TESTING OF GLASS VOLUMETRIC APPARATUS

Department of Commerce and Labor
BUREAU OF STANDARDS
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INTRODUCTION.

The first part of the following circular includes specifications to which apparatus must conform in order to be accepted by the Bureau for test, and mainly concerns the manufacture of such apparatus. The second part describes the manner in which tests will be made by the Bureau. With a few exceptions, the specifications agree with those which were recommended by a committee of the American Chemical Society, consisting of Prof. E. W. Morley, chairman; Prof. Arthur A. Noyes, Prof. Theodore W. Richards, and Mr. E. E. Ewell. The Bureau is also indebted to the manufacturers of chemical glassware for many valuable suggestions. In many cases the regulations of the Kaiserliche Normal Eichungs-Kommission of Germany have been adopted without appreciable change. In other instances, notably in the outflow time of burettes, important changes have been made. Experiments made at the Bureau showed that the accuracy in the use of burettes was considerably increased by lengthening the time of outflow and decreasing the period of subsequent waiting so that the sum of the two would not exceed that usually specified by the German regulations.

A bulletin giving the results of the experiments made and describing in detail the methods and apparatus used in the work of testing will be published in the near future.

I. SPECIFICATIONS FOR GLASS VOLUMETRIC APPARATUS WHICH WILL BE RECEIVED FOR TEST.

1. The Bureau will receive for test apparatus of the following varieties: Flasks, pipettes without subdivisions, pipettes with subdivisions, graduated cylinders, and burettes. Pipettes without subdivisions in which the lower limit of volume is a second mark will be accepted only when accompanied by a written order of the person who is to use them.

2. The cross section of such apparatus must be circular throughout, and portions having different diameters must merge into each other so gradually that there will be no hindrance to uniform drainage. Apparatus which is manifestly fragile or otherwise defective in construction will be rejected.

3. The parts of apparatus on which graduation marks are placed must be cylindrical, of uniform thickness, transparent, and free from striæ.

4. Each piece of apparatus must be marked with a permanent identification number, and stoppers, stopcocks, and detachable burette tips must be marked with numbers corresponding to those placed on the apparatus. Stoppers and stopcocks must not permit leakage.

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*The first edition of this circular was issued November 1, 1904; this edition is a revised reprint, to which is added a schedule of fees.

5. The true liter will be employed as the unit of capacity, or its submultiple, the one one-thousandth liter, called the cubic centimeter.* The liter is defined as the volume of 1 kilogram of water at 4° C.

6. Twenty degrees Centigrade is accepted by the Bureau as the standard temperature. An extra charge will be made for the verification of apparatus which is standard at any other temperature. Every piece of apparatus must be plainly marked with its capacity in liters or cubic centimeters and the temperature for which it is graduated.

7. The width of graduation marks must not exceed one-tenth of the distance between adjacent subdivisions nor in any case be more than 0.2 mm. *All* marks must be permanent and they may be filled with coloring matter.

8. The graduation marks on burettes, on pipettes with subdivisions, and on graduated cylinders must go at least halfway around.

FLASKS.

9. *Flasks*:

(*a*) At the point where the graduation mark is placed the neck of a flask must have an internal diameter not greater than the maximum nor less than the minimum diameter given in the following table:

Capacity of flask ..... liters..		2	1	0.5	0.25	0.2	0.1	0.05	0.025	0.01
Diameter of flask.	Maximum ....millimeters..	25	20	18	15	13	12	10	8	8
	Minimum .....do.....	18	14	12	10	9	8	6	6	6

(*b*) The graduation mark must be placed not less than 6 cm from the upper end and not less than 2 cm from the lower end of the neck of a flask larger than 100 cc, and not less than 3 cm from the upper end or 1 cm from the lower end of the neck of a flask not larger than 100 cc. The graduation mark must extend entirely around the neck.

(*c*) The bottom of a flask must be slightly reentrant, and the flask must be of such form that drainage can take place from the whole interior surface at the same time. The neck of a flask must be perpendicular to a plane tangent to the bottom. The flask must stand solidly when placed on a horizontal plane.

(*d*) All flasks must be marked either "to contain," or "to deliver," but must not be graduated both to contain and to deliver.

PIPETTES.

10. *Pipettes without subdivisions*:

(*a*) Pipettes without subdivisions may be made of any desired capacity from 1 cc to 200 cc.

