

Testing of Glass Volumetric Apparatus

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National Bureau of Standards Circular 602

Issued April 1, 1959

[Supersedes Circular 434]

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C.

Price 20 cents

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This Circular contains specifications and tolerances for glass volumetric apparatus of precision grade. Detailed information is given as to dimensions, graduations, inscriptions, and tolerances for burets, pipets, flasks, cylindrical graduates, and certain kinds of special apparatus.

A description of test methods used and the reports furnished and directions for submitting apparatus are included.

1. Introduction—Purpose and Scope

This Circular presents, to manufacturers and users, information on the design, construction, and verification of glass volumetric apparatus and indicates the facilities available at the Bureau for this verification.

The specifications and tolerances given herein

apply to burets, flasks, pipets, and other glass volumetric apparatus of precision grade.

Information is given on the method of reading the test liquid used, and the methods of test, description of the tests performed and the reports furnished and directions for submitting apparatus are included.

2. Standard Specifications for Glass Volumetric Apparatus

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 their 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 its accurate calibration. Certain of the specifications, such as those regarding quality of glass and process of annealing before calibration, are largely dependent on the integrity of the maker for their fulfillment. 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.

2.1. Types of Apparatus That Will Be Regularly Admitted for Test

Precision grade apparatus of the following types will be accepted for test on written request: transfer pipets and capacity pipets, without subdivisions; burets, measuring pipets, and dilution pipets, with partial or complete subdivisions; volumetric flasks, cylindrical graduates, and specific gravity flasks.

Other types of apparatus should not be submitted without prior arrangement.

2.2. General Specifications¹

a. Units of Capacity

A *liter* is equal to the volume occupied by a mass of 1 kg of pure water at its maximum density (at a temperature of 4° C, practically).

A *milliliter* (ml),² is the one-thousandth part of the liter.

b. Standard Temperature

Although 20° C has been almost universally adopted as the standard temperature for glass volumetric apparatus, many chemists are of opinion that 25° C more nearly approximates average laboratory temperature in the United States. The Bureau therefore will calibrate glass volumetric apparatus marked either 20° or 25°

c. Material and Annealing

The material should be of best quality glass, transparent and free from striae, surface irregularities, and other defects which may distort the appearance of the liquid surface or the portion of the graduation line seen through the glass. The apparatus should be thoroughly annealed before being graduated.

d. Design and Workmanship

The cross section must be circular and the shape must permit complete emptying and draining and thorough cleaning.

Instruments having a base or foot must stand solidly on a level surface. For all instruments

¹ The Bureau reserves the right to reject any apparatus on points of its accuracy or utility not covered by these specifications.

² The terms "milliliter" and "cubic centimeter" are sometimes used interchangeably. While they are not exactly equal (1 ml = 1.000028 cm³) the difference is seldom of consequence in volumetric analysis.

except small flasks (below 25 ml) the base shall be of such size that the instrument, empty and without stopper, will stand on a plane inclined 15° to the horizontal. Flasks smaller than 25 ml shall stand on a 10° incline.

Stoppers and stopcocks must be ground or fitted so as to work easily and prevent leakage.

Apparatus which is manifestly fragile or otherwise defective in construction will not be accepted.

The parts on which graduations are placed must be cylindrical for at least 1 cm on each side of every mark (except on certain small flasks and special apparatus), but elsewhere may be enlarged to provide the desired capacities in convenient lengths.

Two scales are not permitted on the same piece of apparatus. For example, apparatus should not be graduated in both fluid 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 fluid ounces, there is no objection to having the 8-dr line, 16-dr line, etc., marked respectively, 1 fl oz, 2 fl oz, etc., provided that the two series of numbers are placed on opposite sides of the apparatus and the value of each subdivision is suitably indicated.

e. Graduation Lines

Graduation lines shall be fine, clean, permanent, continuous, and of uniform width, perpendicular to the axis and parallel to the base of the apparatus. Line width should not exceed 0.3 mm for subdivided apparatus and 0.4 mm for single-line apparatus.

All graduations must extend at least halfway around; and on subdivided apparatus at least every 10th mark, and on undivided apparatus, all marks must extend completely around the circumference. Subdivided apparatus must be provided with a sufficient number of lines of suitable length to facilitate reading.

The clear space between two adjacent marks must be not less than 1 mm wide. The spacing of marks on subdivided apparatus must show no apparent irregularities, and sufficient divisions must be numbered to readily indicate the intended capacity of any interval.

f. Inscriptions

Every instrument must bear in permanent 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 on instruments which deliver through an outflow nozzle, the time required to empty the total nominal capacity with unrestricted outflow. Etching is preferred for inscriptions, although engraved or fused marking is acceptable provided such marking is neat and clear. Grit-blasted serial numbers will be permitted with the same provision.

Every instrument must bear the name or trademark of the maker. Every instrument must bear a permanent identification number, and detach-

able parts, such as stoppers, stopcocks, etc., belonging thereto, if not interchangeably ground, must bear the same number. Interchangeable ground-glass parts shall be marked on both members with the proper standard taper symbol and the size designation, in accordance with Commercial Standard CS 21-57.

Figure 1 illustrates several arrangements of designating marks that are considered suitable. Marks may be placed elsewhere on apparatus if they are easily readable and do not interfere with the proper use of the apparatus.

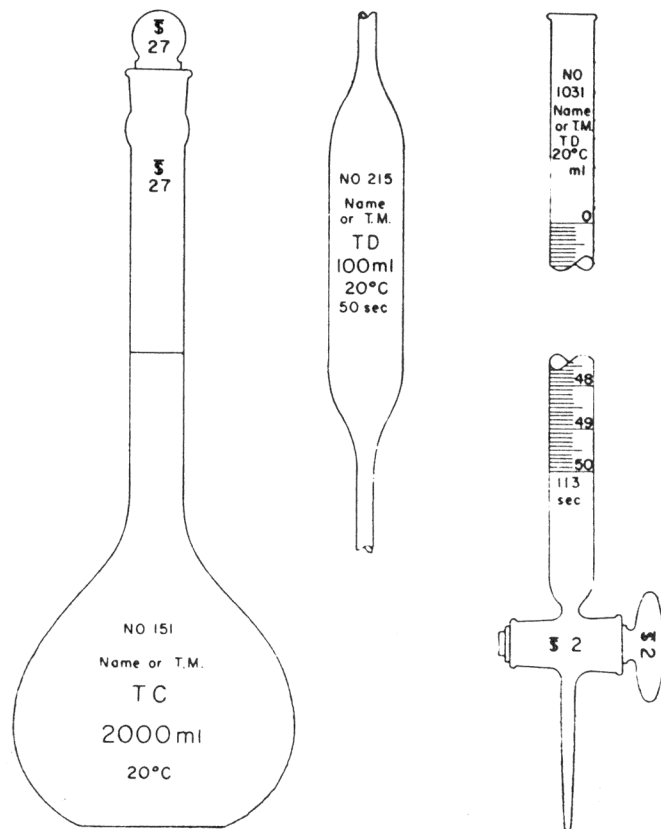


FIGURE 1. Examples of inscriptions suitable for volumetric apparatus.

