

SAMPLING PROCEDURES; LABELING OF SAMPLES FOR ANALYSIS AND CHOICE OF ANALYTICAL TESTS

Introduction

Sampling of milk and milk products shall be done by an experienced person who is familiar with the techniques and is well acquainted with the knowledge of the subject. It is not possible to lay down a single sampling procedure which will be applicable in all the cases. The sampling procedure, therefore, differs according to the nature of the material and the purpose for which it is needed. Sampling may be required for chemical or bacteriological examination. All precautions shall be taken to prevent contamination and adulteration. For chemical examination, the sampling equipment shall be clean and dry. For bacteriological examination, all equipments including plunger, sample bottles and rubber stoppers shall be sterile and the samples shall be collected under aseptic conditions.

If subsequent analysis or interpretation is to be of some value, it is very important that sample should be a true representative of the bulk. Since milk fat is of lower density than the other constituents of milk, it tends to rise to the surface. Therefore, thorough mixing of milk with a proper instrument which will reach the entire depth of the liquid is essential to ensure a representative sample of the entire batch. In small batches, it should be possible to accomplish mixing by pouring the entire quantity of milk from one container to another, three or four times. Larger batches of milk shall be thoroughly agitated by a hand stirrer or by mechanical means. Milk churns easily at 26.5 to 29.5°C and agitation near this temperature shall be avoided.

Sampling from Individual Container

Pour the milk from one container to another, three or four times. Where this is not practicable, mix thoroughly with a plunger. In mixing the milk, the plunger shall be allowed to fall to the bottom of the container and brought to the top of the milk as rapidly as possible not less than 10 times. The position of the plunger shall also be moved from place to place to ensure that the whole of the milk at the bottom of the vessel is thoroughly agitated and mixed with the upper layer. Any milk fat adhering to the neck and under the shoulder of the can shall be well mixed with the remainder of the milk. After thorough mixing, a sample shall be drawn immediately.

Sampling from individual



container

Sampling from several containers

The sample shall be taken after pouring the contents of the containers into a vat and mixing. When this is not possible, a composite sample is taken in the following manner from the containers after milk has been agitated and mixed. First, the milk shall be distributed as equally as possible among a number of containers. The cans shall not be filled, but the same quantity shall be placed in each. After mixing the contents of each can thoroughly, an equal volume of milk shall be taken from each. These portions shall be placed in another vessel, thoroughly mixed as described in case of individual container and a sample then taken.

Alternatively, where facilities exist for accurate measurements, a composite sample may be obtained by taking the same proportion of the milk therein from each container in a consignment after thorough mixing, collecting this in another vessel and taking a sample as described in case of individual container.



Sampling from several containers

Sampling Bulk Units

When milk of uniform quality is supplied in bulk units (for example, cans filled from storage tanks), the number of random units to be sampled shall be as follows:

Unit selection in testing laboratory

Total Number of Units	Number of Units to be Selected
1	1
2-5	2
6-20	3
21-60	4
61-100	5
Over 100	5 plus one for each additional 100 units or fraction thereof

The testing laboratory, may, within its discretion, instruct the person who draws the sample to submit separate samples from each unit selected, or one or more composite samples consisting of aliquot portions from each unit selected.

The latter course should only be applied where the product is likely to be of fairly uniform composition, for example, where the consignment to be sampled is produced from a quantity of properly mixed milk, and where variations in composition from unit to unit are, therefore, small. But, where there is a possibility of wide variations between different units, every selected unit shall be separately sampled.

Sampling from storage tanks and rail and road milk tankers

The method of sampling of milk from storage tanks and rail and road tankers is largely governed by storage/transport conditions. It is, therefore, difficult to lay down any rigid procedure for the sampling, but the following is recommended:



Sampling from storage tanks and rail tankers

In all cases, the milk in the tank/tanker shall be thoroughly mixed by a sufficiently large plunger, a mechanical agitator or by compressed air; the uniformity of the samples being determined, when necessary, by mixing till such time as complete agreement is obtained

between samples taken at the manhole and at the outlet cock in respect of fat and total milk solids.