(*b*) The suction tube of a pipette must be at least 13 cm in length, and the delivery tube must be at least 3 cm and at most 32 cm in length.

(*c*) The internal diameter of the suction and delivery tubes of pipettes without subdivisions must not be more than 8 mm for 200 cc pipettes, and not more than 6 mm for smaller sizes and in no case less than 2 mm.

*The cubic centimeter is not exactly the one-thousandth part of the liter, but for all practical purposes they may be considered identical. In accordance with common practice in the United States, the abbreviation "cc" is used for "cubic centimeter" in this circular instead of the official abbreviation "cm^3."

(*d*) The graduation on the suction tube of a pipette must be at least 10 cm from the upper end of the tube and at least 1 cm from the bulb. If the lower limit of volume is a second mark, this must be at least 3 cm from the bulb and as far from the tip. The graduation must extend entirely around the tube.

11. *Pipettes with subdivisions:*

(*a*) Pipettes with subdivisions may have any capacity from 1 cc to 100 cc.

(*b*) The uppermost mark of such pipettes must be at least 10 cm from the upper end of the tube, and the lowest mark must be at least 4 cm from the lower end.

12. *Delivery of pipettes:*

(*a*) The delivery orifice of all pipettes must be of such a size that the free outflow shall last not more than one minute and not less than—

Twelve seconds if the capacity is 10 cc or less; fifteen seconds if the capacity is more than 10 cc but not more than 50 cc; twenty seconds if the capacity is more than 50 cc but not more than 100 cc; thirty seconds if the capacity is more than 100 cc.

(*b*) The delivery time must be marked on pipettes.

BURETTES.

13. *Burettes:*

(*a*) Burettes may have any capacity from 5 cc to 100 cc. Only those forms which empty through a tip at the bottom will be admitted for verification. Mohr burettes must be furnished with tips.

(*b*) The length graduated must not exceed 65 cm, and the distance between adjacent graduation marks must not be less than 1 millimeter.

(*c*) The rate of outflow of burettes must be restricted by the size of the tip, and the time required for emptying the entire graduated interval must not exceed three minutes, nor be less than the following for the respective lengths:

Length Graduated	Time of Outflow	Length Graduated	Time of Outflow
Centimeters	*Seconds*	*Centimeters*	*Seconds*
65	140	35	60
60	120	30	50
55	105	25	40
50	90	20	35
45	80	15	30
40	70		

(*d*) The time required for emptying the graduated interval must be marked on the burette.

CYLINDERS WITH SUBDIVISIONS.

14. *Cylinders:*

(*a*) Cylinders with subdivisions may have any capacity from 5 cc to 2,000 cc.

(*b*) Cylinders must be graduated and marked "to contain."

(*c*) Every tenth graduation mark on cylinders must extend entirely around the cylinder.

(*d*) The ratio of the graduated length of the cylinder to the internal diameter must not be less than five to one.

LIMITS OF ERROR WHICH WILL BE REGARDED AS ALLOWABLE.

15. The errors of flasks must not exceed the following limits:

Capacity	Limit of Error	
	If to Contain	If to Deliver
cc	*cc*	*cc*
2,000	0.5	1
1,000	0.3	0.5
500	0.15	0.3
250	0.1	0.2
200	0.1	0.2
100	0.08	0.15
50	0.05	0.1
25	0.03	0.05
10	0.01	0.03

Flasks of other capacities shall not have errors greater than those of the next smaller sizes here given.

16. The errors of pipettes without subdivisions must not exceed the following limits:

Capacity	Limit of Error
	cc
1 to 2 cc	0.01
More than 2 and not more than 10 cc	0.02
More than 10 and not more than 30 cc	0.03
More than 30 and not more than 75 cc	0.05
More than 75 and not more than 200 cc	0.1

17. (*a*) The errors of total capacity or of any interval of burettes and of pipettes with subdivisions must not exceed the following limits:

Capacity	Limit of Error	
	Burettes	Pipettes
	cc	*cc*
1 to 2 cc		0.02
More than 2 and not more than 10 cc	0.02	0.04
More than 10 and not more than 30 cc	0.03	0.06
More than 30 and not more than 50 cc	0.05	0.1
More than 50 and not more than 100 cc	0.1	0.2

(*b*) The error in the volume indicated by ten consecutive smallest divisions must on no part of the scale exceed one-fourth of the volume indicated by the smallest subdivision. The error in the time marked on the burette for the delivery of total capacity must not exceed ten seconds.

