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

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Sampling Procedures

- To be done by an **experienced person** who is familiar with the techniques and is well acquainted with the knowledge of the subject.
- Differs according to the **nature of the material** and the **purpose** for which it is needed (chemical or bacteriological examination).

For **chemical examination➔** sampling equipment should 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.

- Precautions should be taken to prevent contamination and adulteration.
- Sample should be a **true representative** of the bulk (thorough mixing of the sample)
- As 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. *IF this is not practicable*, mix thoroughly with a plunger.

- Allow it 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*.
- position of the plunger ➔ be moved from place to place to ensure that the whole of the milk is thoroughly agitated.
- Milk fat adhering to the neck and under the shoulder of the can ➔ to be well mixed with the remainder of the milk.
- After thorough mixing ➔ a sample shall be drawn immediately.

Sampling from several containers

- Sample to be taken *after pouring* the contents of the containers into a vat and **mixing**.

OR

- Firstly, distribute the milk equally among a number of containers. Containers shall not be fixed but the same quantity shall be placed in each
- Secondly, after mixing the contents of each can thoroughly, an equal volume of milk shall be taken from each in another vessel
- Thoroughly mixed as described in case of individual container and a sample then taken.
- Where variations in composition from unit to unit are, the very small then only composite samples consisting of aliquot portions from each unit can e done.

- 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 milk in the tank/tanker shall be thoroughly mixed by a sufficiently large plunger.
- The operator should be 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.

Composite milk samples for fat test

- Suppliers of milk are often paid for milk on the basis of fat test.
- 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 sample shall be analyzed on the same day after complete collection of milk.
- 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

Preparation of Milk Sample 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

- Sample should be mixed either by six transfers, or by plunging not less than ten times evenly in all sides of the container.
- To avoid whipping and churning, the disc of the plunger shall not be brought above the surface of the cream.
- For sour cream, it shall be warmed between 30° and 40°C and, while cooling it to room temperature, the contents stirred.
- Keep the contents covered as much as possible.
- In all cases the sample shall be taken immediately after mixing.

Trier

Preparation of Paneer/Cheese/Chhana Sample for Analysis

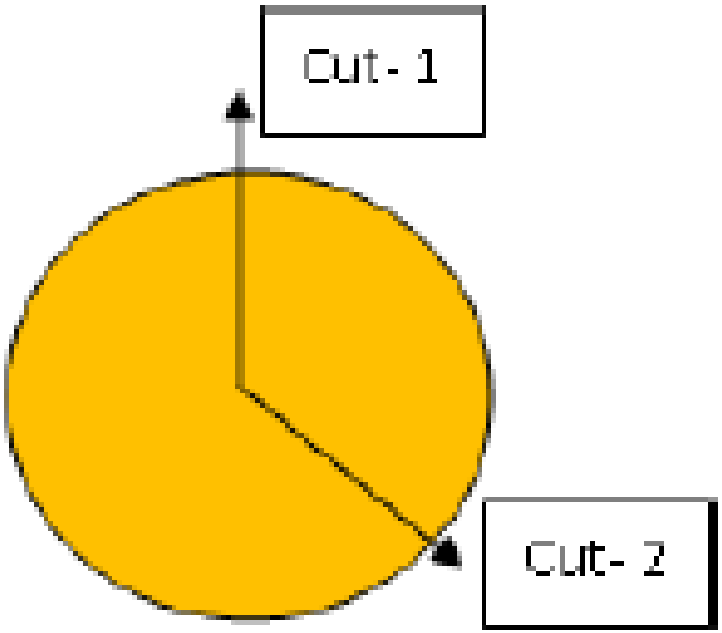
All samples should be prepared for chemical analysis by passing them quickly through a suitable grater, by grinding them quickly in a mortar and returning them to the sample container or by cutting them into small pieces with a sharp knife in the container.

