SPECIFICATIONS FOR BABCOCK AND CERTAIN OTHER VOLUMETRIC GLASSWARE AND METHODS FOR CONFORMANCE DETERMINATIONS

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Methods used currently in New York State for determining conformance with specifications of Babcock and Gerber volumetric glassware (*1*) and glassware for certain quantitative bacterial determinations (*2*) for dairy products have been described. The presentation of the procedures herein does not mean that other suitable ones may not be used satisfactorily. Some of the procedures have been used many years, while others, adaptable to Gerber glassware and to bacteriological transfer pipettes, are relatively new. The information herein may be useful to agencies responsible officially or otherwise for glassware conformance testing methods, and particularly to those in areas not yet requiring the use of certain tested and branded volumetric glassware.

The New York State statute (*3*) provides essentially that whenever determinations of the fat content of milk and/or cream are used for certain specific purposes, the glassware used in the volumetric measurements of the test charge and of the separated fat shall be graduated accurately. It also provides similarly for pipettes and other measuring instruments used when determining for certain purposes the bacterial counts of milk and/or cream as delivered by producers at receiving plants.

When tested for conformance with specifications and found satisfactory, each piece of glassware is indelibly and unmistakably etched by sand blasting or otherwise with a distinctive identifying mark, "N. Y." Any piece which is tested and found not to conform is returned unmarked to the owner. For certain metal syringes (*2*) with a set screw to fix maximal movement of plunger, a corrosive dye is used to etch the flat surface of a drop of solder securely covering the set screw. The set screw cannot be reset without showing that the solder seal has been broken and the marking partially defaced.

The sand blast equipment consists of an air compressor, motor operated to provide about 40 (30-50) lbs./sq. in. pressure, and a reservoir of sand (No. X-1-PBW/C+H Cover, Ottawa Silica Company, Ottawa, Ill.) with valves suitably located to feed the sand by gravity into the airline near the blast outlet end. By pressing a foot-control valve, air blows sand on to a stencil fitted securely around each piece of glass to be marked. Prolonged exposure at unusually high pressures causes excessive etching of exposed areas.

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The reader is referred to published records for specications for Babcock glassware (*1,2,3,4,5*). Circular 632 by the Department of Agriculture and Markets (*1*) listed specifications, prepared prior to this date, for Gerber glassware and includes a copy of the New York State statute providing for the identification of tested and approved glassware. The British Standards Institution (in 1935 and in tentative announcement 1951) included specifications (*6*) for certain pieces of Gerber glassware which differ slightly from those recognized in New York State (*1*). Recent amendments have been made to the 1951 specifications. Also, the

Netherlands Standards Commission revised their specifications (7) for the Gerber pipette, effective October 1, 1951.

The type of Babcock and Gerber glassware to be considered are:

Babcock Fat Test Method

Pipette *to contain* 17.6 ml. ±0.05 ml. of water at 20°C.

Milk Test Bottle, 8%, 18g., grad. in 0.1 percents.

Cream Test Bottle, 50%, 9 g., short (6″), grad. in 0.5 percents.

Cream Test Bottle, 50%, 9 g., long (9″), grad. in 0.5 percents.

Cream Test Bottle, 50%, 18 g., long (9″), grad. in 0.5 percents.

Gerber Fat Test Method

Pipette, *to contain* 10.77 ml.[a] ± 0.03 ml. of water at 20°C.

Milk Test Bottle, 8%, 11.006 g., grad. in 0.1 percents.

Cream Test Bottle, 50%, 5 g., grad. in 0.5 percents.

[a]Revised by Netherlands Standards Commission (9), effective October 1, 1951.

GENERAL PROCEDURE

Since specifications for pipettes and other glassware are given in terms of "to contain" or "to deliver" water (H_2O) at 20° C., it seems essential that the exact contents of Master Pipettes, etc., previously checked with H_2O to determine conformance and unmistakably marked for such identification, be used repeatedly for initial transfers for conformance determinations on each lot of from 10 to 20 pieces of untested glassware. Because mercury (Hg) flows freely from surfaces of glass, because it contains no undissolved air, and because air pockets at glass surfaces are readily noticeable and can easily be removed by gently tapping the glassware, Hg, instead of H_2O, is used routinely for volume conformance determinations.

Certain general precautions, mostly related to handling Hg, follow. The room, the Hg and all apparatus to be used is kept at, and the untested glassware stored for sufficient time before use or testing should come to room temperature, usually about 20 - 24° C. The Hg to be used and both certified and untested glassware should be clean, dry and dust free. Trays are used, as needed, to collect accidental Hg losses. (Hg vaporizes very slowly at room temperature and prolonged exposure to vapors may be a distinct health hazard.)