NOTE- When a plunger is used for mixing the milk in rail or road milk tankers, a convenient and satisfactory method is to insert the plunger in the man-hole, the operator sitting or standing astride (with the legs apart on each side) on top of the tanker. The plunger is thrust forward and pulled back, thrust downwards and pulled back and thrust backwards and pulled back. The cycle of operations should be repeated for at least 15 minutes.



Fig.7.4 Plunger

After proper mixing of the milk, the sample may be taken from the tank, removed through the stopcock in the tank door, or from a valve on the discharge line from the tank when it is being emptied.

Composite Milk Samples for Fat test

Suppliers of milk are often paid for milk on the basis of fat test. The determination of fat contents of the suppliers' daily deliveries is laborious and expensive. Therefore, composite samples of the suppliers' milk are taken over a period and then tested. After thorough mixing, proportionate amounts of the suppliers' daily delivery are collected and placed into the patron's composite sample bottle. The total volume of the individual composite sample shall be not less than 175 ml. For preserving the composite sample, 0.1 ml of 36 percent formaldehyde for 25ml of milk may be used. The bottle containing the composite milk sample shall be tightly stoppered to prevent evaporation and kept in a locker, away from light, till required for analysis. The sample shall be analyzed on the same day as the last portion of milk is transferred to the composite sample bottle.

NOTE – Each time when fresh sample of milk is added, the sample shall be mixed by rotating the bottle to prevent the formation of solid cream layer or cream plug.

Treatment of Milk Sample on Arrival at the Laboratory before Analysis

Warm the sample in the bottle to about 40°C in a water bath and mix thoroughly. Cool to 26°- 28°C. Leave aside the sample for about 4 minutes after mixing to allow air bubbles

to rise and escape. After that, mix the sample by inverting the bottle 3-4 times and start analysis.

Preparation of cream sample for analysis

When cream is thin and in small containers, it shall be mixed either by six transfers, or by plunging not less than ten times. The position of the plunger shall be moved from place to place to ensure that the whole of the cream at the bottom of the vessel has been thoroughly agitated and mixed with the upper layer. To avoid whipping and churning, the disc of the plunger shall not be brought above the surface of the cream.

When cream is thick or in bulk containers, it shall be mixed by plunging as described for thin cream.

When the cream is sour, the material shall be warmed so as to attain a temperature between 30° and 40°C and, while cooling it to room temperature, the container shaken gently or the contents stirred. Keep the contents covered as much as possible.

In all cases the sample shall be taken immediately after mixing.

Sampling of Paneer/Cheese/Chhana

One of the following three methods is employed

Sampling by cutting a sector

Using a knife with a sharp blade, two random cuts are made radially proceeding from the centre of the cheese/paneer towards the edge.

Sampling by means of a trier

The cheese trier is driven obliquely into the surface of the paneer or cheese towards the centre once or several times at a point at least 10 to 20 cm from the edge of the cheese. From the boring or borings thus obtained a part of at least 2 cm length is cut off together with the crust and is used to close the hole of the cheese. The remaining portions of the boring or borings constitute the sample.



Fig.7.5 Trier

However, when the cheese is delivered in drums, cases or other larger containers, sampling may be carried out by driving the trier obliquely through the

content of the container from the top to bottom. This method is suited for sampling of processed cheese.

Sampling by taking a whole cheese

This method is made use of for cheese packed in small containers.

Preparation of Paneer/Cheese/Chhana Sample for Analysis

Samples shall be prepared for chemical analysis by passing them quickly through a suitable grater, by grinding them quickly in a mortar and returning them to the sample container or by cutting them into small pieces with a sharp knife in the container.

Cheese

Sampling by cutting

Use knife with a pointed blade

Circular base

Make two cuts radiating from the centre of the cheese, when cheese is circular in shape, after removing the inedible portion sample should be minimum 150 gm.

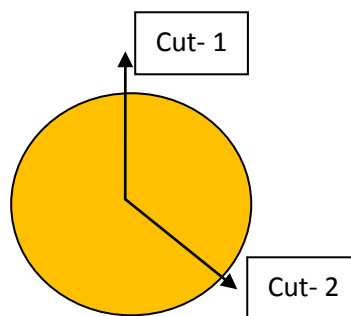


Fig.7.6 Cutting of cheese

Rectangular base

Make cuts parallel to the sides. After removing the inedible portion sample should be minimum 150 gm.



