has a capacity of 0.1 ml at 20° C. The bottles indicate the percentages (by weight) of fat in 18 and 9 grams, respectively, of milk or cream, the fat being assumed to have a specific gravity of 0.9.

In using the test bottle the percentage of fat is read off at relatively high temperatures (40° to 60° C), but the error resulting from the increase in volume of the test bottle between 20° C and the temperature at which the readings are made may be disregarded, as in no case does it amount to more than one-tenth of the error allowed in the graduation of the bottle.

* Unless otherwise requested, the capacity of each test bottle will be determined at two points; and if found correct within the limits allowed, the bottle will be stamped with the official precision stamp of the bureau.

5. TOLERANCES

(a) Flasks.—

Capacity (in milliliters) less than and including—	Limit of error		Capacity (in milliliters) less than and including—	Limit of error	
	If to contain—	If to deliver—		If to contain—	If to deliver—
	ml	ml		ml	ml
25.....	0.03	0.05	1000.....	0.30	0.50
50.....	.05	.10	2000.....	.50	1.00
100.....	.08	.15	3000.....	.75	1.50
200.....	.10	.20	4000.....	1.00	2.0
300.....	.12	.25	5000.....	1.2	2.4
500.....	.15	.30	6000.....	1.5	3.0

(b) Transfer Pipettes.—

Capacity (in milliliters) less than and including—	Limit of error	Capacity (in milliliters) less than and including—	Limit of error
	ml		ml
2.....	0.006	50.....	0.05
5.....	.01	100.....	.08
10.....	.02	200.....	.10
30.....	.03		

(c) Burettes and Measuring Pipettes.—

Capacity (in milliliters) of total graduated portion less than and including—	Limit of error of total or partial capacity		Capacity (in milliliters) of total graduated portion less than and including—	Limit of error of total or partial capacity	
	Burettes	Measuring pipettes		Burettes	Measuring pipettes
	ml	ml		ml	ml
2.....		0.01	30.....	0.03	0.05
5.....	0.01	.02	50.....	.05	.08
10.....	.02	.03	100.....	.10	.15

(d) Cylinders.—

Standard Cylindrical Graduates (Metric Units)

Capacity (in milliliters)	Limit of error of total or partial capacity		Capacity (in milliliters)	Limit of error of total or partial capacity	
	To contain—	To deliver—		To contain—	To deliver—
	ml	ml		ml	ml
5.....	0.02	0.03	200.....	0.40	0.60
10.....	.03	.05	250.....	.45	.70
25.....	.06	.10	500.....	.85	1.30
30.....	.07	.11	1000.....	1.6	2.4
50.....	.10	.15	2000.....	3.0	4.5
100.....	.20	.30			

Standard Cylindrical Graduates (U. S. Customary Units)

Capacity	Limit of error of total or partial capacity			
	To contain—		To deliver—	
	ml	dram	ml	dram
1 liquid ounce=29.57 ml.....	0.07	0.02	0.11	0.03
2 liquid ounces=59.15 ml.....	.12	.03	.18	.05
4 liquid ounces=118.29 ml.....	.24	.06	.36	.10
8 liquid ounces=236.58 ml.....	.45	.12	.68	.18
16 liquid ounces=473.17 ml.....	.80	.22	1.20	.33
32 liquid ounces=946.33 ml.....	1.50	.40	2.25	.60
		Cubic inches		Cubic inches
3 cubic inches=49.16 ml.....	.10	.006	.15	.009
10 cubic inches=163.87 ml.....	.33	.02	.50	.03
35 cubic inches=573.55 ml.....	1.00	.06	1.50	.09
1 dry pint=550.6 ml.....	1.00	.06	1.50	.09

(e) Delivery Time.—The actual delivery time of any instrument must be within the limits prescribed in the specifications, and the error in the marked delivery time must not exceed the following:

Delivery time (in seconds) less than and including—	Limit of error in marked delivery time	Delivery time (in seconds) less than and including—	Limit of error in marked delivery time
	secs		secs
15.....	3	50.....	8
20.....	4	100.....	15
30.....	6	200.....	20

II. 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.

The water remaining in the tip should not be blown out.

