CALIBRATION OF LABORATORY GLASSWARE: BUTYROMETER, PIPETTES, LACTOMETERS, THERMOMETER AND BURETTES

DTC- 311 (Chemical Quality Assurance)

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Contents

1. Introduction

Milk is critically analyzed in dairy plant, for its composition and quality during reception, processing and subsequent dispatch for sale. It is required for pricing, checking suitability of milk for processing and its compliance with the legal standards. In spite of the utmost care taken during analysis of milk and milk products, sometimes the results of the analysis can be wrong due to use of inaccurately calibrated glassware. Therefore, it is essential to check the calibration of glassware before being used for analysis of milk and milk products. Laboratory glasswares are made mostly from distinct types of glass like sodalime (soft) and hard or borosilicate glass (pyrex, phoenix etc.).

Some of the basic apparatus which are of universe use and are general purposes containers used in all laboratory purposes are-

1. Burette	4. Test tubes	7. Beakers
2. Pipette	5. Thermometers	
3. Flasks	6. Cylinders	

The specific apparatus are being used in dairy laboratories

1. Butyrometers	3. Hydrometers
2. Lactometers	4. Milk pipette

Most of the apparatus supplied by the large laboratory furnishers is now standardized to conform to its designs drawn by the standard institutions. Practically any apparatus ordered from laboratory supply houses must be guaranteed to conform to its standard specifications and also must be suitable for the purpose for which it is intended. For very accurate work, it may be necessary to calibrate a particular piece of apparatus in the laboratory. Proper calibration and manufacture of glassware form a reliable source is a critical issue at present. There are a number of manufacturers for butyrometers and lactometers are available but very few of them use the ISI mark.

The personnel using such types of glasswares are also not so stringent on the calibration, accuracy and source of manufacture.

So, the calibration of glasswares is necessary for :

- Control of quality of the product
- Control of process
- Legal requirements
- Analytical accuracy
- Economics to producers/consumers

2. Calibration methods of Milk Butyrometer

Calibration means establishing and recording the measurement uncertainty of measuring equipment. Proper calibration does not involve any adjustment of an instrument but may demonstrate the need for adjustment.

Calibration is a comparison between measurements-

-one of known magnitude or correctness made or set with one device and another measurement made in as similar a way as possible with a second device.

-The device with the known or assigned correctness is called the standard/references. The second device is the unit under test (UUT), test instrument (TI), or any of several other names for the device being calibrated.

Following are some of the methods:

(i) Comparison method and (ii) BIS Method

2.1 (i) Comparison method

This is not accurate method, however, sometimes and usually this method is mostly used. In this the accuracy of newly purchased butyrometers is compared by estimating fat by Gerber method in a one milk sample, along with the butyrometers of the previous batch, well calibrated and known to be accurate. If the readings of the fat values of new butyrometers are the same as of old ones, then the new butyrometers are accepted otherwise it will be rejected.

Limitation-

The previous butyrometers may not be accurate or their internal volume may have been changed due to acid corrosion.

2.2 (ii) BIS Method

A specially designed mercury pipette is used to calibrate the butyrometers to this method. This method is based on the principle that the internal volume of the graduated tube of the milk butyrometer i.e. 0.125 ml corresponding to each 1% fat range. Then the full scale of graduated tube from 0 to 10% fat marks, has the internal volume of 1.25 ml. Accordingly an automatic mercury pipette has been designed to dispense exactly 0.3125 ml mercury which fills the tube corresponding to 2.5% fat graduation limits. To calibrate the full scale from 0 to 10% fat marks, the bulb of the butyrometer is first filled up to mark of 10% graduation as the base point. Then the mercury is filled in the butyrometer from the mercury pipette 3-4 times, each time dispensing exactly 0.3125 ml of mercury corresponding to 2.5% fat graduation limit on the butyrometer column. If the graduated column of the butyrometers are exactly filled from 0 to 10% fat marks in four deliveries of the mercury pipette, then the butyrometers are accepted, otherwise rejected.

The selection of mercury as the filling liquid due to its following properties because-

(a) It does not stick to the sides of the butyrometer, hence any error due to sticking of liquid at the unwanted sides of the butyrometers will not be there.

(b) Mercury has very high density, therefore, a small change in volume, will be shown clearly by a great change in weight.

● For cream butyrometer, calibration may be checked at each 10% graduation (at any three points). In cream butyrometer, the internal volume of each 10% graduation is checked which should be 0.568 ± 0.004 ml.

● For cheese butyrometer, calibration may be checked at each 5% graduation (at any three points). In cheese butyrometer, the internal volume of each 5% graduation is checked which should be 0.169 ± 0.002 ml.

3. Calibration methods of Milk Pipettes

There are two methods for calibration of Milk Pipettes:

(i) Comparison method and (ii) BIS method

3.1 (i) Comparison method

This is not accurate method, however, sometimes and usually this method is mostly used. In this the accuracy of newly purchased pipettes is compared by estimating fat by Gerber method in a one milk sample, along with the pipettes of the previous batch, well calibrated and known to be accurate. If the readings of the fat values of new pipettes are the same as of old ones, then the new pipettes are accepted otherwise rejected.

Limitation-

In this method, the previous pipettes may not be accurate or their internal volume may have been changed due to breakage of their tips.

3.2 (ii) BIS method

This method is based upon the definition of milk pipette as given by BIS. According to BIS the milk pipette is defined as to dispense 10.75 ± 0.03 ml of distilled water at 27ºC when holding time for 15 s. For calibrating the milk pipettes, the water dispensed by the pipette is taken in a previously weighed beaker and its mass is recorded.