Cheese

1. Sampling by cutting

Use knife with a pointed blade

2. Circular base

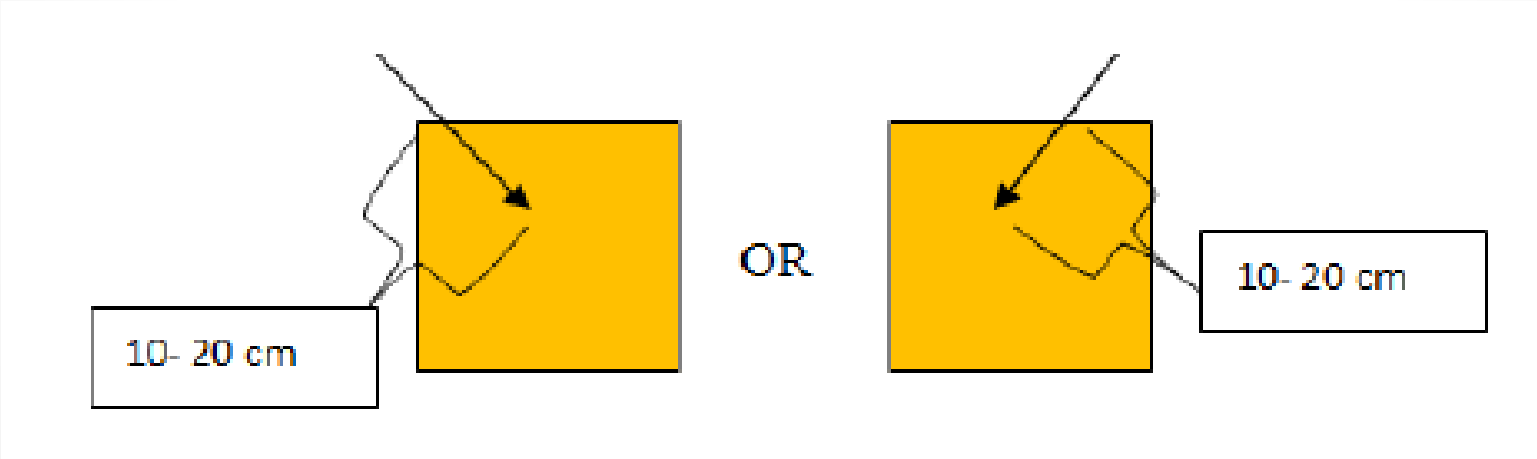


After removing the inedible portion sample should be minimum 150 gm.

3. Rectangular base

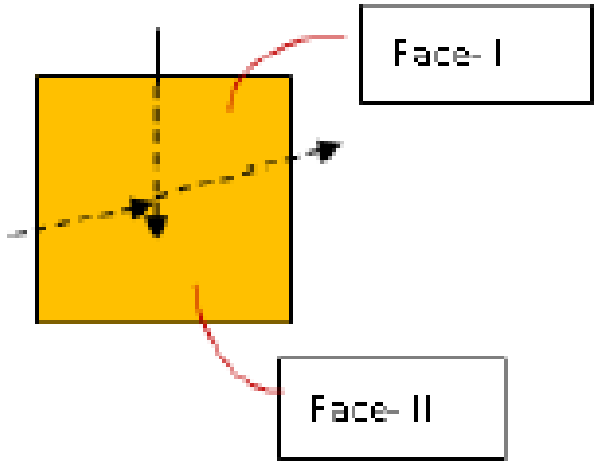
4. Sampling by using trier

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.



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 of Khoa

- The sampling of khoa follows the procedure used for sampling cheese/paneer/chhana.

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 up to 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

Preparation of sample of condensed milk for analysis

Storage causes separation of the constituents such as fat, lactose may occur. It is necessary to mix the contents prior to analyses in the following manner:

- Heat the container in a water bath at about 40°C.
- 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

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

a) 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

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 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

- **When the 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.

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

- **When the 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

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 -15°C.
- Sample size shall not be less than 100 g.
- For 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.
- For the purpose of melting, the frozen sample may be kept at room temperature or 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

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)

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 thereo

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

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

Sampling technique for chemical analysis

Hard and semi-hard butter kept under cold storage

1. 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.

2. 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.

3. 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.

4. From casks

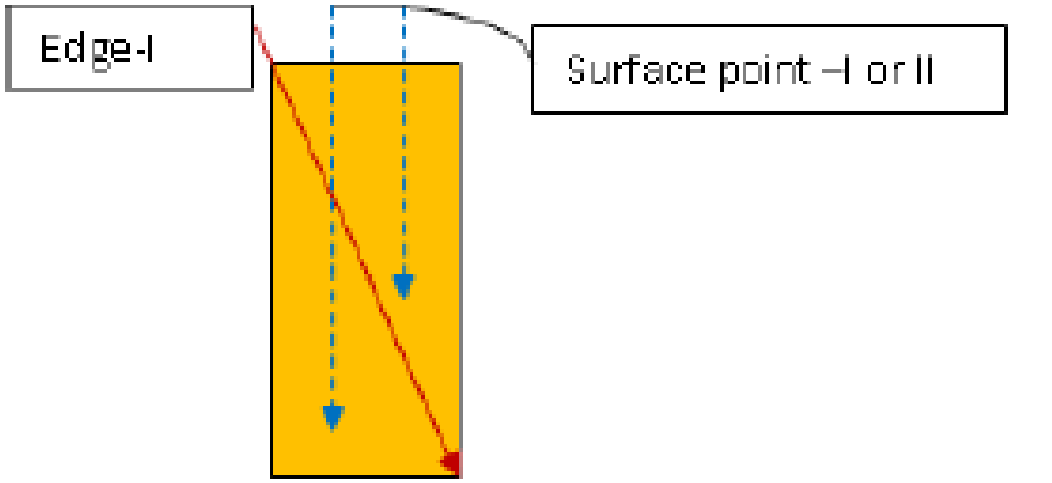
- Three cores shall be drawn by inserting a trier at three points equidistant from the circumference of one end of the block and directed through the centre of the block.

5. 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.