Fig.7.7 Cutting of cheese on rectangular base

Sampling by using trier

Use trier

- i) Insert Trier obliquely towards the centre of the cheese into one of the surfaces at a point not less than 10 cm from the edge.

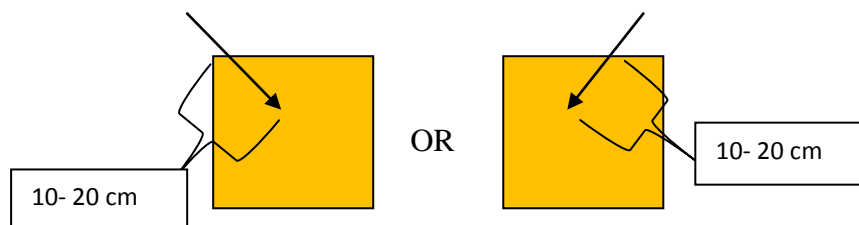
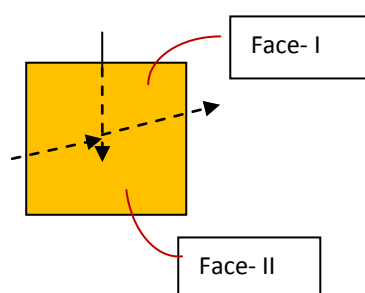


Fig.7.8 Sampling by insertion of Trier towards center of cheese

- ii) Insert Trier horizontally into the vertical face midway between the two plain faces, towards the centre of the cheese
- iii) Insert Trier perpendicularly into one face and pass through the centre of the cheese to reach the opposite face.



Sampling by insertion of Trier perpendicularly

All samples should be prepared for chemical analysis by passing them quickly through a suitable grater, by grinding them quickly in a mortar and returning them to the sample container or by cutting them into small pieces with a sharp knife in the container.

Sampling of Khoa

The sampling of khoa follows the procedure used for sampling cheese/paneer/chhana, except that a clean dry stainless steel knife with sharp pointed blade is used to cut khoa for sampling.

Sampling of Condensed Milk

Scale of sampling

The number of containers to be selected from each lot shall be as follows:-

a) *For containers of 400 gm to 5 Kg.*

Lot Size (N)	No. of containers to be selected (n)
Upto 300	3
301 – 500	5
501 – 1000	7
1001 and above	10

b) *For containers of more than 5 Kg and upto 20 Kg*

Lot Size (N)	No. of containers to be selected (n)
Upto 100	2
101 – 300	3
301 – 500	4
501 and above	5

c) *The scale of sampling for containers of 200 g and above 20 kg shall be an agreed to between the purchaser and the vendor.*

The containers from the lot shall be chosen at random. For example, starting from any container, count them as 1, 2, 3 ----- etc. up to r in one order, where $r = N/n$ (N being the size of the lot and n being the number of containers to be selected). Every r^{th} container thus counted shall be separated until the requisite number of containers is obtained from the lot to give the sample for test.

Preparation of sample of condensed milk for analysis

On storage of condensed milk, separation of the constituents such as fat, lactose may occur. It is necessary to mix the contents of the container prior to analyses in the following manner:

Heat the container in a water bath at about 40°C until the sample has nearly reached this temperature. Open the container at the edge of the lid. Re-incorporate all the material adhering to the lid into the container. Mix the contents thoroughly by stirring with a

spoon or spatula, in such a way that the top layers as well as contents of the lower corners are moved and mixed. Repeat the stirring before drawing the sample for testing various parameters.

Sampling of Milk Powder

Scale of sampling

The no. of containers to be selected from each lot shall be as follows:

a) For containers of 500 gm and upto 5 kg

For containers of 500 gm and upto 5 kg

Lot Size (N)	Sample Size (For tests other than Microbiology) (n)	Sub sample size(For Microbiology test) (n)
Upto 100	3	1
101 to 300	5	2
301 to 500	7	3
501 & above	9	4

b) For containers of more than 5 kg

For containers of more than 5 kg

Lot Size (N)	Sample Size (For tests other than Microbiology) (n)	Sub sample size(For Microbiology test) (n)
Upto 100	2	1
101 to 300	3	1
301 to 500	4	2
501 & above	5	3

Preparation of sample of milk powder for analysis

Draw with a suitable sampling instrument approximately equal quantities of the material from different parts of the same container till about 150 g of the material is obtained. Transfer the material immediately to thoroughly clean and dry container and seal air-tight.

