
International Standard



707

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Milk and milk products — Methods of sampling

Lait et produits laitiers — Méthodes d'échantillonnage

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 707 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*.

It cancels and replaces ISO/R 707-1968, of which it constitutes a technical revision, and ISO 1193-1973, ISO 1194-1973 and ISO 3003-1974, the contents of which are incorporated in this International Standard.

NOTE — The methods specified in this International Standard have been developed jointly with the IDF (International Dairy Federation) and the AOAC (Association of Official Analytical Chemists, USA). The text as approved by these organizations will also be published by FAO/WHO (Code of principles concerning milk and milk products and associated standards), by the IDF and by the AOAC (Official Methods of Analysis).

Contents

	Page
0 Introduction	1
1 Scope and field of application	1
2 References	1
3 Definitions	1
Section one : General considerations	2
4 Administrative arrangements	2
5 Apparatus	2
6 Sampling technique	3
7 Preservation and storage of samples	3
8 Dispatch of samples	3
Section two : Methods of taking samples	5
9 Milk and liquid milk products	5
10 Evaporated milk, sweetened condensed milk and concentrated whey products	12
11 Jellified milk products and "set" fermented milk products	13
12 Edible ices and frozen products with milk constituents	14
13 Dried milk and dried milk products	15
14 Butter and related products	17
15 Anhydrous milkfat and related products	20
16 Cheese	21
Annex A — Routine sampling procedures for milk quality payment schemes	26
Annex B — Suppliers of sampling equipment for dairy products	28
Table 1 — Sample preservation, storage temperature, and minimum sample size	4
Table 2 — Dimensions of borers	15
Table 3 — Dimensions of butter triers	17
Table 4 — Dimensions of cheese triers	21

Figures

	Page
1 Recommended plunger for cans and buckets	8
2 Suitable plunger for road, farm and rail tanks	8
3 Suitable dipper for liquids	8
4 Sample extraction cylinder	9
5 Chamber probe	10
6 Probe or partial sample extractor	11
7 Suitable stirrer for mixing sweetened condensed milk in barrels	13
8 Thermally insulated box suitable for the transport of samples of edible ices ...	16
9 Dried-milk borers	18
10 Butter trier	19
11 Cheese trier	22
12 Sampling a cylindrical cheese by means of a trier	24
13 Sampling a cubic cheese by means of a trier	24
14 Sampling a block-shaped or loaf-shaped cheese by means of a trier	24
15 Sampling a spherical cheese, with flattened upper and lower sides, by means of a trier	24
16 Sampling a cheese having a circular cross-section and a mass over 2 500 g, by cutting out a sector	25
17 Sampling a cheese having a circular cross-section and a mass over 1 100 g, but less than 2 500 g, by cutting out a sector	25
18 Sampling by cutting out a piece from a block-shaped or loaf-shaped cheese in which the largest face is rectangular, but not square	25
19 Sampling by cutting out a piece from a block-shaped cheese in which the largest face is square	25

Milk and milk products — Methods of sampling

0 Introduction

Correct sampling is an operation that requires most careful attention. Emphasis cannot therefore be too strongly laid on the necessity of obtaining a properly representative sample.

The procedures described in this International Standard are recognized as good practice to be followed whenever practicable. However, it is impossible to lay down fixed rules to be followed in every case, and, however explicit, they cannot fully take the place of judgement, skill and experience. In particular, unforeseen circumstances may render some modifications desirable.

The samples obtained by the methods described in this International Standard are "laboratory samples" as defined in ISO 78/2, *Layouts for standards — Part 2: Standard for chemical analysis*.

1 Scope and field of application

This International Standard specifies methods of sampling milk and milk products for microbiological, chemical, physical and sensory analysis.

It is not applicable for the selection of a number of units from a consignment, nor does it apply to subsequent operations in the laboratory.

Instructions for sampling for milk quality payment schemes are given in annex A.