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

Burettes and pipettes should be filled to about 1 cm above the zero, and the setting to the zero mark made by slowly emptying from this point.

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.

For the convenience of those who may wish to calibrate their own volumetric apparatus a brief description of the method to be employed is here given.

In the test of burettes, measuring pipettes, and transfer pipettes the capacity of any interval may be determined by weighing the water delivered by the interval in question. The water is delivered into a weighing flask of convenient size provided with a stopper. The temperature of the water is observed either immediately before or immediately after delivery from the apparatus. A convenient method of observing the temperature is to have the weighing flask provided with a rubber stopper in which a thermometer is securely held, with its bulb near the bottom of the flask. If the thermometer is read immediately after the water is delivered into the flask, the observed temperature will be very close to that of the water while in the apparatus under test.

By means of Tables 35 to 47, Circular No. 19, the capacity of the apparatus is determined from the weight of the water contained or delivered by the apparatus. For example, if it is desired to test the total interval of a 50-ml burette, the procedure is as follows: The burette is first carefully cleaned, and then filled with distilled water to about 1 cm above the zero mark. The water surface is then set accurately on the zero by delivering into any convenient vessel the excess water above the zero. The tip of the burette is then stroked off with filter paper to remove any excess water from the tip, and the water in the interval under test is then delivered into a weighing flask of convenient size fitted with a rubber stopper and thermometer.

In making the weighings it is both convenient and accurate to use the method of substitution. By this method a constant tare is kept on one pan of the balance, while on the other pan is placed the object to be weighed and with it sufficient weights to secure equilibrium. In testing the burette or other volumetric apparatus the flask is first weighed empty and then again after having deliv-

ered into it the water from the interval under test. The difference between the two weights is the weight of the water delivered.

Suppose, for example, that with a certain tare on the right-hand pan and the empty flask on the left a load of 151.276 grams is required in addition to the flask to restore equilibrium, and that after the water from the 50-ml interval of the burette under test is delivered into the flask only 101.448 grams are required, and that the water is at a temperature of $23^{\circ}.4$ C. The weight of the water added is then $151.276 - 101.448 = 49.828$ grams. From Table 41, Circular No. 19, it is seen that to determine the capacity of a 50-ml vessel at 20° from the weight of water delivered at $23^{\circ}.4$ C, 0.174 must be added to the weight; that is, the capacity of the 50-ml interval of the burette under test at 20° C is $49.828 + 0.174 = 50.002$ ml.

The correction to be applied to the weight of water delivered by any other interval to obtain its capacity at 20° C is obtained in the same way. For example, to the weight of water delivered by the 10-ml interval at 18° C must be added one-tenth of the amount shown in Table 38 for 100 ml; for a 20-ml interval, one-tenth of that shown in Table 36.

In testing a flask or cylinder it is only necessary, after cleaning and drying the apparatus, to weigh it empty, fill it accurately to the graduation mark, and again weigh. The procedure to find the capacity at 20° from the weight of the water contained at a known temperature is the same as outlined above for the burette.

In case flasks are to be tested in large numbers a volumetric method may be used to advantage. In the method employed at this Bureau a series of volumetric standards have been made up, each standard having a capacity slightly less than that of the flask it is intended to test. When the water from the appropriate standard is delivered into a flask, the flask is filled nearly to the graduation mark. The filling is then completed by means of a finely graduated burette. The capacity of the flask is then found from the known volume delivered by the standard and the additional volume delivered by the burette.

The flask standard and the burette are themselves calibrated as outlined above by weighing the water delivered.

III. 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 volumetric flasks, specific gravity flasks, cylinders, transfer pipettes, and Babcock test bottles 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, in addition to the precision stamp, be assigned a Bureau of Standards certificate number as shown below:

B. S. No. 1763
1916

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

When desired, certificates of capacities of volumetric flasks, specific gravity flasks, cylinders, transfer pipettes, and Babcock test bottles will also be furnished.