When calibrating, all air trapped while adding each charge of Hg is released from the column by gently tapping the glassware until the top of the column forms a well-rounded meniscus. All instruments and Hg are shielded from temperature changes caused by drafts, manual handling, etc. Use of apparatus or the wearing of personal adornments potentially amalgamizable is avoided.

Conformance observations in pipettes and in bottle necks may be made at the extreme top of the inverted meniscus of the Hg column or at such other place with reference to the calibration line as the examiner chooses, provided a uniform practice is followed. When examining a water meniscus in the Master Standards, it is always adjusted so that the extreme lowest part of the meniscus coincides with the middle of the graduation line.

BABCOCK AND GERBER MILK PIPETTES

A Babcock pipette *to contain* 17.6 ml. at 20°C. or a Gerber pipette *to contain* 10.77 ml. at 20°C. tested for accuracy and bearing certification thereof by the National Bureau of Standards, is used as a Master Pipette for determining the initial charge of Hg for each group of untested pipettes, as hereinafter directed. Since the amount of H_2O retained in the pipette will be determined in part by the shape of the tip and the total interior surface area, and since the volume of the meniscus depends in part on the bore of the suction tube at the graduation line, it is necessary that untested pipettes and the Master Pipette have practically the same dimensions. In the interest of accuracy, it is essential to know the exact content of the Master Pipette and to correct for any error observed either by marking the stem with a narrow paint line or by making an appropriate linear allowance when pipettes being tested do not hold the same amount as the Master Pipette.

To arrange a supply rack of untested pipettes with tip ends extending outward at left of operator is often convenient. With index finger of left hand placed securely over tip end, the Master Pipette (Babcock or Gerber) is filled with Hg exactly to graduation line. With index finger of right hand placed securely over mouth end, the pipette is tilted so that tip end is slightly above horizontal. A pipette to be tested is grasped with the left hand with index finger placed securely over tip end. The Hg charge is then gradually but completely transferred to the pipette in the left hand by inserting the portion below the bulb into the mouth end of the pipette. If the top of the Hg column is level with the graduation line, tolerance ±0.05 ml. in terms of equivalent length on stem, when pipette being tested is held vertically, the pipette is accepted and identified as described above.

The pipette in the left hand is then transferred to the right hand with the index finger placed securely over the mouth end, and is then tilted as before. Making certain that no Hg has been lost, from 10 to 20 pipettes are tested in succession with the same Hg charge. Before discarding the Hg charge so used, its

volume is redetermined by returning it to a Master Babcock pipette. If loss has occurred, all pipettes in the last batch examined are retested.

A rubber pad over the finger which closes the tip of the receiving pipette protects the finger and reduces the chances for Hg losses. To facilitate speed, pipettes are held nearly horizontal when making transfers.

BABCOCK TEST BOTTLES

Several methods for determining the accuracy of the graduations of Babcock test bottles appear in the Laboratory Manual of the Milk Industry Foundation (8). The Hg calibration method, as described therein, has been used in New York for more than 20 years. Use of the Nafis Tester (Figure 1) a special plunger-in-cylinder type device, made by Kimble Glass Company simplifies the testing procedure by making possible the repeated delivery of known amounts of Hg without loss into the graduated neck of successive test bottles within only a few seconds.

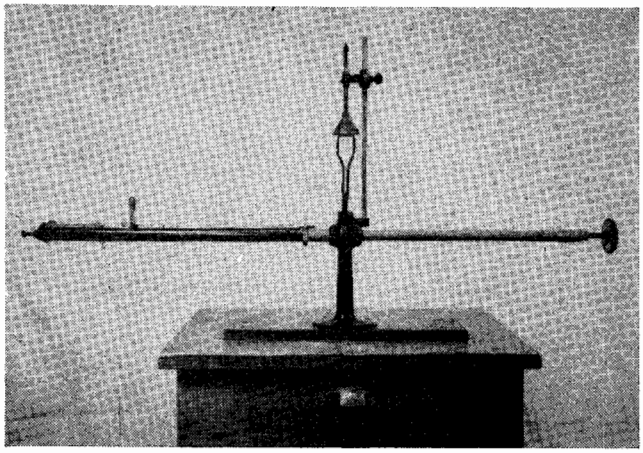

Figure 1. Nafis Tester with Inverted Babcock Milk Test Bottle in Position.

Before use, and periodically thereafter, the Nafis Tester is checked for plunger displacement of Hg using a calibrated Master Tube for Babcock milk test bottles (Figure 2). The volume displaced applies to the graduated portion only of the neck of the bottle. The accuracy of the displacement of the left-hand plunger is determined by forcing two successive 0.8-ml. charges of mercury into the Master Tube, each volume equivalent to that occupied by the milk fat from an 18-g charge of milk containing 4% milk fat. Before use, the tube is clamped in position as described below for the milk test bottle.