NOTE — The number of units to be selected for sampling by inspection by attributes may be determined according to IDF Standard 113, *Milk and milk products — Sampling — Inspection by attributes*. Sampling by inspection by variables will form the subject of a future International Standard.

2 References

ISO 3918, *Milking machine installations — Vocabulary*.

ISO 7002, *Agricultural food products — Layout for a standard method of sampling from a lot*.¹⁾

IDF 68 A, *Anhydrous milkfat, anhydrous butteroil or anhydrous butterfat, butteroil or butterfat, ghee (compositional standards)*.

3 Definitions

For the purpose of this International Standard, the definitions given in ISO 7002 apply. (See also clause 0.)

In relation to milking machine installations the definitions of ISO 3918 apply.

1) At present at the stage of draft.

Section one : General considerations

4 Administrative arrangements

NOTE — The following instructions are not necessarily applicable for routine sampling.

4.1 Sampling personnel

Sampling shall be performed by an authorized person, properly trained in the appropriate technique. The person shall be free from any infectious disease. Sampling for microbiological examination shall always be undertaken by a person experienced in the technique of sampling for microbiological purposes.

4.2 Representation of the parties concerned

The parties concerned, or their representatives, shall be given the opportunity to be present when sampling is performed.

4.3 Sealing and labelling of samples

Samples shall be sealed and a label attached, indicating the nature of the product and, at least, the identification number, name and signature of the person responsible for taking the samples. If necessary, additional information may be included, such as the purpose of sampling, the mass or volume of sample, and the unit from which the sample was taken.

4.4 Replicate samples

Unless otherwise agreed, samples shall be taken in duplicate and kept at the temperature indicated in table 1. They shall be made available immediately on request.

It is recommended that additional sets of samples be taken and retained for arbitration purposes, if agreed between the interested parties.

4.5 Preparation of a sampling report

Samples shall be accompanied by a report, signed by the authorized sampling person¹⁾ and countersigned by any witnesses present. The report shall give the following particulars :

- a) the place, date and time of sampling;
- b) the names and designations of the sampling personnel and of any witnesses;
- c) the precise method of sampling, if this differs from the instructions given in this International Standard;
- d) the nature and number of units constituting the consignment, together with their batch code markings, where available;

- e) the identification number and any code markings of the batch from which the samples were taken;
- f) the number of samples duly identified as to the batches from which they were taken;
- g) the place to which the samples are to be sent;
- h) if possible, the name and address of the producer or of the persons responsible for packing the product.

When appropriate, the report shall also include any relevant conditions or circumstances (for example the condition of the product containers and their surroundings, temperature and humidity of the atmosphere, the age of the product, method of sterilization of the sampling equipment, whether a preservative substance has been added to the samples), and any special information relating to the product being sampled, for example difficulty in achieving homogeneity of the product.

5 Apparatus

5.1 Sampling equipment

Sampling equipment shall be made of stainless steel, or of other suitable material of adequate strength, which does not bring about a change in the sample which could affect the results of subsequent examinations. The equipment shall be of sufficiently robust construction to prevent distortion in use. They shall, however, be sufficiently light to enable the operator to move them rapidly through the product. If solder is used in the manufacture of the equipment, it shall be capable of withstanding sterilization at 180 °C. All surfaces shall be smooth and free from crevices. All corners shall be rounded.

Sampling equipment shall comply with the particular requirements appropriate to the product sampled (see section two).

5.1.1 Sampling for microbiological examination

Sampling equipment shall be thoroughly clean and, if not already sterile, shall be sterilized. If possible, sterilization shall be performed by one of the following methods :

- a) exposure to hot air at 170 to 175 °C for not less than 1 h;
- b) exposure to steam at 121 ± 1 °C for not less than 20 min in an autoclave. The equipment shall be dry when used.

After sterilization, equipment may be stored prior to use, if kept under sterile conditions.