18. The time of delivery marked on pipettes must not be in error by more than 10 per cent.

19. (*a*) The errors of maximum capacity or of any interval of graduated cylinders must not exceed the following limits :

Capacity	Limit of Error
	cc
5 to 30 cc	0.05
More than 30 and not more than 50 cc	0.1
More than 50 and not more than 100 cc	0.2
More than 100 and not more than 200 cc	0.5
More than 200 and not more than 500 cc	1
More than 500 and not more than 1,000 cc	2
More than 1,000 cc	5

(*b*) The error of the volume indicated by ten consecutive smallest subdivisions must on no part of the scale exceed the following limits: 0.1 cc when the smallest divisions indicate 0.1 or 0.2 cc; 0.2 cc when the smallest divisions indicate 0.5 or 1 cc; 0.4 cc when the smallest divisions indicate 2 cc; 1 cc when the smallest divisions indicate 5 or 10 cc; 2 cc when the smallest divisions indicate 20 cc.

II. REGULATIONS FOR TESTING VOLUMETRIC APPARATUS.

20. *Methods of testing:*

(*a*) Apparatus will be tested by means of water, and the capacity will, therefore, be expressed as the volume of water the vessel will contain or will deliver, as the case may be, when at its standard temperature.*

(*b*) In filling flasks to deliver, the entire neck of the flask will be wetted.

(*c*) In flasks and graduated cylinders to contain, the vessel will be dry above the meniscus when read, except for the wetting necessary to secure a normal meniscus (see footnote).

(*d*) In all apparatus where the volume is limited by a meniscus, the reading will be made where the wall of the apparatus is cut by a plane perpendicular to the axis of the apparatus and tangent to the meniscus at its lowest point.

(*e*) In filling a pipette the meniscus will be brought to the mark while the tip is in contact with a wetted surface.

(*f*) Pipettes will be held vertical during free outflow, and allowed to drain fifteen seconds, after which the suspended drop will be removed by contact with a wetted surface and included in the delivered amount.

(*g*) Burettes will be filled to the zero mark, the interval under test emptied by free outflow, and the reading taken after ten seconds.†

(*h*) The natural period of outflow of the burette will be determined by the Bureau.

(*i*) For stopcock burettes this will be considered the time required to deliver the entire nominal volume with the stopcock fully open and the outflow free at the jet.

* When apparatus is used for other liquids than water, the capacity may differ, the amount of difference depending on the surface energy and viscosity of the liquid measured. This should be particularly borne in mind in the use of burettes and pipettes, where the highest accuracy is expected.

The apparatus must be thoroughly cleaned, especially of oil or grease, the slightest trace of which changes the surface energy of the water and glass, thus producing a change in capacity either by distorting the meniscus or by altering the residue of water in vessels used to deliver.

Freedom from oil may be recognized by the continuous film of water adhering to the glass until removed by evaporation. The apparatus must be wet a short distance above the meniscus in order that the meniscus may assume its normal shape.

† The capacity of a burette depends on the rate of emptying, and therefore, in use, the burette should be emptied at a rate corresponding to the time of emptying marked on the burette by the manufacturer, and used in its verification.

(*j*) For Mohr burettes the time will be determined using the tip attached by rubber tubing of such a length as to bring the tip 6 cm below the extreme end of the burette.*

(*k*) Graduated cylinders will be tested by filling to the mark under test, keeping the vessel dry above except to secure a normal meniscus, as explained before.

(*l*) Flasks which are graduated to deliver will, after emptying, be held for one minute in an inclined position in which both the bottom and the parts near the neck are equally permitted to drain, at the end of which time the suspended drop will be removed, and included in the delivered amount.

OFFICIAL STAMP.

21. *Stamp:*

(*a*) Apparatus which fulfills the requirements noted on pages 1–5 will receive the official stamp of the Bureau.

(*b*) The stamp consists of the letters U. S. and the last two figures of the year date, the whole being surrounded by a circle, thus:

(*c*) On flasks the stamp will be placed on the neck immediately above the mark; on graduated cylinders, immediately above the upper mark; on pipettes for delivering a single volume, on the bulb and tip of the pipette; on burettes and graduated pipettes, immediately above the upper mark and on the tip.