Master Tubes are required for checking displacements of the plunger when testing Babcock cream test bottles and the 1.0, 1.0 and 1.1, and the 2.2-ml. bacteriological transfer pipettes, described subsequently. The following description of operations is designed in part to enable official agencies to adapt the Nafis Tester to determining conformance of bacteriological transfer pipettes to specifications.

To determine the accuracy of the graduated portion in the neck of a Babcock milk test bottle, the bottle is first inverted over the Hg outlet above the cylinder. The Hg outlet, surrounded immediately below the orifice by a cushion-type fitting, is slightly recessed in the center of a disc, with a rim to prevent Hg loss. The bottle is seated securely over the outlet by clamping it in position from above.

At the right hand end of the metal cylinder, the plunger, with attached actuating rod threaded into the cylinder head, is manually operated to adjust the top of the Hg column level with the first graduation at the 8% line in the neck of the inverted bottle. At the left-hand end, the guide bar, to which an arm is attached for actuating another plunger, is manually operated to force a known amount of Hg in two successive charges into the graduated portion of the neck of the bottle. The guide bar has a check stop corresponding to the correct amount for forcing the Hg first to the 4% graduation, and a second check stop corresponding to the correct amount to continue forcing it to the 0% graduation.

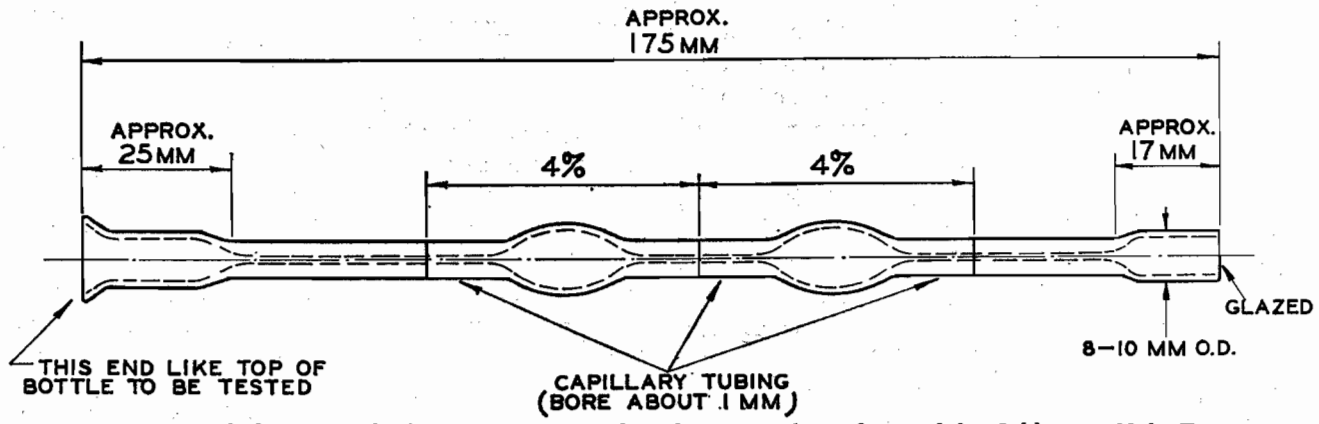

Figure 2. Certified Master Tube for Volume in Babcock Milk Test Bottles to be Used for Calibrating Nafis Tester. (Courtesy Drawing by Kimble Glass Company, Subsidary Owens, Illinois)

The accuracy of the graduations on Babcock cream test bottles is determined in a similar manner. The chief difference consists of rotating the guide bar so that the stops conform to the desired displacement volumes of Hg. A suitable Master Tube for Babcock cream test bottles is used for conformance determinations for the stops.

The amount of Hg required for each of the two successive charges in the Babcock test bottles, according to their design, is listed below:

Bottle	*Successive Charges*
Milk test, 8%, 16 gm.	0.8 ml.
Cream test, 50%, 9 gm.	2.5 ml.
Cream test, 50%, 18 gm.	5.0 ml.

The maximum error in the total graduation of any bottle or in any part thereof shall *not exceed* the volume of the smallest unit of the graduation on that type of bottle, as observed by the examiner when viewed at eye level with the graduation line.