If, in a particular situation, sterilization by the above methods is impossible, the following alternative methods, which should be

1) In some countries, it is the practice to employ a sworn person for sampling.

regarded as secondary methods only, are recommended, provided that the sampling equipment is used immediately after sterilization :

- c) exposure to saturated steam at 100 °C for 1 h;
- d) immersion in boiling water for at least 1 min;
- e) immersion in 70 % (V/V) ethanol solution and ignition to burn off the ethanol;
- f) exposure to a suitable flame so that all working surfaces come into contact with the flame.

5.1.2 Sampling for chemical and/or physical analysis

Sterile equipment is desirable, but in any case, the equipment shall be clean and dry and shall not influence the properties and composition of the product.

5.1.3 Sampling for sensory examination

Sterile equipment is desirable, but in any case, the equipment shall be clean and dry and shall not affect the odour or flavour of the product.

5.2 Sample containers

Sample containers and closures shall be of materials and construction which adequately protect the sample and which do not bring about a change in the sample which could affect the results of subsequent analyses or examinations. Materials which are appropriate include glass, some metals and some plastics. The containers should preferably be opaque. If transparent, filled containers shall be stored in a dark place.

Containers and closures shall be clean and dry and either sterile, or suitable for sterilization by one of the methods described in 5.1.1 if the sample is to be examined microbiologically. The shape and capacity of the containers shall be appropriate to the particular requirements for the product to be sampled (see section two).

Single-service plastic containers as well as aluminium foil (sterile and non-sterile) may be used. Suitable plastic bags, with appropriate methods of closure, may also be used.

Containers other than plastic bags shall be securely closed, either by means of a suitable stopper or by means of a screw-cap of metal or plastic material, having, if necessary, a liquid-tight plastic liner which is insoluble, non-absorbent and greaseproof, and which will not influence the odour, flavour, properties or composition of the sample.

If stoppers are used, they shall be made from, or covered with, non-absorbent, flavourless material.

Containers for solid, semi-solid or viscous products shall be wide-mouthed.

6 Sampling technique

Samples for microbiological examinations shall be taken first, using aseptic techniques and sterilized equipment and containers (see 5.1.1).

The precise method of sampling and the mass or volume of product to be taken varies with the nature of the product and the purpose for which samples are required (see section two).

The sample container shall be closed immediately after sampling.

For products in small retail containers, the sample shall consist of the contents of one or more unopened containers.

If necessary provide means, for example a temperature control sample, for the temperature of samples to be checked on arrival at the laboratory.

7 Preservation and storage of samples

Preservatives shall normally not be added to samples intended for microbiological or sensory examination.

A suitable preservative may be added to some milk products, provided that :

- a) an instruction to do so is issued by the testing laboratory;
- b) the preservative is of a nature that does not interfere with subsequent analyses;
- c) the nature and quantity of preservative are indicated on the label and in the sampling report.

Table 1 indicates whether preservatives may be added and gives the recommended storage temperatures before transport for samples of various milk products. The storage temperature shall be attained as quickly as possible after sampling.

The storage time before transport shall be as short as possible (see clause 8). Storage temperatures other than those recommended in table 1 may be used if requested by the testing laboratory (for example higher temperatures may be requested for some cheeses which could be adversely affected by the temperatures given in table 1).

The time and temperature shall be considered in combination and not independently.

8 Dispatch of samples

Samples shall be dispatched to reach the testing laboratory as soon as possible after sampling (preferably within 24 h). During transit, precautions shall be taken to prevent exposure to contaminating odours, to direct sunlight and to temperatures outside the ranges given in table 1 or outside the ranges requested by the testing laboratory.