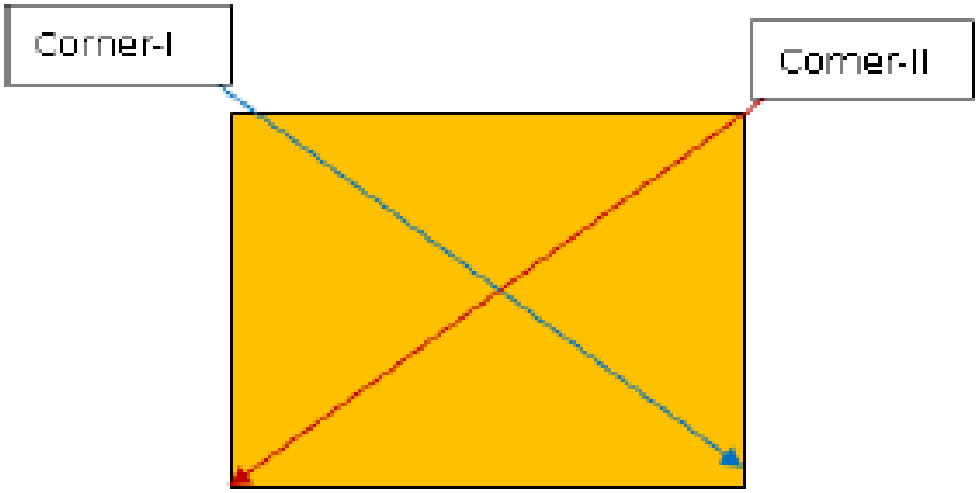
Barrel

- 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 25 mm of the plug and use remaining 75 mm of the plug as sample and mix the samples drawn (200 gm) for analysis

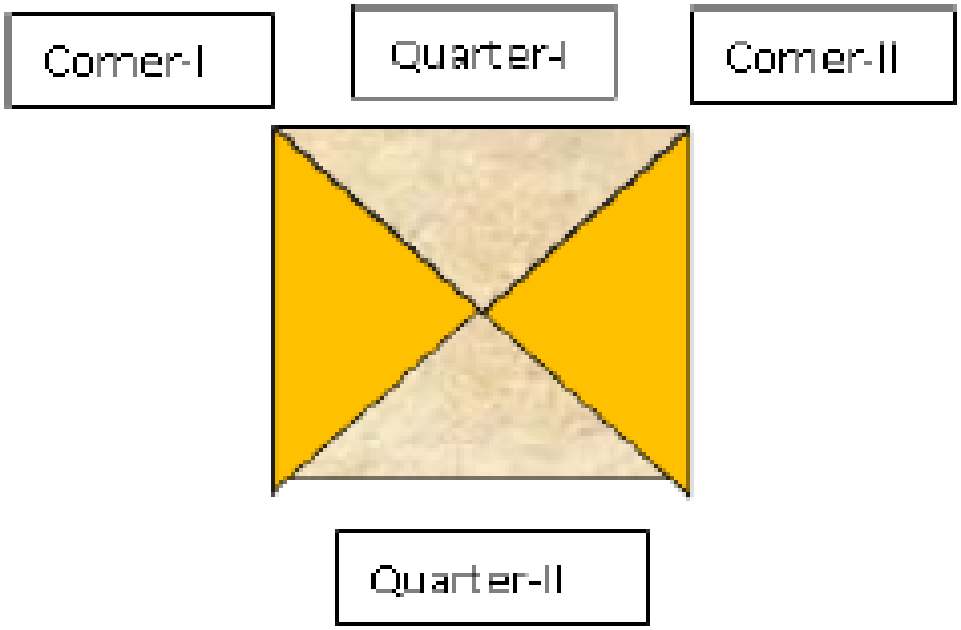


i) Block

- 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 25 mm portion of the butter core drawn.



- Butter in pats or rolls less than 500 gm: In this case take whole unit as sample
- Butter in pats more than 500 gm: Divide unit into four parts and take two opposite quarters as sample.



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 in an oven or water-bath not exceeding 39°C with 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. 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.
- 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.

Sampling of Ghee

- 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.

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

- Sample 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 300 gm.

Labelling of Samples for Analysis

- Shall be sealed or air-tight after filling and a label marked
- Sampling is generally done for Chemical analysis, Bacteriological analysis and Sensory analysis

Fore easy tractability the sample should bear the following information:

- 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, Agmark etc, 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.

Choice of Analytical Test

- There are array of methods for quantitative analysis and they are capable of achieving the desired analysis
- But to select one may depend on a variety of factors.
- These are as follows:

1. Speed: It should be less time consuming method.

2. Convenience: The method should not be difficult.

3. Accuracy/Precision: The method should be error free.

4. Sensitivity/Detection limits of the method: Higher the sensitivity better will be the results. And the method should be able to detect lower levels of the components in a food.

5. Selectivity: Non-interference of other compounds present in the sample

6. Availability of instruments/Specific apparatus

7. Amount of sample: The selected method should use only small amounts of sample.

Level of analysis/Nature of analysis

Depending of the level and nature of the analysis different methods are available considering for qualitative and quantitative analysis.

1. Low Cost of the method
2. Hazards free or Risk free method
3. The published literature should be available to choose or select a method.