GERBER TEST BOTTLES

The closed end of the tube, and in particular the small volume of the bulb above the highest graduation, in the Gerber type of bottles makes it impossible to use the Nafis Tester for determining the accuracy of the neck graduations. To make this determination, it is now customary to invert the 8%, 11.006-g., milk-test bottle and fill the closed bulb with Hg to the first graduation (the 8% line). Formerly a special glass cup with volume confidentially known to the manufacturer of the Gerber test bottles was then filled with Hg. A glass straight-edge was drawn across the top of the cup to remove any excess charge and 2 successive charges transferred to the Gerber bottle. Each charge consists of 0.5 ml. which, if the graduations are correct, should bring the top of the mercury column first to the 4% graduation level and then to the 0% graduation.

Formerly a similar procedure requiring a larger cup was used for determining conformance of the 50%, 5-g, Gerber cream-test bottles. The maximum error in the total graduation of any bottle or in any part thereof shall *not exceed* the volume of the smallest unit of the graduation on that type of bottle. It has been customary to interpret this requirement in the identical manner as described above for Babcock test bottles.

Because of the possible error when straight-edging the Hg in the special cups with previously undisclosed capacity (9), upon request by the senior author a precision calibrated funnel (Sketch XA-1956) was prepared by the Corning Glass Works, Corning, N.Y. (Figure 3). The specifications of 0.5 ml. at 20° C. for calibrating with two successive charges the volume in the neck of the milk test bottle was given the manufacturer first. Use of this apparatus was so satisfactory that arrangements were made to construct a similar device to deliver a 1.42-ml. charge of Hg for two successive additions to cream test bottles (9).

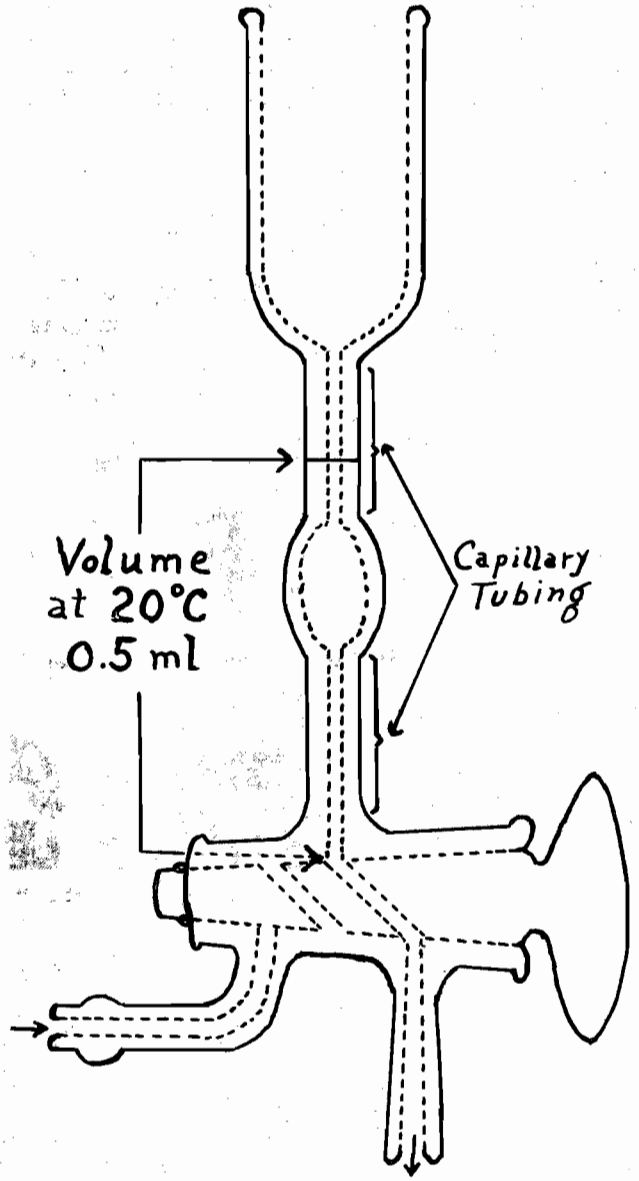

Figure 3. Certified Precision Calibrated Funnel for Measuring Test Portions of Mercury into Gerber Milk Test Bottles.

Originally the intent was to use a leveling bulb for the reserve Hg source, with outlet attached by tubing to intake opening on apparatus and with a screw-threaded column to support the Hg reservoir for close adjustment of the Hg level at the graduation line in the capillary tube. Instead of using this scheme, it was decided to reduce the size of the intake hole in the stopcock by inserting a wood plug through which was made a capillary opening about 1 mm. in diameter. This modification served to control the sudden inward flow of Hg satisfactorily without aid of a