Table 1 — Sample preservation, storage temperature, and minimum sample size

Product	Preservatives permitted for samples intended for chemical and physical analysis	Storage temperature before and during transport °C	Minimum sample size ¹⁾
Non-sterilized milk and liquid milk products	Yes	0 to 4	200 ml or 200 g
Sterilized milk, UHT milk and sterilized liquid milk products in unopened containers	No	Ambient, 25 max.	200 ml or 200 g
Sterilized milk, UHT milk and sterilized liquid milk products if removed from a production pack	Yes	0 to 4	200 ml or 200 g
Evaporated milk in unopened containers and sweetened condensed milk, and concentrated whey	No	Ambient, 25 max.	200 g
Jellified milk products and "set" fermented milk products	No	0 to 4	200 g
Edible ices and frozen products with milk constituents	No	- 18 or below	100 or 200 g
Dried milk and dried milk products	No	Ambient, 25 max.	100 or 200 g
Butter and whey butter	No	0 to 4 (in the dark)	100 or 250 g or 2 kg
Anhydrous milk fat and related products	No	0 to 4 (in the dark)	100 g
Cheese and cheese products	No	0 to 4	100 or 200 g

1) Varies according to the tests required and the type of product.

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Section two : Methods of taking samples

9 Milk and liquid milk products

9.1 Applicability

The instructions given in this clause are applicable to whole, partly skimmed and skimmed milk, flavoured milk, buttermilk, cream, liquid fermented milks and liquid whey.

They are not applicable to the various types of evaporated (unsweetened condensed) milk and sweetened condensed milks, to liquid milkfat products, as described in IDF Standard 68A, and to jellified fermented milks.

Furthermore, they are not applicable to raw milk for quality payment purposes (see annex A).

9.2 Apparatus¹⁾ (see also 5.1)

9.2.1 Plungers and agitators

Plungers and agitators, for mixing liquids in bulk, shall be of sufficient area to produce adequate disturbance of the product, without causing the development of a rancid flavour. In view of the different shapes and sizes of containers, no specific design of plungers can be recommended for all purposes, but they shall be designed in such a way as to avoid scratching the inner surface of the container.

A form of plunger recommended for mixing liquids in buckets or in cans is shown in figure 1. It comprises a disc of diameter 150 mm, perforated with six holes, each of diameter 12,5 mm on a pitch circle of diameter 100 mm. The disc is fixed centrally to a metal rod, the other end of which forms a loop handle. The length of the rod, including the handle, is approximately 1 m.

A suitable plunger for use for small tanks is shown in figure 2. It comprises a rod of length not less than 2 m, fitted with a disc of diameter 300 mm, perforated with twelve holes, each of diameter 30 mm, on a pitch circle of diameter 230 mm.

For mixing the contents of large vessels or storage, road and rail tanks, mechanical agitation is advisable.

9.2.2 Dippers and extractors

9.2.2.1 Dippers

A dipper of suitable size and shape for collecting samples is shown in figure 3. It is fitted with a solid handle of length at least 150 mm. The capacity of the dipper is not less than 50 ml. It is an advantage for the handle to be bent over. The tapered form of the cup permits nesting of the dippers.

Alternatively, a dipper may be used which is of similar capacity, but which has parallel sides graduated into five equal sections for use in proportionate sampling of consignments held in more than one container.

9.2.2.2 Extractors

9.2.2.2.1 Sample extraction cylinder

A suitable sample extraction cylinder is shown in figure 4. It consists of two perfectly adjusted concentric tubes, one of which revolves inside the other, and which are operated by a control in the head of the cylinder which turns through 90°. Along the length of the two concentric tubes, in opposing positions, are slots 50 mm long and 6 mm wide, with a separation of 20 mm.

When the inner tube is turned to one extreme, the slots in both tubes coincide in an open position which allows liquid to flow into the cylinder. When the head is turned through 90° to the other extreme, the slots do not coincide and the cylinder is closed. The top and bottom ends of the cylinder are fitted with screw-caps for easy cleaning. The length is variable, depending on the depth of the containers in which the cylinder is to be used, but in general, approximately 1 m is sufficient.

9.2.2.2.2 Chamber probe

A suitable chamber probe is shown in figure 5. It consists of a half-round tube closed by a strip or plate which slides along grooves in the upper surface of the tube. The inner chamber is divided into compartments which increase in size from the lower end of the probe. The total length may vary, according to the depth of the container in which it is to be used, but, in general, approximately 1 m is sufficient.