2.3. Special Requirements

a. Flasks

The limiting dimensions for precision-grade volumetric flasks are given in table 1. For flasks smaller than 5-ml capacity, the limits on location of the graduation line will be the same as shown for a 5-ml flask.

Stopper sizes should be so chosen that the smallest diameter of the ground zone in the flask neck is approximately equal to the inside diameter at the graduation line. The inside diameter of the neck at the graduation line should never exceed the smallest diameter of the ground zone by more than 1.5 mm. The shoulder at the base of the ground zone should be smoothly rounded to permit complete drainage.

TABLE 1. *Limiting dimensions for volumetric flasks*
(In millimeters)

Capacity	Inside diameter of neck at graduation line		Minimum distance between graduation line and:		
	Minimum	Maximum	Top of flask		Bulb ^a
			Stopped type	Unstopped type	
<i>ml</i>					
5.....	6	7	22	22	5
10.....	6	8	28	28	7
25.....	6	8	35	30	10
50.....	6	10	40	30	10
100.....	8	12	40	30	10
200.....	9	14	55	45	10
250.....	10	15	55	45	10
500.....	12	18	60	60	20
1,000.....	14	20	70	60	20
2,000.....	18	25	70	60	20

^a The distance between the graduation line and the bulb is measured to the point where the neck begins to expand into the bulb (on outside).

A flask may be graduated both *to contain* and *to deliver*, provided the intention of the different marks is clearly indicated and provided the distance between the two marks is not less than 1 mm.

b. Cylindrical Graduates

Cylinders may be graduated either to contain or to deliver, but a scale numbered both up and down the length of the graduate is not permitted, as it is obvious that the same graduate cannot be correct both to contain and to deliver.

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 cylinder 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. To avoid excessive height, the subdivisions on 10-ml, 25-ml, and 50-ml cylinders may be 0.2 ml, 0.5 ml, and 1.0 ml, respectively.

Subdivision lines shall be omitted between the base and the first numbered line. This will eliminate readings near the base which are difficult and not always accurate. (See fig. 2.)

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

Method of use—Graduates that are to be used dry to receive and measure liquids should be calibrated to contain.

Graduates that are to be used to pour water 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 cali-

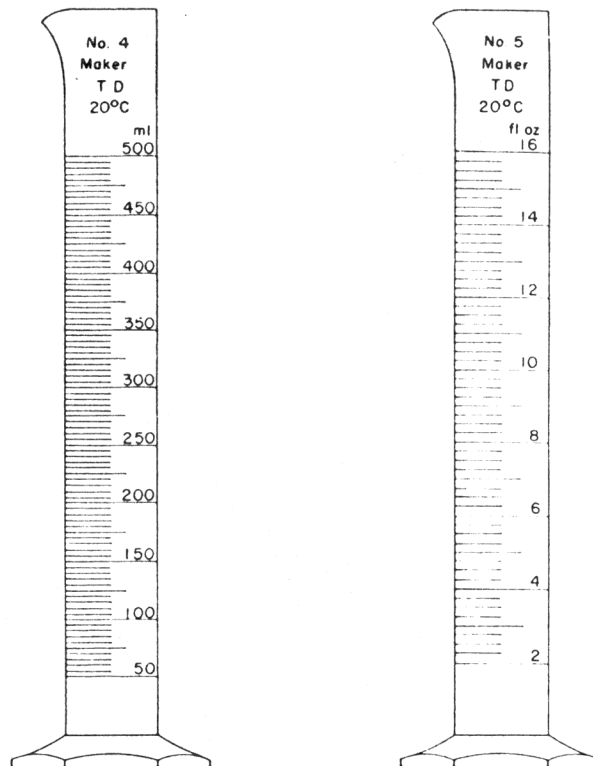


FIGURE 2. *Examples of cylindrical graduates.*

brated 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.

Basis of graduation—Graduates in customary units should be graduated in accordance with the following relations:

$$\begin{aligned}
 60 \text{ minims} &= 1 \text{ fl dr} \\
 8 \text{ fl dr} &= 1 \text{ fl oz} \\
 32 \text{ fl oz} &= 1 \text{ liq qt} \\
 4 \text{ liq qt} &= 1 \text{ U. S. gal} \\
 1 \text{ U. S. gal} &= 231 \text{ in.}^3
 \end{aligned}$$

For conversion to the metric system, the relation is:

$$1 \text{ fl oz} = 29.5729 \text{ ml}$$

TABLE 2. *Limits of distance from highest graduation to top of graduate*

Total nominal capacity (customary units)	Distance of highest graduation from top		Total nominal capacity (metric units)
	Minimum	Maximum	
<i>Minims</i>	<i>cm</i>	<i>cm</i>	<i>ml</i>
60.....	2	4	5
120.....	2	4	10
<i>fl oz</i>			
1.....	2	5	25
2.....	3	5	50
4.....	3	6	100
8.....	4	8	250
16.....	5	10	500
32.....	5	10	1,000

TABLE 3. Minimum neck diameter of cylinders with stoppers

Capacity.....ml..	5	10	25	50	100	250	500	1,000
Minimum diameter of neck.....mm..	6	7	8	9	10	12	15	18

c. Transfer Pipets

Pipets for delivering a single volume are designated "transfer" pipets.

The suction tube of each transfer pipet must be at least 16 cm long, and the delivery tube must be not less than 3 cm nor more than 25 cm long. The top of the suction tube must be finished with a smooth plane surface, at right angles to the axis.

The outside diameter of the suction and delivery tubes of transfer pipets, exclusive of the tip, must be not less than 5 mm. Limits of inside diameter at the capacity mark are given in table 4.

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

The outlet of any transfer pipet must be of such size that the free outflow shall last not more than 1 min and not less than the times shown in table 5 for the respective sizes.

TABLE 4. Inside diameter of transfer pipets at capacity mark

Capacity of pipets (in milliliters) up to and including.....	25	50	200
Diameter (in millimeters): Max.....	4	5	6
Min.....	2	2	2

TABLE 5. Minimum delivery times for transfer pipets

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

d. Burets and Measuring Pipets

Only those burets emptying through a nozzle permanently attached at the bottom are accepted for test.

Side tubes, unless provided with stopcocks, are not permitted on burets.

So-called Schellbach burets—that is, those having a milk-glass background with a colored center line—will not be accepted for test on account of possible errors resulting from parallax.

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

The rate of outflow of burets and measuring pipets must be restricted by the size of the tip, and for the graduated length the time of free outflow must be not more than 3 min nor less than the times shown in table 6.

The upper end of any measuring pipet 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- and 100-ml burets, the highest graduation mark should be not less than 4 cm nor more than 10 cm from the upper end of the buret.