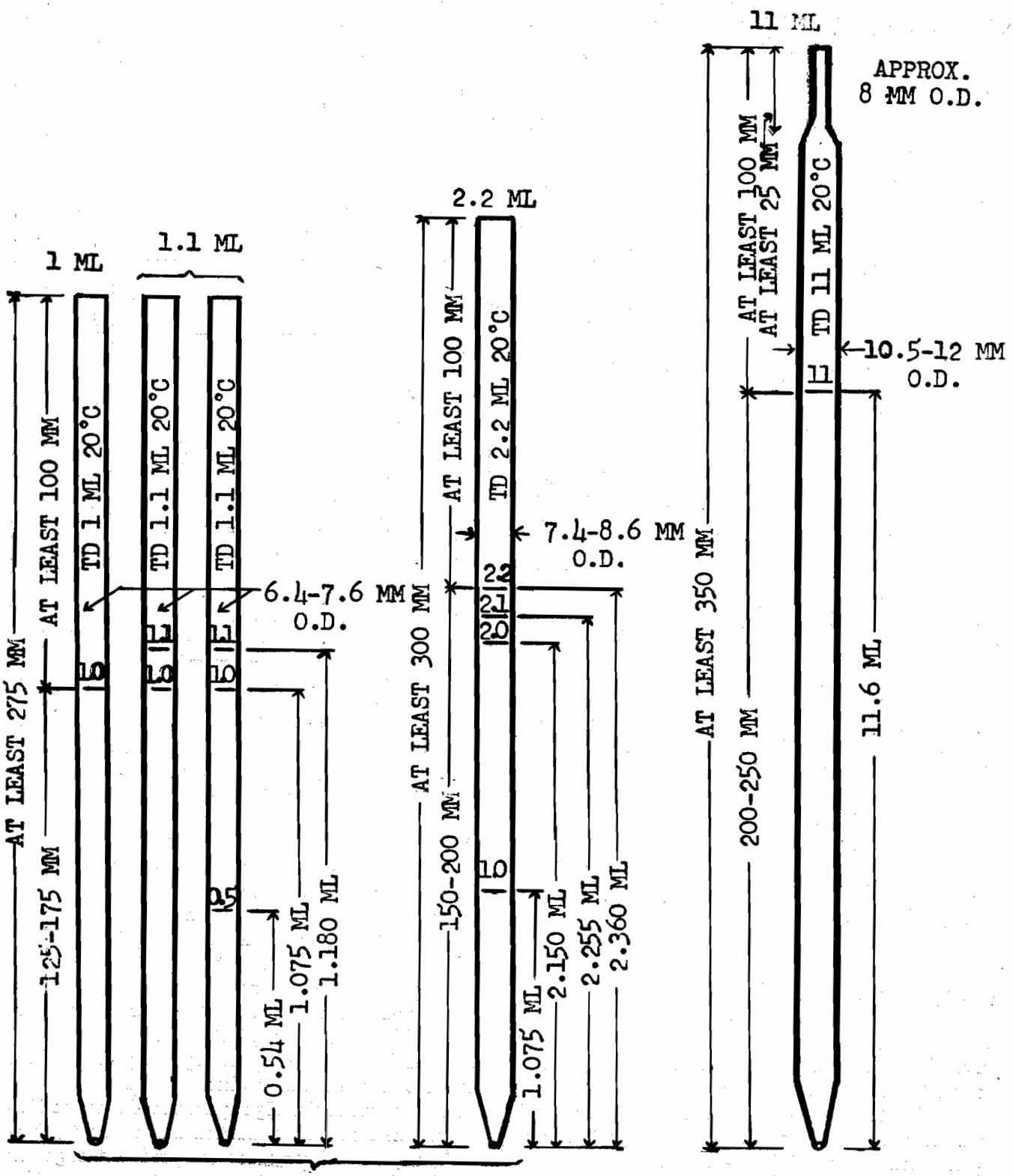

Figure 4. Bacteriological Transfer Pipettes. Delivery 1 ml; Delivery 1.1 ml in 0.1 and 1 ml amounts; Delivery 1.1 ml in 0.1, 0.5, 0.5 ml amounts; Delivery 2.2 ml successively in 0.1, 0.1, 1, and 1 ml. amounts; and Delivery 11 ml.

screw-threaded column. Obviously, before using the charge-measuring apparatus, it is necessary to fill the bulb in each bottle with Hg until it is level with the first graduation line.

In order that the Fibu key-plunger-type stoppers (No. 4285) may be adjusted with minimal chance of leakage, it is essential that the openings in the test bottles be uniformly round, with diameter range from 11.0-11.6 mm. It is also essential to limit necessary adjustment of the column to not over ± 0.1% graduation.

BACTERIOLOGICAL TRANSFER PIPETTES

Specifications for the 3 general types of bacteriological transfer pipettes; (*a*) the 1.0 ml. and 1.1 ml., (*b*) the 2.2 ml., and (*c*) the 11.0 ml., are given in Figure 4.