Sampling of Ice-Cream

Scale of sampling

The no. of containers to be selected from each lot shall be as follows:

A) When the product is supplied in bulk units

Product is supplied in bulk units

Total Number of Units (N)	No. of Units to be selected (n)
1	1
2 to 5	2
6 to 20	3
21 to 60	4
61 to 100	5
Over 100	5 plus one for each additional 100 units or fraction thereof.

NOTE- When there is a possibility of wide variations between different units, every unit shall be sampled.

B) When the product is supplied in retail units

Product is supplied in retail units

Total Number of Units (N)	No. of Units to be selected (n)
1 to 100	1
101 to 1000	2
1001 to 10,000	4
Over 10,000	4 plus one for each additional 2500 units or fraction thereof

NOTE - Each batch is to be dealt separately in a similar manner.

Preparation of sample of Ice-Cream for analysis

The samples shall be stored at a temperature not higher than -15°C . During transit the samples shall be maintained at a temperature not exceeding -15°C .

Any sample of ice-cream shall not be less than 100g. If necessary several packages of smaller size shall be taken to make up the required size of sample.

In the case of multilayered ice-cream, the sample shall be such as to contain the same proportion of each layer as is present in the original ice-cream. Different layers shall not be separated at the time of sampling and a complete sample of all layers shall be placed in the sample jar.

For the purpose of melting, the frozen sample may be kept at room temperature or, if required, in water bath at a temperature not exceeding 45°C for not more than 15 minutes. Thoroughly mix the samples before removal of the test portion.

Sampling of Butter

Scale of Sampling: The no.of containers to be selected from each lot shall be as follows

A) When the product is supplied in bulk units (like casks or boxes)

Product is supplied in bulk units

Total Number of Units (N)	No. of Units to be selected (n)
1	1
2 to 9	2
10 to 49	3
50 to 99	4
100 to 199	5
Over 200	5 plus 1 for each additional 250 units or fraction thereof

B) When the product is supplied in small units (like packets or tins)

Product is supplied in small units

Total Number of Units (N)	No. of Units to be selected (n)
1 to 100	1
101 to 1000	2
1001 to 10,000	4
Over 10,000	4 plus 1 for each additional 2500 or fraction thereof

Sampling technique for chemical analysis

Hard and semi-hard butter kept under cold storage

a) *From churns*

Four cores shall be drawn with the help of a trier at equal distances. At least two should be near the centre of the churn.

b) *From trollies*

Four cores (one each from the two ends and the other two from the sides) shall be drawn with the help of a trier.

c) *From boxes*

Three cores shall be drawn by inserting a trier vertically through the block. One core would be at the centre and the other two near diagonally opposite corners of the open end.

d) *From casks*

Three cores shall be drawn by inserting a trier at three points equidistant from the circumference of one end of the block and directed through the centre of the block.

e) *From small packets*

The samples shall consist of the unopened packets. After taking the sample for bacteriological test, the rest shall be used for chemical analysis.

f) *Barrel*

Butter barrel (a) Insert butter Trier diagonally from the edge of the barrel and rotate the Trier through complete turn and take out Trier with a plug of butter (b) Take another plug by inserting Trier arbitrarily at any point of the surface vertically down to the bottom rotate the Trier through complete turn and take out trier with a plug of butter. Plug the holes with about 25mm of the plug and use remaining 75mm of the plug as sample and mix the samples drawn (200gm) for analysis

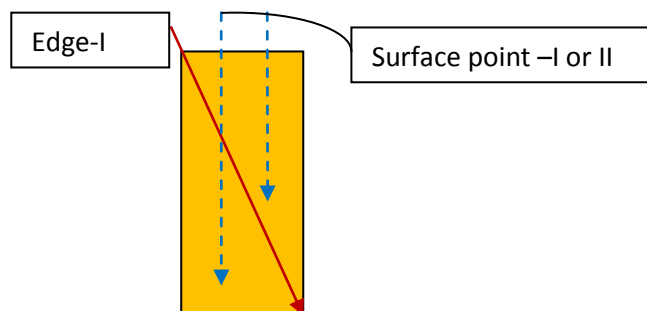


Fig.7.10Sampling of butter barrel

i) Block

Insert trier from top corner-I diagonally through out the centre to the bottom and rotate trier through one complete turn and withdraw the full core. Repeat the sequence from top corner-II and plug the holes with 25mm portion of the butter core drawn.