TABLE 6. Minimum delivery times for burets and measuring pipets

Length graduated	Time of outflow not less than—	Length graduated	Time of outflow not less than—
<i>cm</i>	<i>sec</i>	<i>cm</i>	<i>sec</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

On burets having a capacity of 25 ml or less this distance should be not less than 3 cm nor more than 6 cm. (These requirements do not apply to burets where the "zero" is at the end of an overflow tip.)

e. Buret and Pipet Tips

Buret and pipet tips should be made with a gradual taper of from 2 to 3 cm, the taper at the extreme end being slight.

A sudden contraction at the orifice is not permitted, and the end of the tip must be ground perpendicular to the axis of the tube. The outside edge should be beveled slightly and all ground surfaces polished.

In order to facilitate the removal of drops and to avoid splashing, the tip of a buret may be bent slightly.

Approved forms of tips for burets, measuring pipets, and transfer pipets are shown in figure 3.

2.4. Special Apparatus

a. Giles Flasks

The permissible error in the volume at the first mark shall be the tolerance allowed for a flask of that capacity. The permissible error in the volume at the second mark shall be the tolerance at the first mark plus the tolerance for a flask of capacity equal to the difference between the two marks, provided, however, that the error in the volume between the two marks shall not exceed the permissible error in the volume indicated by the first mark.

At the capacity mark the inside diameter of the neck shall be within the limits given in table 7.

The neck shall be cylindrical on each side of every graduation mark for at least the distances given in table 8.

On account of the bulb in the neck of a Giles flask it is more unstable or topheavy 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.

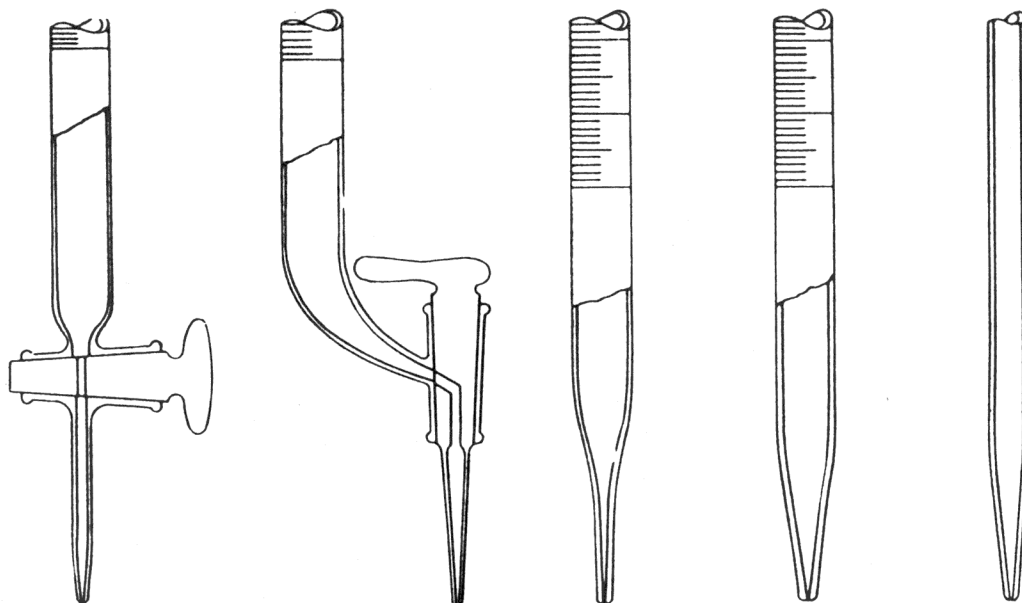


FIGURE 3. Types of buret and pipet tips.

TABLE 7. Neck diameters of Giles flasks

Capacity	Inside diameter of neck	
	Minimum	Maximum
<i>ml</i>	<i>mm</i>	<i>mm</i>
25 to 27.5.....	6	9
50 to 55.....	6	11
100 to 110.....	8	13
200 to 220.....	9	15
500 to 550.....	12	18
1,000 to 1,100.....	14	20
2,000 to 2,200.....	18	25

TABLE 8. Minimum length of cylindrical portions of neck on Giles flask

Capacity	Above upper graduation mark	Between upper graduation mark and bulb	Between lower graduation mark and bulb	Below lower graduation mark
<i>ml</i>	<i>cm</i>	<i>cm</i>	<i>cm</i>	<i>cm</i>
25 to 27.5.....	3	0.8	1	1
50 to 55.....	3	.8	1	1
100 to 110.....	3	.8	1	1
200 to 220.....	4	.8	1.5	2
500 to 550.....	4	1.0	1.5	2
1,000 to 1,100.....	5	1.0	1.5	2
2,000 to 2,200.....	5	1.0	1.5	2

b. Specific Gravity Flasks

Material and annealing—The material from which the flasks are made shall be glass of the best quality, transparent, and free from striae. It shall adequately resist chemical action and have small thermal hysteresis. The flasks shall be thoroughly annealed 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 figure 4. 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. 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 to 24 ml into 0.1-ml divisions. There shall be two 0.1-ml graduations below the 0 and two above the 1-ml graduation.

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, if not interchangeably ground, 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 be not 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, and the length, spacing, and uniformity of graduations will, however, be rigidly observed.

d. Babcock Milk Test Apparatus

Because most of the States now require that all Babcock glassware used in the State be approved by officials of that State, the National Bureau of Standards no longer accepts Babcock bottles and pipets for calibration, except when specifically requested by a State to settle a dispute. Specifications for Babcock glassware may be found in official procedures of the Association of Official Agricultural Chemists and certain dairy associations.

e. Dilution (Hemocytometer) Pipets

The pipets should be of good quality glass, free from bubbles and other defects, and sufficiently strong to withstand normal usage. Tubing with milk-glass backing is permissible. The capillary should be of uniform diameter, except that the tip of a white cell pipet may be tapered slightly. The inside diameter at the tip should be between 0.3 and 0.5 mm. On all pipets the tip should be ground smooth and at right angles to the axis of the pipet, and beveled so that the external diameter at the end does not exceed 2.0 mm. All ground surfaces should be polished to restore transparency.

The bulb or mixing chamber should contain a small, nonspherical glass bead to aid in mixing the blood and diluting fluid. Pipets for red blood cells may be designated by a red bead, by a red stripe running parallel to the axis, or both. White cell pipets should have white or clear beads.

The bulb should be so shaped that it can be filled without entrapping air bubbles. The capacity of the bulb is usually about 0.8 to 1.2 ml for the red cell pipet, and 0.2 to 0.4 ml for the white cell pipet.

The pipet should be so graduated as to give dilution ratios of 1 to 10, or 1 to 100, and may be further graduated to give ratios of 0.1, 0.2, 0.5, etc., to 10, and to 100; that is, the interval 0 to 1 may be subdivided to 0.1 or 0.5. The subdivisions, when present, should be uniformly spaced along the capillary tube. The graduations at 0.5, 1.0, 11, and 101 should be numbered.

