CALIBRATION OF LABORATORY GLASSWARE

BUTYROMETER, PIPETTES, LACTOMETERS, THERMOMETER AND BURETTES

Need of calibration of dairy glasswares:

1. Milk is critically analyzed for composition and quality during reception, processing and subsequent dispatch for sale.
2. This is required for pricing, checking its suitability for processing and to know its compliance with the legal standards.
3. In spite of the utmost care taken during analysis of milk and milk products,
4. Sometimes the results of the analysis can be erroneous due to use of inaccurately calibrated glassware.
5. Therefore, it is very essential to check the calibration of glassware before being used for analysis.

Commonly used glasswares for analysis of milk in a dairy industry:

1. Milk Butyrometer
2. Pipette
3. Lactometer
4. Thermometer
5. Burette

Definition of calibration:

It is the act of marking units of measurement on an instrument so that it can e used for measuring something accurately.

1. Calibration of milk butyrometer

Following are some of the methods:

(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 rejected.

Limitation:

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

(ii) BIS Method

In this method, a specially designed mercury pipette is used to calibrate the butyrometers.

- The method is based on the principle that the internal volume of the graduated tube of the milk butyrometer is 0.125 ml corresponding to each 1% fat range.

- In other words, 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 upto 10% graduation mark as the base point.

- Then the mercury is added in the butyrometer from the mercury pipette four 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 mercury is chosen as the filling liquid due to its following properties:

(a) It does not stick to the sides of the container, hence less chances or error.
(b) It has very high density, therefore, a small change in volume, will be evidenced clearly by a great change in weight.

For cream butyrometer, calibration may be checked at each 10% graduation (at any three points). Here 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). Here the internal volume of each 5% graduation is checked which should be 0.169 ± 0.002 ml.

2. Calibration of milk pipettes

Following are some of the methods:

(i) Comparison method

This is not accurate method, 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:
The previous pipettes may not be accurate or their internal volume may have been changed due to breakage of their tips.

(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 27°C when held 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.
- Knowing the density of the water at 27°C i.e. 0.99654, 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.

(iii) Mathematical method of calibration and graduation

For checking the marks/points corresponding to 10.75 ml capacity of milk pipettes, the following procedure can be applied:

As the stem of the pipette is of uniform cross-section, therefore, internal volume per unit length of stem at any point is constant. Fill the pipette with distilled water to a temporarily marked point A on the upper stem of the pipette and dispense in a tarred` beaker and weigh. Knowing density of water, calculate the volume of pipette up to the mark A. Similarly again fill up the pipette with the water up to another point B (above A) and find the volume of water dispensed.

Let volume of water up to point A = V_a ml
And volume of water up to point B = V_b ml
Therefore, volume between A and B = $(V_b – V_a)$ ml
Now note the distance between A and B points, let it is = d cm
Therefore, stem of the pipette has volume = $(V_b – V_a)/d$ ml per cm length.
Now, let V_a is less than 10.75 ml
Therefore, difference of volume = $(10.75 – V_a)$ ml
Now as we know that volume $(V_b – V_a)/d$ ml occupies length = 1 cm
And $(10.75 – V_a)$ ml occupies length = d x $(10.75 – V_a)/(V_b – V_a)$ cm

Therefore, mark a point above A at a distance = d x $(10.75 – V_a)/(V_b – V_a)$ cm, which will correspond to 10.75 ml mark.

3. Calibration of lactometers

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

Its calibration can be checked by the following methods:

(i) Comparison method

In this 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.

(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.

Specific gravity and lactometer reading of pure Sodium carbonate Solution

Sl. No.	**Wt. of anhydrous Na_2CO_3 (gm/500ml)**	**Specific gravity at 27°C (gm/ml)**	**Lactometer Reading (LR)**
1	19.2	1.025	25
2	21.6	1.030	30
3	24.0	1.034	34

(iii) BIS method of testing accuracy of Quevenne lactometer

This can be conveniently done by taking the specific gravity of suitable salt solutions at 15.5°C. The solutions used are given below

Specific gravity of pure Sodium Chloride Solution

Sl. No.	Pure sodium chloride solution	Specific gravity at 15.5°C (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 sp. gr. bottle, and then the lactometer readings are taken in exactly the same way as with milk.

4. Calibration 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 100°C) and (b) freezing point depression thermometer (range –0.5 to 0°C). Following are the methods to calibrate these thermometers:

Calibration of 0-100°C thermometer

(i) Comparison method

Though it is not very accurate method, however, usually it is mostly used. In this thermometers are 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.

(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.

Calibration of freezing point depression thermometer

Before calibration of these thermometers, the definitions of two types of solutions are to be understood

Molar solution

It is defined as a solution prepared by dissolving one gm molecule of any substance in solvent so as to make 1000 ml solution.

Molal solution

It is defined as a solution prepared by dissolving one gm ion (in case of ionized molecules) or molecule (in case of non-ionized molecules) in 1000 gm of solvent.

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 1.86°C.

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 1.86°C.
However, one molal solution of ionized molecules depresses the freezing point by (1.86 x numbers of ions per molecules)°C.
For example, one molal solution of sodium chloride ($Na^{+}Cl^{-}$) will depress the freezing point by 1.86 x 2 = 3.72°C.
Now, 1.86°C is depressed by a solution = 1 molal
1°C is depressed by a solution = 1/1.86 = 0.538 molal
0.5°C is depressed by a solution = 0.269 molal
Therefore, -0.5°C and -1°C points on the freezing point depression thermometer are checked by using the 0.269 and 0.538 molal solutions, respectively.
For pure milk –0.54°C = 0.29 molal or –0.55°C = 0.3 molal.

Calibration of burette

Burette is checked at various intervals say 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.

Table 20.4 Tolerance for burette

Capacity (ml)	**Burettes**	
	Class A	Class B
1	0.006	0.01
2	0.01	0.02
5	0.01	0.02
10	0.02	0.05
25	0.05	0.1
50	0.05	0.1
100	0.1	0.2