Conformance Determination of Bacteriological Transfer Pipettes

For determining conformance with linear specifications, several pipettes are arranged parallel with the side wall of a tray and the tray tilted so that the tips rest against the base wall (Figure 5). Trays are prepared in 3 different sizes, for use according to the style of pipette to be tested, with floor lines appropriately spaced parallel with the base to indicate tolerance limits as follows:

	Type of Pipette		
	1.0 ml. and 1.1 ml.	2.2 ml.	11.0 ml.
Total length at least, mm	275	300	350
Tip to line at ml.	1.0	2.2	11.6
Minimum, mm.	125	150	200
Maximum, mm.	175	200	250
Tip constriction length			
Minimum, mm.	10	10	20
Maximum, mm.	15	15	30

Determination of conformance with the above tolerance limits serves indirectly to assure conformance with bore tolerance measurements also.

For determining conformance to length from top of pipette to the graduations at 1.0 ml., 2.2 ml. and 11.0 ml., the tray is tilted in the opposite direction so that the mouth ends of the pipettes rest against the opposite end of the tray and parallel with the side walls. The line drawn across the floor of the tray 100 mm. from the top guides in determining whether the graduation on the pipette is less than the tolerance.

When the pipettes are in this position, the tip ends are exposed so that a cylindrical taper gauge (Figure 6), tested and marked by the calibrating agency at the appropriate diameter tolerance limits, is inserted in each pipette. The degree of insertion determines whether the tip of the pipette conforms with specifications. Other similar cylindrical taper gauges, as shown in Figure 7, may be improvised.

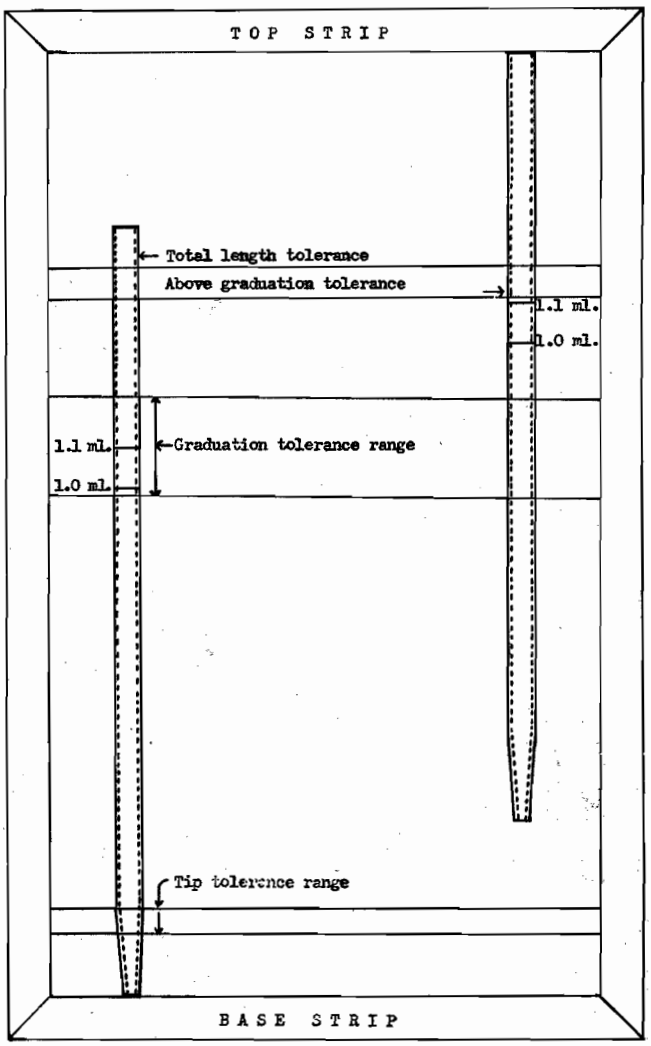

Figure 5. Tray for Linear Conformance Determinations with Specifications for Bacteriological Transfer Pipettes.

For determining volume conformance of the 1.0 ml., the 1.0 and 1.1 ml., and the 2.2-ml. pipette, the Nafis Tester is used. A special seat is inserted around the Hg outlet so that the mouth end of the clamped pipette will fit firmly against a cushioned area. Due to variable bores, each pipette is filled to the tip, and then by withdrawing the Hg to a prescribed bar stop, the graduation is checked for accuracy.