4. SPECIAL TESTS

The Bureau will gladly cooperate with scientific investigators, manufacturers of apparatus, and others who need higher precision than is provided in the regular tests, as far as the regular work of

the Bureau will permit. Kinds of tests not at present provided for may be undertaken if the work is important and the facilities and time are available. Approved tests not provided for in the regular schedules will be considered special, and a special fee will be charged for them. The test should be arranged for by correspondence before shipment of the apparatus. The application should state fully the purpose for which the apparatus has been used or is to be used in the future, the need for the test, and the precision desired. The special fee charged will depend chiefly upon the time consumed and the amount of alteration required in the regular testing apparatus. An estimate of the fee will be given when possible.

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

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. Representatives of National or State institutions entitled to tests free of charge must make application in writing for each test in order to avail themselves of the privilege. Unless certificates are requested, volumetric flasks, specific gravity flasks, transfer pipettes, cylinders, and Babcock test bottles 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 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. Delay and cost of transportation on apparatus not entitled to verification will thus be avoided.

The Bureau does not sell volumetric apparatus. It may be purchased from manufacturers, importers, or jobbers, and submitted to the Bureau for test.

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 Bureau of Standards test number should also be used.

SHIPPING DIRECTIONS.—Apparatus should be securely packed in cases or packages which will not be broken in transportation and which may be used in returning them to the owner. The shipment in both directions is at the applicant's risk. It is recommended that shipment be made by express. Great care should be taken in packing. Clean, dry excelsior is a suitable packing material in most cases. Each instrument should also be wrapped in strong paper or other covering to prevent dust and excelsior from getting into it. The tops of boxes should be put on with screws, as the jar due to nailing and the subsequent opening is liable to cause damage. The tops of the shipping boxes should have the return or forwarding address on the underside. Transportation charges are payable by the party requesting the test. The charges for shipment to the Bureau must be prepaid, and, unless otherwise arranged, articles will be returned or forwarded by express "collect."

When apparatus is submitted by one party with instructions to test and forward to another, directions should be given for the disposition of such of the apparatus as may be rejected by the Bureau. If such instructions are not given at the time the test is submitted, much unnecessary delay results.

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, 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 or check 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, Washington, D. C."

V. SCHEDULE OF FEES

Schedule 25.—VOLUMETRIC APPARATUS

1. FLASKS, TRANSFER PIPETTES, AND BABCOCK TEST BOTTLES

- (a) For testing and stamping, each capacity tested \$0.25
 (b) For testing, stamping, and certificate of capacity, each capacity tested35

2. BURETTES AND MEASURING PIPETTES

- (c) For testing and certifying capacity of five intervals 1.25
 (d) For testing and certifying capacity of additional intervals, each additional interval25

3. CYLINDERS

- (e) For testing and stamping, each capacity tested25
 (f) For certificate of capacity at five points 1.50

4. SPECIFIC-GRAVITY FLASKS

- (g) For testing and stamping, each capacity tested25
 (h) For certificate of capacity at five points 1.75

5. TEMPERATURES OTHER THAN 20° C

- (i) For testing apparatus intended for temperatures other than 20° C between 15° C and 30° C, additional charges for each piece20

6. UNITS OF CAPACITY OTHER THAN MILLILITERS

- (j) For testing apparatus of indicated capacity other than in milliliters, additional charges for each piece20

7. DISQUALIFIED APPARATUS

- (k) For preliminary examination of apparatus disqualified for test, charges for each piece10

8. IDENTIFICATION NUMBERS⁸

- (l) For supplying missing identification numbers, charges for each number20

S. W. STRATTON,
Director.

Approved:
 WILLIAM C. REDFIELD,
Secretary.

⁸ In the case of apparatus submitted for an institution for which tests are made free of charge the bill for testing rejected apparatus and for supplying missing identification numbers will be rendered to the concern furnishing the apparatus.