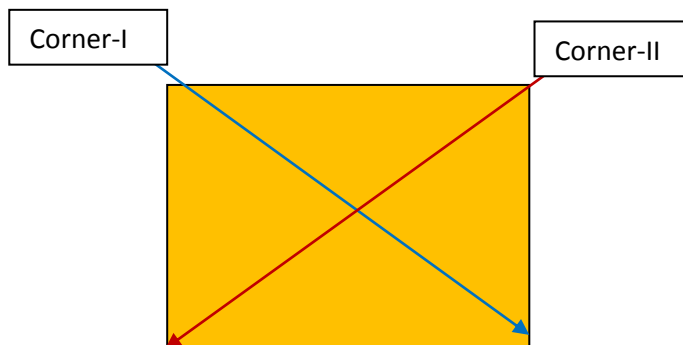


Fig 7.11 Sampling of butter block

ii) Butter in pats or rolls less than 500 gm: In this case take whole unit as sample

iii) Butter in pats more than 500 gm: Divide unit into four parts and take two opposite quarters as sample

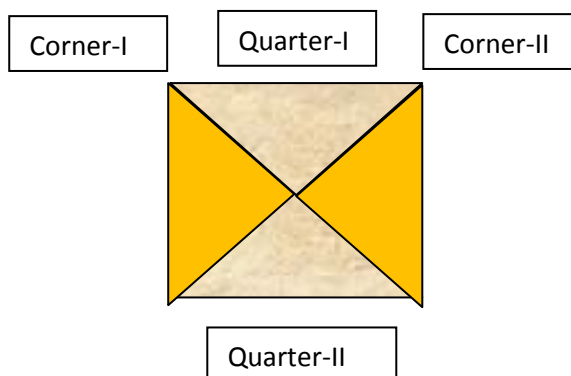


Fig 7.12 Division of butter block in quarters

Pasty butter kept under warm conditions

When the product is in small quantities, remove a sample from the deeper layers of the product at the centre of the block and two other points roughly equidistant from the central point, located 2 to 3 cm away from the ends. A suitable, clean, dry spoon, spatula or a trier should be used.

When the product is in the form of large heaps or blocks, select three points, one at the centre, the second about 2 to 3 cm away from the bottom and the third at an equal distance from the centre on the opposite side. At each point, draw from the deep layers

three cores, roughly equidistant on the circumference. A suitable, clean, dry spoon, spatula or a trier should be used.

Preparation of sample of butter for chemical analysis

Sample for analysis of butter

Warm the sample in an air-tight container with the lid screwed down tightly or with the glass stopper, in an oven or water-bath not exceeding 39°C until by frequent vigorous shakings a homogenous fluid emulsion (free from un-softened pieces) is obtained at the lowest possible temperature.

Sample for analysis of butterfat

Heat a portion of emulsified butter in a beaker to a temperature of 50-60°C until the fat separates. Filter the fat layer through a dried filter paper into a dry vessel at a temperature above the solidification point of the fat, using a hot-water funnel, if necessary. Re-filter the filtrate under the same conditions, until it is clear and free from water. Liquefy the fat completely and mix before taking samples for analysis.

Note: Exposure to light and air of the butter sample or the butterfat obtained from it shall be as short as possible and analysis shall be carried out without delay.

Labeling of Samples for Analysis

Each sample container (bottles or jars or other container) shall be sealed air-tight after filling and a label marked with the following particulars should be put on the container:

Purpose of sampling

Sampling of milk and milk products is generally done for Chemical analysis, Bacteriological analysis, Sensory analysis etc. The sample should bear the following information to ensure the tractability of the sample.