Density of the water at 27ºC i.e. 0.99654,

Then the volume of the water dispensed is calculated as Volume of water = (Mass of water dispensed)/(0.99654)

If the calculated volume of water dispensed by the pipette is equal to 10.75 ± 0.03 ml, then the pipette is accepted, otherwise rejected.

4. Calibration methods of Lactometers

Lactometer are works on the Archimedes principle, is basically a specific gravity (sp. gr.) hydrometer specifically designed for milk. Stem of the BIS lactometer has graduation marks in range of 20-35.

Its calibration can be checked by the following methods:

(i) Comparison method (ii) BIS methods

4.1 (i) Comparison method

In comparison method each lactometer is calibrated before use by floating side by side in a liquid, against a standard lactometer. If the lactometer readings of newly purchased lactometers resemble with that of standard lactometer, then the new lactometers should be accepted otherwise rejected.

4.2 (ii) BIS method of testing accuracy of BIS lactometer

Dissolve appropriate mass of anhydrous sodium carbonate given below in 300 ml of distilled water. Add 50 ml 92% ethanol to the solution so obtained and make up the total volume of 500 ml with distilled water. Compare the accuracy of each and every lactometer in all the solutions.

Table 1. Specific gravity and lactometer reading of pure Sodium carbonate solution

Sl. No.	Wt. of Na2Co3 (gm/500ml)	Sp. Gravity at 27C (gm/ml)	Lactometer Reading (LR)
1	19.2	1.025	25
2	21.6	1.030	30
3	24.0	1.034	34

4.3 (iii) BIS method of testing accuracy of Quevenne lactometer

This can be conveniently done by taking the specific gravity of suitable salt solutions at 15.5ºC.

Table 2. Specific gravity of pure Sodium Chloride Solution

Sl. No.	Pure Sodium Chloride Solution	Sp. Gravity at 15.5º (gm/ml)	Lactometer Reading (LR)
1	3.863%	1.026	26
2	4.415	1.032	32

The sp. gr. of these salt solutions must be checked by a specific gravity bottle, and then the lactometer readings are taken in exactly the same way as with milk.

5. Calibration methods of thermometers

There are several types of thermometers depending upon the types of temperatures and their measuring ranges. In milk testing glassware, mainly two types of thermometers are used-

(a) Thermometer (range 0 to 100ºC) and

(b) Freezing Point Depression Thermometer (range –0.5 to 0ºC).

Following are the methods to calibrate these thermometers:

5.1 **Calibration of 0-100°C Thermometer**

5.1.1 (i) Comparison method

It is not very accurate method, however, usually it is mostly used. This thermometer is compared with one or more thermometers which are known to be accurate. At least two different temperature marks (preferably nearer to lower and upper limits) are compared.

5.1.2 (ii) Physical method

In this method the zero point is located by dipping the mercury bulb of the thermometer in the melting ice kept in a wide funnel. Where the temperature becomes stationary, mark that 0°C point. The 100°C point is located by keeping the mercury bulb of the thermometer in steam at normal pressure. If the pressure is not 760 mm then appropriate correction is applied. In general at the natural atmospheric pressure 0.038°C is added or deducted from the observed boiling point per mm pressure of mercury lower or higher than 760 mm, respectively.

6. Calibration methods of Freezing Point Depression Thermometer

The soluble ions or molecules play very important role in depressing the freezing point of solvent such as water. Milk is a solution in which water is the solvent and mainly lactose and minerals are solutes, which depress its freezing point. The one molal solution of any ion or unionized molecule depresses the freezing point of water by 1.86ºC. It means one molal solutions of non-ionized substances like sugar (360g in 1000g of water), glucose (180 g in 1000 g of water) etc. depress the freezing point by 1.86ºC. However, one molal solution of ionized molecules depresses the freezing point by (1.86 x numbers of ions per molecules) ºC.

For example, one molal solution of sodium chloride will depress the freezing point by 1.86 x 2 = 3.72ºC.

Now, 1.86ºC is depressed by a solution = 1 molal

1ºC is depressed by a solution = 1/1.86 = 0.538 molal

0.5ºC is depressed by a solution = 0.269 molal

Therefore, -0.5ºC and -1ºC points on the freezing point depression thermometer are checked by using the 0.269 and 0.538 molal solutions, respectively.

For pure milk –0.54ºC = 0.29 molal or –0.55ºC = 0.3 molal.

7. Calibration methods of Burette

Burette is checked at different intervals such as 5, 10, 25 or 50 ml for determining the accuracy of its scale and the capacity of the burette. The volume actually delivered for each interval is obtained as per the procedure outlined for milk pipette.

Capacity of Burette (ml)	Burettes	
	Class A	Class B
1	0.006	0.01
2	0.010	0.02
5	0.010	0.02
10	0.020	0.05
25	0.050	0.10
50	0.050	0.10
100	0.100	0.20

8. Conclusion

Laboratory glass wares are made mostly from distinct types of glass. Therefore, it is very essential to check the calibration of glassware before being used for analysis. For very accurate work, it may be necessary to calibrate a particular piece of apparatus in the laboratory. Proper calibration and manufacture of glassware is a critical issue at present. There are a number of manufacturers for butyrometers and lactometers are available but very few of them use the ISI mark. The calibration of glasswares is necessary for: control of quality of the product, control of process, legal requirements, economics to producers/consumers and analytical accuracy.

9. References

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