All numbered graduation lines should extend at least halfway around the pipet, diametrically opposite to the milk-glass backing. Etched lines are preferred, but engraved or fused lines are acceptable provided they meet the requirements of paragraph 2.2.e. The "1" line shall be located not less than 2 mm nor more than 5 mm below the bulb, and the "11" (or "101") line shall be not less than 2 mm nor more than 4 mm above the bulb. Measurement is from the points where the capillaries begin to expand into the bulb.

Dilution pipet tolerances—If V represents the volume between the marks immediately above and below the bulb, v_1 represents the volume of the capillary between the tip and the line marked "1", and v_2 represents the volume of the capillary between the tip and the line marked "0.5", the tolerances may be expressed as follows:

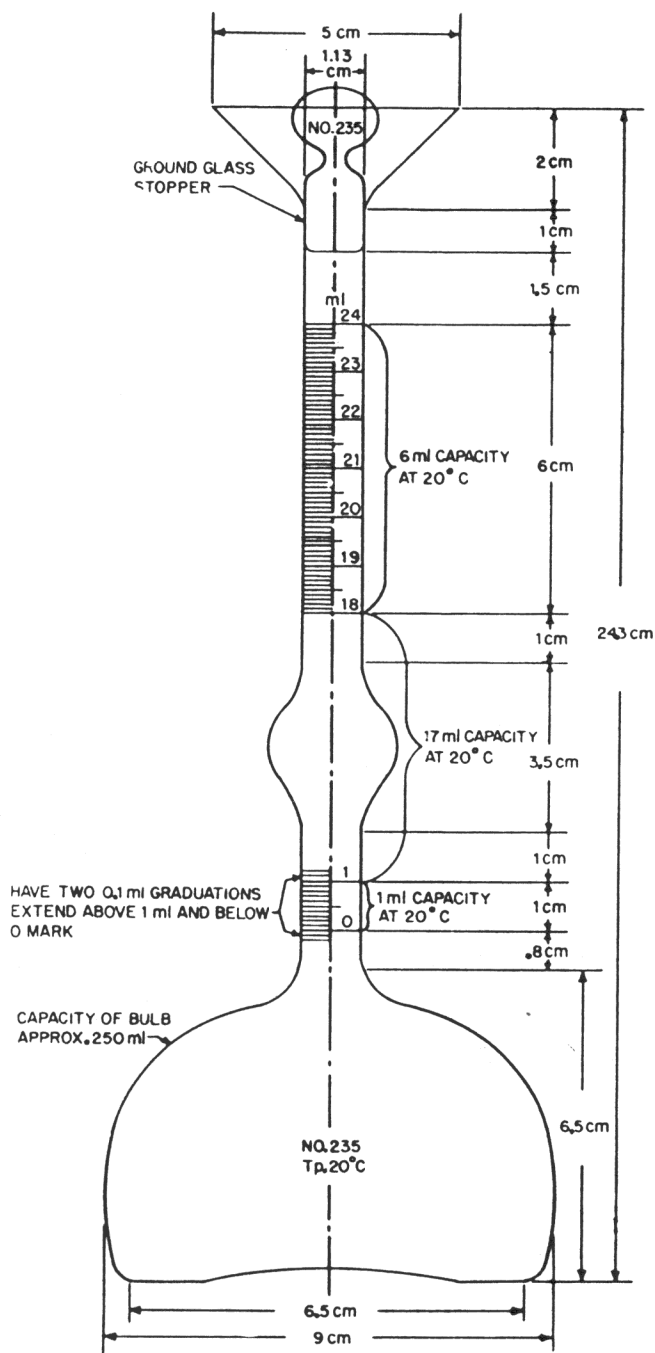


FIGURE 4. *Specific-gravity flask.*

c. Sugar-Testing Flasks (Bates)

The flask shall have a height of 130 mm, and the neck shall be 70 mm in length; a tolerance of ± 5 mm is allowed for each. The internal diameter shall be not less than 11.5 mm and not more than 12.5 mm. The upper end of the neck shall be flared, and the graduation mark shall be not less than 30 mm from the upper end and 15 mm from the lower end of the neck. The flask shall be standardized to contain 100 ml at 20°C with a tolerance of 0.08 ml.

- Red cell pipets—nominal ratio 100:1,
 V/v_1 must be not less than 95 and not more than 105
 V/v_2 must be not less than 190 and not more than 210.
- White cell pipets—nominal ratio 10:1,
 V/v_1 must be not less than 9.65 and not more than 10.35
 V/v_2 must be not less than 19.30 and not more than 20.70.

In an effort to give more exact information as to accuracy, certain manufacturers have marked dilution pipets with a so-called "correction factor" This is usually written simply "+2", "-1", etc. Because of the possibility that such a correction will not be applied as the manufacturer intended, and because mass-production test methods cannot always be relied upon to fix the corrections within 1 percent, the Bureau does not recommend this practice. In those cases where greater accuracy is required than is provided by the application of the prescribed tolerances ($\pm 5\%$ for red cell pipets and $\pm 3.5\%$ for white cell pipets), it is suggested that the purchaser specify the desired limits of error. This would provide the needed accuracy without the application of corrections.

The Bureau does not accept for test pipets bearing a "correction factor" except in very special cases and only in small quantities.

f. Sahli Hemoglobin Pipets

The capacity tolerance for Sahli pipets is ± 2 percent of the nominal volume.

g. Immunity Unit Cylinders and Pipets

These instruments were designed by the Hygienic Laboratory of the U. S. Public Health Service (USPHS) for use in the measurement of toxin and serum dilutions. Specifications may be found in USPHS Hygienic Laboratory Bulletin 21. The instruments are made to conform in general with the specifications contained in this Circular, but with certain modifications necessary to adapt them to the specific application.

The pipets are of three types: (1) Capacity or "wash-out" pipets, calibrated to contain the nominal volume; (2) delivery pipets, calibrated to deliver the nominal volume when the residue in the tip after free outflow has ceased is blown out and added to the initial delivery; (3) graduated delivery pipets, called Ehrlich pipets, calibrated to deliver the contents in the same manner as transfer pipets.

Tolerances for the first two types of pipets named are the same for ordinary transfer pipets; for the Ehrlich delivery pipets, the tolerance is 0.01 ml. For the cylinders, the tolerances are: For capacities less than and including 50 ml—0.05 ml; for capacities over 50 ml and including 100 ml—0.08 ml.

Certain types of special-purpose volumetric flasks such as the Engler viscosity flask and the Kohlrausch and Stift sugar flasks, while not in conformity with all the special requirements for volumetric flasks listed in section 2.3.a because of their specific applications, will be calibrated and marked with the precision stamp if they are in satisfactory conformity with the general specifications in section 2.2 and the errors in capacity do not exceed the tolerances for volumetric flasks given in section 2.5.

i. Other Special Apparatus

Other types of special-purpose instruments may be calibrated and marked with the precision stamp if they conform with the general specifications (section 2.2) and the errors in capacity do not exceed the applicable tolerances in section 2.5.

Permanent identification numbers are required on all glass volumetric apparatus submitted for certification.