Since the volume of Hg required for filling the 11.0-ml. pipette from the graduation line to the tip is so large, the Nafis tester cannot be used. Although one could be designed for this purpose, it is now considered unnecessary because so few pipettes of this type are submitted for testing. The procedure now used is similar to that employed for testing the Babcock and Gerber milk pipettes, except that a small glass

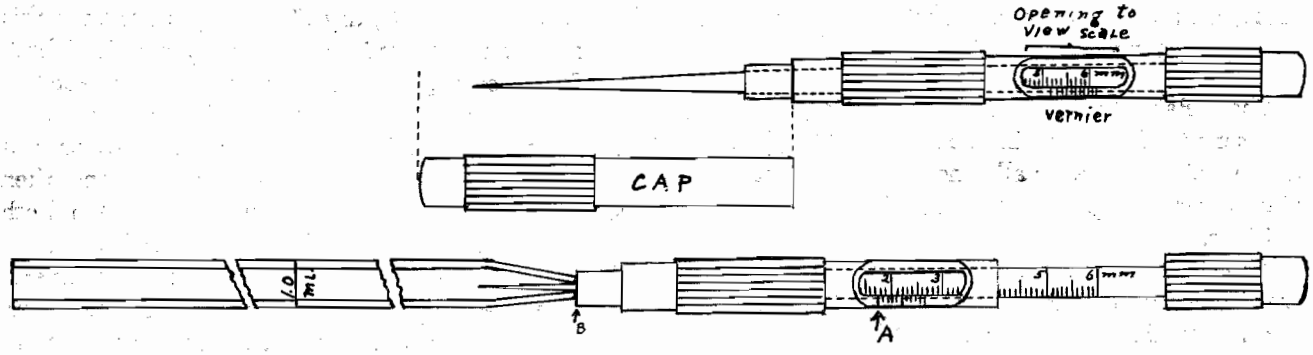

Figure 6. Telescoping Round Taper Gauge for Determining Diameter of Bore of Pipettes at Tip. In Lower section Needle Is Withdrawn to Minimal Tolerance Diameter of 1.75 mm. at A and 1.0 ml Pipette Tip Held Close to Shield Case at B. (Gauge available as Item 50-733 from E. Machlett & Son, 220 E. 23rd. St., New York 10, N.Y.; now a subsidiary of Fisher Scientific Co., New York.)

Figure 7. Improvised Round Taper Gauge, Marked at Range Limits.

funnel is inserted in the top of the 11.0-ml. pipette when transferring the Hg from one pipette to another.

Tolerances for bacteriological pipette graduations are as follows:

1.0 and 1.1 ml.	± 0.025 ml.	From tip to any graduation and between successive graduations
2.2 ml.	± 0.040 ml.	
11.0 ml.	± 0.200 ml.	

Testing the 0.01-ml. Capillary Pipette

Because of the small amount of Hg required and the small bore to be filled when testing the 0.01-ml. pipette shown in Figure 8, (for use with the direct microscopic method (2) for determining the bacterial content of milk and cream) special care is essential to avoid Hg losses. The 0.01-ml. pipette is tested for volume conformance by transferring to it from a standard tested pipette 0.1395 g. (approximately 0.1400 g.) of Hg at 20°C. The pipette has an overcapacity of 0.0004 ml. to allow for residual milk adhering within the bore when transferring test portions to slides.

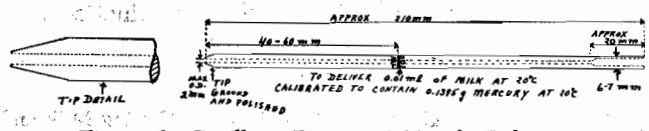

Figure 8. Capillary Pipette, 0.01 ml. Delivery.

Transfer Syringe for 0.01 ml. Bacteriological Test Portions

Because use of the 0.01-ml. pipette to transfer test portions of milk often retarded speed of deck operations at plants and because it could not be used satisfactorily on creams at low temperatures or those with high fat content, need for a substitute transfer instrument led first to the use of a 0.01-ml. transfer loop (2) and in 1951 to the discovery that for about 3 years the California State Department of Agriculture had been using a metal plunger-in-bore type syringe (2) for transfering 0.01-ml. test portions of milk and cream. The time required for each transfer with the syringe (Figure 9) is about 40% greater than

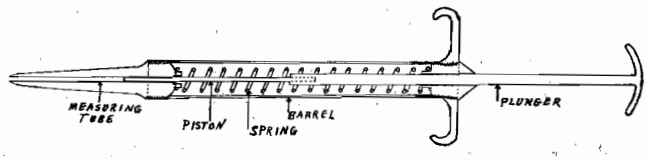

Figure 9. Semi Automatic Syringe, 0.01 ml. Delivery.

that for the transfer loop and about 25% of that required for the pipette. Because of the *positive* action of the plunger, use of the syringe assures uniform delivery of the charge measured regardless of the temperature and/or the composition of the milk and cream. Furthermore, the error of delivery can be determined by weighing on an analytical balance the charged syringe before depositing the test portion and again weighing it after the deposit has been made. The total error, including the deposited portion, could be controlled with the syringe to 0.0103, ± .0005 g., whereas the error for the initial calibration alone on the pipette, exclusive of operational measuring and depositing errors, was 0.0103, ± .0010 g.