Name of the supplier/manufacturer:

Date and time of sampling and place of sampling:

Nature of the product like: Milk sample, Butter sample, Ghee sample, Milk powder etc.

Identification number, name, designation and signature of the person responsible for taking the sample:

Mass or volume of the sample:

Particular of the stock/ unit from which the sample is taken i.e.

Stock number

Batch number

Code number

Preservative added or not to keep the sample suitable/ fit for analysis.

- If yes, then
- The nature of preservative added
- Quantity of preservative added

If no preservative is added, then storage, temperature during transit or transportation till analysis is done, should be mentioned.

e.g.

- Store at refrigerated temperature
- Store at room temperature etc.

Additional Information: if sample is taken from a food which has some certification mark like ISI, Agmarketc, then in that case, give additional information such as

Mark (ISI/Agmark)

Grade (Special, General etc)

Agmark label no./Batch no.

Name packing station where the food was packed etc.

Sampling of Ghee

Sampling shall be carried out by an experienced person. A sample which is representative of the bulk is essential. All the containers in a single consignment belonging to the same batch of manufacture shall be grouped together to constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots. The

number of containers to be selected for sampling shall depend upon the lot size and shall be in accordance with following Table.

Sampling ghee containers

Number of containers in lot	Number of containers to be selected
1	1
2 to 40	2
41- 110	3
111-300	5
301-600	7
601 and above	10

These containers shall be selected at random from the lot. Samples drawn from the consignment should be placed in appropriate containers, which could be *wide mouth jar* and *bottles* and *tin containers* of 50, 100 and 200/250 ml capacities. The jars shall be closed by means of a screw cap lined with butter paper. Bottles shall be glass-stoppered. Tin containers shall be closed with the, press-on type of lids. For chemical analysis, bottles may also be closed with rubber stoppers lined with butter paper if organoleptic tests are not to be made.

For the preparation of composite sample, collect equal quantity from each of the selected containers so as the total quantity is at least 300gm.

Choice of Analytical Test

Introduction

Generally an analytical chemist or scientist will confront with the problem of selection of proper method from array of methods for quantitative analysis. A variety of methods may be capable of achieving the desired analysis and the decision to select one may depend on a variety of issues.

There are several factors or criteria or issues which determine the choice of selecting a method from the available number of methods. These are as follows:

- *Speed*
Time taken by a method to complete the analysis is one of the criteria. It should be less time consuming method.
- *Convenience*
The method should be convenient to use. It should not be cumbersome i.e. difficult.
- *Accuracy/Precision*
The method of analysis should give accurate results. That means the method should be error free.
- *Sensitivity/Detection limits of the method*
The method should be sensitive enough to estimate even the small traces of component. Higher the sensitivity better will be the results. Therefore, as far as possible the detection limit should be low means the method should be able to detect lower levels of the components in a food.
- *Selectivity/Non-interference of other compounds present in the sample*
The method should selectively estimate the component which we want to estimate. Other components present in the sample should not interfere in the estimation of a particular component in the sample.
- *Availability of instruments/Specific apparatus*
Instrument/ Specific apparatus required in a selected method should be available in the laboratory or a department where work is being carried out.
- *Amount of sample*
The selected method should be such that only small amounts of sample should be required for analysis.
- *Level of analysis/Nature of analysis*
The method of selection of a particular test will also depend upon the type or nature or level of analysis. For example, the test may be required for qualitative purposes. Generally, separate tests or methods are available for qualitative and quantitative analysis.

In case of quantitative analysis, the selection of a test or method will also depend on whether you want to estimate the given component from a gross composition point of view or a contamination point of view. It may also be required to check the residual level of the component after the processing of a product so as to check the permissible limits of the residual components (e.g. pesticides, antibiotics etc) as per the food laws. For all these

purposes different types of methods are required. For example, for the estimation of traces or residual contents, more sensitive methods are required.

1. *Cost of the method*

The method of analysis should be such that the cost of estimation of a component should not be high.

2. *Hazards free/Risk free*

The method selected should be such that there should be no hazard/risk involved in the analysis. There should be appropriate precautions needed to minimize the risk involved, if any.

3. The published literature should be available to choose or select a method.