Instruments obviously not capable of precision measurements, as for example, centrifuge tubes, are not accepted because the time expended in their calibration is not warranted by the degree of accuracy required in their use.

2.5. Tolerances

a. Flasks

The capacity tolerances for flasks are given in table 9.

TABLE 9. Capacity tolerances for volumetric flasks

Capacity (in milliliters) less than and including—	Limit of error	
	If to contain—	If to deliver—
	<i>ml</i>	<i>ml</i>
1.....	0.01
3.....	.015
5.....	.02
10.....	.02	0.04
25.....	.03	.05
50.....	.05	.10
100.....	.08	.15
200.....	.10	.20
300.....	.12	.25
500.....	.15	.30
1,000.....	.30	.50
2,000.....	.50	1.00
Above 2,000.....	1 part in 4,000	1 part in 2,000

b. Transfer Pipets

The capacity tolerances for transfer pipets are given in table 10. These tolerances are applicable to pipets of similar design made to contain the nominal volumes.

c. Burets and Measuring Pipets

The capacity tolerances for burets and measuring pipets are given in table 11.

TABLE 10. Capacity tolerances for transfer pipets

Capacity (in milliliters) less than and including—	Limit of error
	<i>ml</i>
2.....	0.006
5.....	.01
10.....	.02
30.....	.03
50.....	.05
100.....	.08
200.....	.10

TABLE 11. Capacity tolerances for burets and measuring pipets

Capacity (in milliliters) of total graduated portion less than and including—	Limit of error of total or partial capacity	
	Burets	Measuring pipets
	<i>ml</i>	<i>ml</i>
2.....	0.01	0.02
5.....	.02	.03
10.....	.03	.05
30.....	.05	.08
50.....	.08	.15
100.....	.10	

d. Cylindrical Graduates

The capacity tolerances for cylindrical graduates are given in table 12.

e. Delivery Time

The actual delivery time of any instrument must be within the limits prescribed in section 2.3, and the error in the marked delivery time must not exceed the limits given in table 13.

TABLE 12. Capacity tolerances for cylindrical graduates

Metric units				Customary units			
Capacity of total graduated portion less than and including—	Maximum diameter	Limit of error of total or partial capacity		Capacity of total graduated portion less than and including—	Maximum diameter	Limit of error of total or partial capacity	
		If to contain—	If to deliver—			If to contain—	If to deliver—
<i>ml</i>	<i>mm</i>	<i>ml</i>	<i>ml</i>	<i>Minims</i>	<i>in.</i>	<i>Minims</i>	<i>Minims</i>
5.....	11	0.05	0.06	60.....	$\frac{3}{8}$	0.6	0.7
10.....	14	.08	.10	120.....	$\frac{1}{2}$	1.0	1.3
25.....	19	.14	.18		<i>fl oz</i>	<i>fl oz</i>	<i>fl oz</i>
50.....	23	.20	.26	1 (8 fl dr)....	$\frac{3}{4}$	0.005	0.006
100.....	29	.35	.40				
250.....	40	.65	.80	2.....	1	.008	.010
500.....	50	1.1	1.3	4.....	$1\frac{1}{4}$.014	.017
1,000.....	63	2.0	2.5	8.....	$1\frac{1}{2}$.021	.027
2,000.....	80	3.5	5.0	16.....	$1\frac{5}{16}$.035	.045
				32.....	$2\frac{1}{16}$.060	.080

TABLE 13. Limits of error in marked delivery time

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

f. Special Apparatus

Capacity tolerances for the special purpose instruments described in section 2.4 are included in the appropriate parts of that section.

3. 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.

3.1. Test Liquid

Apparatus will ordinarily be calibrated with distilled water, and the capacity determined will therefore be the volume of water contained or delivered by an instrument at its standard temperature.

Certain special types of apparatus, such as pipets having a capacity of 0.01 ml or less, are more accurately calibrated with mercury. Instruments designed to *deliver* their contents should always be calibrated with water.

3.2. Method of Reading

a. Using Water or Other Wetting Liquid

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.

The position of the lowest point of the meniscus with reference to the graduation line is such that it is in the plane of the middle of the graduation line. This position of the meniscus is obtained by making the setting in the center of the ellipse formed by the graduation line on the front and the back of the tube as observed by having the eye slightly below the plane of the graduation line. This is illustrated in figure 5. The setting is accurate if, as the eye is raised and the ellipse narrows, the lowest point of the meniscus remains midway between the front and rear portions of the graduation line. By this method it is possible to observe the approach of the meniscus from either above or below the line to its proper setting.

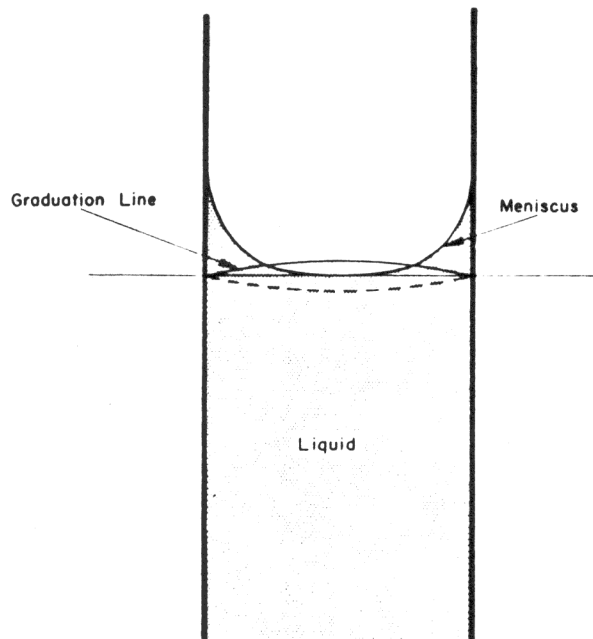


FIGURE 5. Method of setting water meniscus.

b. Using Mercury

In setting a mercury meniscus on a graduation line, the instrument must be tapped sharply in order that the meniscus may assume a normal shape. The *highest* point of the meniscus is set on the middle of the graduation line by employing the principles outlined for water, but observing from *above* the line.

3.3. Cleanliness of Apparatus

Apparatus must be sufficiently clean to permit uniform wetting of the surface and to minimize contamination of the liquid surface.

Of the various cleaning agents in common use, the Bureau prefers fuming sulfuric acid or a chromic-sulfuric acid mixture. For removal of oil and grease, an organic solvent is used and the cleaning completed with one of the above agents.

When drying is required before calibration, it is done with unheated compressed air which is cleaned and dried by passing through concentrated sulfuric acid and a drying compound such as calcium chloride.

3.4. Flasks and Cylinders

In filling flasks, the entire interior of the flask below the stopper will be wetted; in the case of cylinders, the liquid is allowed to flow down one side only. After filling to a point slightly below the graduation line, these instruments are allowed to drain for about 2 min. They are then placed below a buret having a long delivery tube and a bent tip, and the filling is completed by discharging water from the buret against the wall of the flask or cylinder about 1 cm above the graduation line, and rotating the receiving vessel to re-wet the wall uniformly.