A description of the loop, referred to above, is not included because of actual operational volume transfer errors which vary widely as compared with transfers with the syringe (2) and because after initial calibration there was no positive assurance of maintaining the same loop dimensions after repeated use following sterilizing treatments between samples.

DETERMINING ACCURACY OF GRADUATIONS ON SPECIAL SULFURIC ACID HYDROMETERS

Where Babcock and Gerber methods are used, it is necessary to determine the specific gravity (sp. gr.) of the acid with a small, short-scale Babcock acid hydrometer, sp. gr. range 1.800-1.850 in 0.001 subdivisions.

While it is not generally advisable to use reagents like concentrated sulfuric acid as test solutions, it seems that this is one instance where such use is practical. The sp. gr. of two lots of concentrated acid with strengths approaching the respective range limits of the test hydrometer may be determined with a standard hydrometer, sp. gr. range 1.780-1.850 with 0.0005 subdivisions, which has been tested for accuracy and certified thereto by the National Bureau of Standards. After determining the strength of the two lots of acid, the test hydrometers can be checked for accuracy in the same acids. Since the concentrated acids absorb H_2O readily, it is essential that the sp. gr. of the acid be checked frequently and also before and after each use for testing a batch of instruments. Those instruments which differ from the determinations by the certified hydrometer by more than 0.0005 should not be used.

DETERMINING ACCURACY OF GRADUATIONS ON LACTOMETERS

Before thermo-lactometers are issued for field or for laboratory determinations of the specific gravity (sp. gr.) of milk, checking the accuracy of these instruments at two or more points and their temperature scales at two or more points is desirable. The following procedure has been found satisfactory:

Fill a 500 ml. cylinder with distilled H_2O and submerge it to near its top in an H_2O-bath at 60°F. A constant temperature bath is convenient, but not necessary. When the temperature of the H_2O in the cylinder becomes constant at 60°F., check this constancy with a thermometer of known accuracy, and then carefully observe and record the readings for both the gravity and temperature scales of each untested, clean, dry lactometer. Be sure that the distilled H_2O is at 60°F. when each observation is made. Readings on properly graduated instruments should be 0 on the sp. gr. and 60°F. on the temperature scale.

For conformance with sp. gr. determinations, make a solution containing about 48 g. of sodium chloride per liter. This should have a sp. gr. about equal to that of normal milk *i.e.*, 103 to 105 on the Board of Health scale, and 30.0 on the Quevenne scale. Determine the sp. gr. accurately with a pycnometer, or with a standard lactometer or hydrometer of certified accuracy. Fill a clean, dry 500-ml. cylinder with this solution and submerge it to near its top in an H_2O-bath at 80°F., or other convenient temperature between 75° and 80°F. When the temperature becomes constant at the point selected, check it for constancy with a standard thermometer of known accuracy and then carefully observe and record the readings on both scales of each clean, dry lactometer, as before. Be sure that the salt solution is at the selected temperature when each observation is made. From the sp. gr. of the salt solution, calculate the lactometer reading at the temperature used, or determine the actual reading with a standard lactometer of known accuracy.

Lactometers with errors exceeding 1°F. on the temperature scale, or with errors exceeding the smallest graduation unit (usually 0.2) on the sp. gr. scale, are rejected. Usually not more than two lactometers are rejected from a gross of well prepared instruments.

If the scale of the lactometer to be tested does not extend to 0, a salt solution of proper strength, instead of distilled water, should be used to make a reading possible near the upper end of the scale. The exact strength of the salt solution will have to be determined in the same manner described for the stronger solution.

SUMMARY

The methods now used in New York State for determining the accuracy of: (*a*) volumetric glassware required when certain uses are made of determinations of fat in milk and/or cream by the Babcock or Gerber methods, and (*b*) transfer pipettes required when certain uses are made of bacterial count determinations in milk and/or cream, have been described. In addition, simple laboratory procedures for testing the accuracy of Babcock acid hydrometers and of milk lactometers are included.

Some of the above methods have been described for the first time. Other methods have been used for many years. The aim has been to assemble in one place a statement which may be a useful guide to regulatory agencies and to others charged with making certain analytical determinations on dairy products and with certain conformance tests to be applied to apparatus used when making such determinations.

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