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

CERTIFICATION OF CORRECTIONS.

22. *Certification:*

(*a*) The Bureau will, when desired, determine the corrections to apparatus which fulfills the requirements noted on pages 1–5.

(*b*) The test number will be marked on the apparatus and a certificate will be issued stating the correction to be added to the reading to give the true capacity.

(*c*) When certificates for apparatus of variable capacity are requested, the points at which the corrections are desired should always be specified.

(*d*) When desired, the Bureau will determine the correction to graduated cylinders when used to deliver.

(*e*) Special forms of apparatus will be received for test only by special arrangement.

CERTIFICATE.

The certificate furnished by the Bureau of Standards will contain the following data: (*a*) Description of apparatus; (*b*) Bureau of Standards test number when allowed; (*c*) name of party for whom apparatus is tested; (*d*) conditions of the test; (*e*) corrected value of each space tested; (*f*) date of certification; (*g*) seal of the Bureau and the signature of the Director.

*When the Mohr burette is set up in the ordinary way—that is, with the pinchcock and rubber tubing—the capacity may be affected by the stretch of the rubber tubing, caused by the greater pressure when the burette is full than when empty, and making the capacity appear greater than it really is.

SCHEDULE OF FEES FOR VOLUMETRIC APPARATUS.

SCHEDULE 23.

I. Pipettes Without Subdivisions and Flasks, "To Contain," of 25, 50, 100, 200 cc Capacity.

(*a*) For testing and stamping similar pieces, 10 or less, each	$0.25
(*b*) For testing and stamping similar pieces in excess of 10, each additional piece	.20
(*c*) For testing, stamping, and certificate of corrections for similar pieces, 10 or less, each	.35
(*d*) For testing, stamping, and certificate of corrections for similar pieces, in excess of 10, each additional piece	.25

II. Flasks of 500, 1,000, 2,000 cc Capacity and all Flasks Graduated "To Deliver" the Indicated Volume.

(*e*) For testing and stamping similar pieces, 10 or less, each	.30
(*f*) For testing and stamping similar pieces in excess of 10, each additional piece	.25
(*g*) For testing, stamping, and certificate of corrections for similar pieces, 10 or less, each	.40
(*h*) For testing, stamping, and certificate of corrections for similar pieces, in excess of 10, each additional piece	.30

III. Burettes and Pipettes with Subdivisions.

(*i*) For testing and stamping similar pieces, 10 or less, each	.75
(*k*) For testing and stamping similar pieces in excess of 10, each additional piece	.60
(*l*) For testing, stamping, and certificate of corrections at 5 points for similar pieces, 10 or less, each	1.25
(*m*) For testing, stamping, and certificate of corrections at 5 points for similar pieces in excess of 10, each additional piece	1.00
(*n*) For testing, stamping, and certificate of corrections at more than 5 points, each additional point	.15

IV. Graduated Cylinders.

(*o*) For testing and stamping 10 or less similar pieces, each	.50
(*p*) For testing and stamping similar pieces in excess of 10, each additional piece	.40
(*q*) For testing, stamping, and certificate of corrections at 5 points, 10 or less similar pieces, each	.75
(*r*) For testing, stamping, and certificate of corrections at 5 points, each similar piece in excess of 10	.60
(*s*) For testing, stamping, and certificate of corrections at more than 5 points, each additional point	.15
(*t*) Additional charge for certifying volume delivered by total capacity, each	.20

V. For Testing any other Temperature than 20° C.

(*u*) For testing at any temperature other than 20° C. between 15° and 30° C., additional charge for each piece	.20

SPECIAL DIRECTIONS.

When submitting apparatus for test, the nature of the test required should always be specified.

The word "test" will be construed to mean a determination of the corrections to the capacity and also an examination of the apparatus as to conformity with the Bureau regulations in regard to construction and marking. It also implies stamping, in accordance with section 21 of this circular.

The word "certify" will be understood to mean test as explained above, and in addition a certificate giving the corrections at not more than five points, as per section 22.

Requests for tests other than the above should be accompanied by a complete statement as to how, and for what, the apparatus is to be used, the number of points to be tested, and any other information liable to affect the test.

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, and this number should be referred to in the correspondence concerning the test.

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

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

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

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

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

S. W. Stratton,
Director.

Approved:
V. H. Metcalf,
Secretary.