Flasks and cylinders which are to be used to

deliver are filled approximately to the test point, then emptied by gradually inclining them, avoiding as much as possible agitation of the contents and re-wetting of the walls. Allow half a minute for emptying. When the continuous outflow has ceased, the vessel should be nearly vertical and should be held in this position for another half-minute. The adhering drop is removed by contact with the wetted wall of the receiving vessel. The rest of the calibration is performed in the same manner as in calibrating to contain.

In calibrating a flask or cylinder to contain it is only necessary, after cleaning and drying the apparatus, to weigh it empty, fill it accurately to the graduation mark, and again weigh. Table 16 or table 17 may be used to determine the capacity at 20° C from the weight of the water contained at a known temperature.

In case flasks are to be tested in large numbers, a volumetric method may be used to advantage. The Bureau uses a series of volumetric standard pipets (illustrated in fig. 6) each having a capacity slightly less than that of the flask it is intended to test. The water delivered from the appropriate standard pipet fills the flask nearly to the graduation mark. After the 2-min drainage period, the filling is completed from an accurately calibrated buret. The capacity of the flask is then found from the known volume delivered by the standard pipet and the additional volume delivered by the buret. The standard pipets and burets are themselves calibrated by weighing the water delivered.

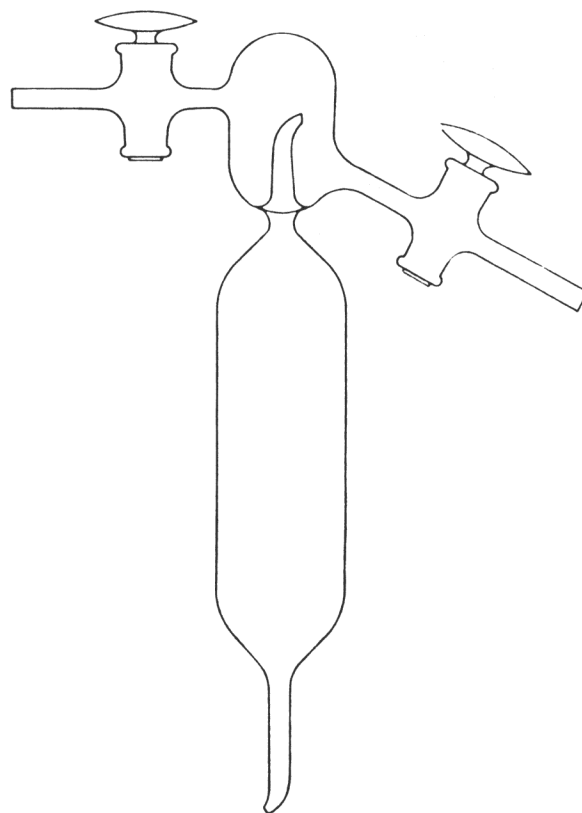


FIGURE 6. Standard pipet for flask calibrations.

The conical graduate is not classed as a precision volumetric measure, and is not recommended for use in checking the accuracy of other volumetric apparatus. The Bureau does not accept these graduates for calibration except when requested by a State weights and measures official.

Conical graduates are calibrated in the same manner as cylindrical graduates, except that in calibrating "to deliver", the emptying time is reduced from 30 sec to 10 sec and the drainage time (after emptying) is also reduced to 10 sec, following customary methods of use.

3.5. Burets

Burets are calibrated in a vertical position. After cleaning, they are filled with distilled water to about 1 cm above the zero line. They are allowed to stand for about 30 min to check for leakage, not only at the tip but also around the stopcock plug. The water is then allowed to discharge with unrestricted outflow and the delivery time is noted and recorded. This flushing serves to equalize the temperatures of apparatus and water. The water temperature is taken on the next filling by inserting a thermometer in the buret or, if it is not large enough to receive a thermometer, in a plain glass tube of larger diameter mounted near the buret.

If the temperature was taken in the buret, the thermometer is then removed and enough water added to raise the level to about 1 cm above the zero line. Any water on the outside of the tip is removed with absorbent paper. The setting of the meniscus on the zero line is made by allowing the water to run out slowly. After the setting is made, any excess water on the tip is removed by touching the tip with the wetted wall of a beaker or other vessel. The very small quantity of water which remains on the extreme end, or ground portion, of the tip is not removed, as the same quantity of water will remain after delivery into the weighing flask.

Delivery of the water into the weighing flask is made with the buret tip in contact with the inside of the flask neck, the flask being inclined at an angle of about 20°. When delivery has been completed, the weighing flask is immediately removed horizontally from contact with the instrument being calibrated. The flask is then stoppered and weighed. The buret and thermometer tube (if used) are refilled preparatory to measuring the next interval.

Buret stopcocks should be completely open during delivery until the water level is only a few millimeters above the test point, when the discharge must be slowed in order to make an accurate setting.

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 suffi-

cient weights to secure equilibrium. In testing volumetric apparatus, the receiving flask is first weighed empty and then again after having delivered 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 g is required in addition to the flask to restore equilibrium, and that after the water from the 10-ml interval of the buret under test is delivered into the flask, only 141.310 g are required, and that the water is at a temperature of 23.4°C. The weight of the water added is then $151.276 - 141.310 = 9.966$ g. From table 16 (appendix), it is seen that to determine the capacity of a 10-ml soft glass vessel at 20°C from the weight of water delivered at 23.4°C, 0.035 must be added to the weight; that is, the capacity of the 10-ml interval of the buret under test at 20°C is $9.966 + 0.035 = 10.001$ ml.

3.6. Measuring and Transfer Pipets

Measuring and transfer pipets are calibrated in much the same manner as burets, except that the tip must be in contact with the wet wall of a beaker or other vessel when the setting is made on the zero line of a measuring pipet or the capacity mark of a transfer pipet. Also, the delivery time of a transfer pipet is measured with the tip in contact with the vessel.

In calibrating transfer pipets, the water surface must be observed closely as it approaches the tip to make certain that outflow has ceased before the weighing flask is removed. On the other hand, if the flask removal is unduly delayed, after-drainage will affect the result.

The water remaining in the tip of a pipet is not blown out unless, as in the case of certain types of special-purpose pipets, there is a wide band or two narrower bands permanently marked near the top of the instrument.

The water temperature is measured in the beaker from which the pipet is filled (by suction).

3.7. Special Apparatus

The flasks and cylinders mentioned in section 2.4 are calibrated in the manner described in section 3.4, except that the inside of the neck is wetted for a distance of only about 1 cm or less above the test point.

A specific gravity flask is calibrated at the 1, 18, and 24 ml graduations.

Capacity pipets made to contain the nominal volume are calibrated by weighing before and after filling.

In calibrating "blow-out" pipets, the water remaining in the tip after free outflow has ceased is blown out, without waiting for drainage, and added to the quantity delivered. The blow-out is accomplished by a single strong puff, with the pipet tip in contact with the neck of the weighing flask.

4. Tests Performed by the Bureau

4.1. Nature of Tests

Apparatus submitted for test is first examined as to its conformity with the specifications concerning quality and workmanship, design, markings, outflow time, etc.

If the apparatus meets these requirements, it is calibrated either to ascertain whether the capacity is correct within the prescribed limits of error or to determine the correction for use in precise measurements.

4.2. Precision Stamp

If the results of the preliminary examination indicate a satisfactory conformity with the specifications, and the error in capacity is within the prescribed tolerance, the official precision stamp, consisting of the letters NBS and the year date, surrounded by a circle, is etched on the instrument as shown below:



4.3. Certificates and Reports of Capacity

Burets will be calibrated for at least five intervals, and if found to conform with the specified requirements will, in addition to the precision stamp, be marked with an NBS identification number as shown below:

NBS No. 1234
1958

A certificate will be issued giving the volumes delivered from the intervals tested.

5. Directions for Submitting Apparatus for Test

5.1. 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 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 otherwise requested, burets 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 the construction specifications. Delay and cost of transportation on apparatus not entitled to verification will thus be avoided.

Only in special cases will certificates of capacity be issued for apparatus other than burets. When the measurements depending upon the use of the volumetric glassware are required to be so precise that a statement to the effect that the apparatus is within the tolerances prescribed in this Circular does not give sufficiently exact information, certificates may be issued. The request for certificates, when required, should be made at the time the apparatus is submitted for test.

Reports of capacity may be issued under the following circumstances:

(a) For burets when the error in capacity is greater than, but not more than twice, the tolerance.

(b) When certificates are requested for other apparatus, and the error in capacity exceeds, but is not more than twice, the prescribed tolerance.

(c) For special types of volumetric apparatus not covered by the specifications in this Circular.

When a report of capacity is issued, a number is applied as shown above, but the precision stamp is omitted.

4.4. Special Tests

The Bureau will gladly cooperate with scientific investigators, manufacturers of apparatus, and others in the calibration of precision volumetric apparatus not covered by the specifications in this Circular, as far as the regular work of the Bureau will permit. Tests of instruments not included in the current fee schedules should be arranged for by correspondence before shipment of the apparatus; the application should state fully the purpose for which the apparatus is to be used, the need for the test, and the precision desired.

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.

5.2. Shipping Directions

The apparatus should be securely packed in cases or packages which will not be broken in transportation and which may be used in returning the tested material to the owner. 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.

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".

After the material is received at the Bureau, the shipper will be notified of the test number assigned. This number should be mentioned in any correspondence pertaining to the test.

5.3. Breakage

A considerable number of pieces of glassware are received broken, either on account of improper packing or rough treatment in transportation, or both. Some are broken in return shipment. A small percentage is broken in the testing laboratory.

There is no legal way in which the Bureau can make reimbursement for breakages of this kind. It is therefore a matter of necessity, and not of choice, that the Bureau makes those who send apparatus for test assume all the risks involved.

Under the circumstances, all that the Bureau can do is to make every effort to reduce such breakage to the absolute minimum.

5.4. Address

Articles and communications should be addressed, "National Bureau of Standards, Washington 25, D.C."; delays incident to other forms of addresses will thus be avoided. Articles delivered personally or by messenger should be left at the receiving office of the Bureau, and should be accompanied by a written request for test.

5.5. Remittances

Payment of test fee should be made promptly upon receipt of bill. Remittances may be made by money order or check drawn to the order of the "National Bureau of Standards".

Copies of the current Test Fee Schedules may be obtained from the Bureau upon request.

6. Appendix

Tables 14 and 16 are reprints of tables 38 and 43 from NBS Circular 19, 6th edition, Standard Density and Volumetric Tables. They are included in this Circular for the convenience of users of volumetric glassware. Tables 15 and 17 have been added because of the increasing popularity of borosilicate glass apparatus.

6.1. Temperature Corrections for Volumetric Solutions

Table 14 gives the correction to various observed volumes of water, measured at the designated temperatures, to give the volume at the standard

temperature, 20° C. Conversely, by subtracting the corrections from the volume desired at 20° C, the volume that must be measured at the designated temperatures in order to give the desired volume at 20° C will be obtained. It is assumed that the volumes are measured in glass apparatus having a coefficient of cubical expansion of 0.000-025/° C. The table is applicable to dilute aqueous solutions having the same coefficient of expansion as water.

Table 15 gives temperature corrections for water, when measured in borosilicate glass apparatus having a coefficient of cubical expansion of 0.000010/° C.

TABLE 14. *Temperature corrections for water*

Measured in soft glass apparatus having a coefficient of cubical expansion of 0.00025/°C

Temperature of measurement	Capacity of apparatus in milliliters at 20° C						
	2,000	1,000	500	400	300	250	150
	Correction in milliliters to give volume of water at 20° C						
° C							
15.....	+1.54	+0.77	+0.38	+0.31	+0.23	+0.19	+0.12
16.....	+1.28	+0.64	+0.32	+0.26	+0.19	+0.16	+0.10
17.....	+0.99	+0.50	+0.25	+0.20	+0.15	+0.12	+0.07
18.....	+0.68	+0.34	+0.17	+0.14	+0.10	+0.08	+0.05
19.....	+0.35	+0.18	+0.09	+0.07	+0.05	+0.04	+0.03
21.....	-0.37	-0.18	-0.09	-0.07	-0.06	-0.05	-0.03
22.....	-0.77	-0.38	-0.19	-0.15	-0.12	-0.10	-0.06
23.....	-1.18	-0.59	-0.30	-0.24	-0.18	-0.15	-0.09
24.....	-1.61	-0.81	-0.40	-0.32	-0.24	-0.20	-0.12
25.....	-2.07	-1.03	-0.52	-0.41	-0.31	-0.26	-0.15
26.....	-2.54	-1.27	-0.64	-0.51	-0.38	-0.32	-0.19
27.....	-3.03	-1.52	-0.76	-0.61	-0.46	-0.38	-0.23
28.....	-3.55	-1.77	-0.89	-0.71	-0.53	-0.44	-0.27
29.....	-4.08	-2.04	-1.02	-0.82	-0.61	-0.51	-0.31
30.....	-4.62	-2.31	-1.16	-0.92	-0.69	-0.58	-0.35

TABLE 15. *Temperature corrections for water*

Measured in borosilicate glass apparatus having a coefficient of cubical expansion of 0.000010/°C

Temperature of measurement	Capacity of apparatus in milliliters at 20° C						
	2,000	1,000	500	400	300	250	150
	Correction in milliliters to give volume of water at 20° C						
° C							
15.....	+1.69	+0.84	+0.42	+0.34	+0.25	+0.21	+0.13
16.....	+1.39	+0.70	+0.35	+0.28	+0.21	+0.17	+0.10
17.....	+1.08	+0.54	+0.27	+0.22	+0.16	+0.13	+0.08
18.....	+0.74	+0.37	+0.19	+0.15	+0.11	+0.09	+0.06
19.....	+0.38	+0.19	+0.10	+0.08	+0.06	+0.05	+0.03
21.....	-0.40	-0.20	-0.10	-0.08	-0.06	-0.05	-0.03
22.....	-0.83	-0.41	-0.21	-0.17	-0.12	-0.10	-0.06
23.....	-1.27	-0.64	-0.32	-0.25	-0.19	-0.16	-0.10
24.....	-1.73	-0.87	-0.43	-0.35	-0.26	-0.22	-0.13
25.....	-2.22	-1.11	-0.55	-0.44	-0.33	-0.28	-0.17
26.....	-2.72	-1.36	-0.68	-0.54	-0.41	-0.34	-0.20
27.....	-3.24	-1.62	-0.81	-0.65	-0.49	-0.41	-0.24
28.....	-3.79	-1.89	-0.95	-0.76	-0.57	-0.47	-0.28
29.....	-4.34	-2.17	-1.09	-0.87	-0.65	-0.54	-0.33
30.....	-4.92	-2.46	-1.23	-0.98	-0.74	-0.62	-0.37

In using the above tables to correct the volume of certain standard solutions to 20° C, more accurate results will be obtained if the numerical values of the corrections are increased by the percentages given below:

Solution	Normality		
	N	N/2	N/10
HNO ₃	50	25	6
H ₂ SO ₄	45	25	5
NaOH.....	40	25	5
KOH.....	40	20	4

6.2. Tables of Corrections for Determining the True Capacities of Glass Vessels From the Weight of Water in Air

Table 16 gives, for a nominal capacity of 100 ml and observed temperatures from 15° C to 32.9° C, the amounts to be added to the apparent weight in grams (in air against brass weights) of the water contained in or delivered by a glass vessel to give the capacity in milliliters at 20° C. It is calculated on the following data assumed as approximating ordinary conditions: Observed barometric pressure—760 mm; relative humidity—50 percent; coefficient of expansion of glass—0.000025/° C.

Example of use of table. Determination of capacity of glass volumetric flask marked "To contain 100 ml at 20° C".

Apparent weight of water at the observed temperature, 24.5° C ..	99.615 g
From table 16, correction.....	+0.372
Actual capacity at 20° C.....	99.987 ml

For capacities other than 100 ml, the corrections given must be multiplied by the appropriate

factor. For example, the correction for 500 ml would be five times the correction for 100 ml. For borosilicate glass having a coefficient of cubical expansion of approximately 0.000010/° C Table 17 should be used.

6.3. Change in Capacity Caused by Change in Temperature

Having determined the capacity at 20° C, if it is desired to know what the capacity of the same vessel will be at another temperature, the following formula may be used:

$$V_t = V_{20}[1 + \alpha(t - 20)],$$

where

V_t = capacity at t ° C,
 V_{20} = capacity at 20° C,
 α = coefficient of cubical expansion of material which the instrument is made. (For soft glass 0.000025/° C)

This formula is applicable to instruments measures made of any material of which the coefficient of cubical expansion is known. A few materials commonly used in volumetric apparatus and their approximate coefficients are:

	Coefficient of cubical expansion ⁴ per deg C
Soft glass.....	0.000025
Pyrex or KG-33.....	.000010
Copper.....	.000050
Brass.....	.000054
Steel.....	.000035
Stainless steel.....	.000031

⁴ Coefficients shown are averages, but are sufficiently accurate for volumetric determinations.

TABLE 16. Table of corrections for determining the true capacities of glass vessels from the weight of water in air (Soft glass, coefficient of cubical expansion 0.000025/° C)

Temperature ° C	Tenths of degrees									
	0	1	2	3	4	5	6	7	8	9
15.....	0.207	0.208	0.210	0.211	0.212	0.213	0.215	0.216	0.217	0.219
16.....	.220	.221	.223	.224	.225	.227	.228	.230	.231	.232
17.....	.234	.235	.237	.238	.240	.241	.243	.244	.246	.247
18.....	.249	.250	.252	.253	.255	.257	.258	.260	.261	.263
19.....	.265	.266	.268	.270	.272	.273	.275	.277	.278	.280
20.....	.282	.284	.285	.287	.289	.291	.293	.294	.296	.298
21.....	.300	.302	.304	.306	.308	.310	.312	.314	.315	.317
22.....	.319	.321	.323	.325	.327	.329	.331	.333	.336	.338
23.....	.340	.342	.344	.346	.348	.350	.352	.354	.357	.359
24.....	.361	.363	.365	.368	.370	.372	.374	.376	.379	.381
25.....	.383	.386	.388	.390	.392	.395	.397	.399	.402	.404
26.....	.406	.409	.411	.414	.416	.418	.421	.423	.426	.428
27.....	.431	.433	.436	.438	.440	.443	.446	.448	.451	.453
28.....	.456	.458	.461	.463	.466	.469	.471	.474	.476	.479
29.....	.482	.484	.487	.490	.492	.495	.498	.501	.503	.506
30.....	.509	.511	.514	.517	.520	.522	.525	.528	.531	.534
31.....	.536	.539	.542	.545	.548	.551	.554	.556	.559	.562
32.....	.565	.568	.571	.574	.577	.580	.583	.586	.589	.592

TABLE 17. Table of corrections for determining the true capacities of glass vessels from the weight of water in air

(Borosilicate glass, coefficient of cubical expansion 0.000010/° C)

Indicated capacity 100 ml

Temperature ° C	Tenths of degrees									
	0	1	2	3	4	5	6	7	8	9
15	0.200	0.201	0.202	0.204	0.205	0.207	0.208	0.210	0.211	0.212
16	.214	.215	.217	.218	.220	.222	.223	.225	.226	.228
17	.229	.231	.232	.234	.236	.237	.239	.241	.242	.244
18	.246	.247	.249	.251	.253	.254	.256	.258	.260	.261
19	.263	.265	.267	.269	.271	.272	.274	.276	.278	.280
20	.282	.284	.286	.288	.290	.292	.294	.296	.298	.300
21	.302	.304	.306	.308	.310	.312	.314	.316	.318	.320
22	.322	.324	.327	.329	.331	.333	.335	.338	.340	.342
23	.344	.346	.349	.351	.353	.355	.358	.360	.362	.365
24	.367	.369	.372	.374	.376	.379	.381	.383	.386	.388
25	.391	.393	.396	.398	.400	.403	.405	.408	.410	.413
26	.415	.418	.420	.423	.426	.428	.431	.433	.436	.438
27	.441	.444	.446	.449	.452	.454	.457	.460	.462	.465
28	.468	.470	.473	.476	.479	.481	.484	.487	.490	.492
29	.495	.498	.501	.504	.506	.509	.512	.515	.518	.521
30	.524	.526	.529	.532	.535	.538	.541	.544	.547	.550
31	.553	.556	.559	.562	.565	.568	.571	.574	.577	.580
32	.583	.586	.589	.592	.595	.598	.602	.605	.608	.611

WASHINGTON, September 24, 1958.