The probe is inserted into the container until it touches the bottom, with the chamber closed. With the probe in a vertical position, the closing strip is gradually raised until it reaches the surface level of the liquid. Immediately the strip is lowered and the probe removed. By placing it in a horizontal position and opening the chamber, it is easy to observe the samples taken at different depths, which are kept separate by the partitions dividing the total length of the chamber.

The chamber is emptied by gently tipping the open probe over the sample container.

With this probe, samples can be taken to a depth of 5 mm from the bottom of a container.

9.2.2.2.3 Probe or partial sample extractor

A suitable probe or partial sample extractor is shown in figure 6. It comprises a well-sealed compartment allowing sampling of a particular section of the tank or vessel. It allows samples to be taken at 10 mm or less from the bottom of the product container. The valve shall be perfectly adjusted so that the extractor can be removed without loss of the contents.

The valve can be opened easily by hand, by means of a cord. The extractor should be suspended from a cable which marks the depth reached by the lower end of the probe. It should weigh at least 3 kg.

1) For a list of suppliers of sampling equipment, see annex B.

9.2.3 Sample containers

See 5.2.

The capacity of the sample containers shall be such that they are almost completely filled by the sample and allow proper mixing of the contents before testing, but avoid churning during transport.

9.3 Procedure

9.3.1 General

9.3.1.1 Thoroughly mix all liquids, by plunging or stirring, by mechanical agitation, by pouring from one product container to another, or by the use of clean compressed air (see the notes to 9.3.3.2.6) until sufficient homogeneity is obtained. The equipment described in 9.2.1 may be used.

9.3.1.2 If it proves difficult to obtain sufficient homogeneity, take a sample of not less than 200 ml from appropriate portions of the container. Take the various subsamples from the bulk according to a plan which gives a total sample representative of the bulk.

The equipment described in 9.2.2.3 may be used.

Note on the label and in the sampling report if the sample is a mixture of subsamples.

9.3.1.3 Take the sample immediately after mixing by means of a dipper or extractor.

The size of sample shall not be less than 200 ml.

9.3.1.4 For taking samples from shallow tanks, trucks and large vessels, the equipment described in 9.2.2.2.1 and 9.2.2.2.2 may be used. The first is preferably for homogeneous products and the second for products which are not homogeneous.

If the container is deep, the equipment described in 9.2.2.3 may be used. In the case of homogeneous products, take samples mixed together. If the product is not homogeneous, take samples at depths of every 10 to 15 cm and shake and mix these together.

9.3.1.5 For small retail containers, the contents of intact and unopened containers constitute the sample.

Take one or more retail containers to obtain a sample of not less than 200 ml, if not otherwise prescribed by the testing laboratory.

9.3.2 Sampling for microbiological examination

Take a sample of not less than 200 ml.

Take samples for microbiological examination first and, whenever possible, from the same product containers as those taken for chemical and physical analysis and for sensory examination.

Sterilize the sampling equipment and containers as described in 5.1.1.

Proceed as described in 9.3.3 using aseptic techniques, but do not take a sample for microbiological examination from any discharge outlet.

9.3.3 Sampling for chemical and/or physical analysis and for sensory examination

9.3.3.1 Sample size

Take a sample of not less than 200 ml.

9.3.3.2 Whole milk

9.3.3.2.1 Milk from individual animals

It is customary at the start of milking to take by hand a small quantity of milk, from each quarter, and to place this in a strip cup for examination; this foremilk is usually discarded. The strippings of milk induced from the animal by manipulation of the udder at the end of milking are referred to as hand strippings in the case of hand milking or when taken after milking machine clusters have been removed, and as machine strippings when taken when the milking machine clusters are still in position.

The sample taken shall be representative of the milk of the animal when milked in the usual manner.

9.3.3.2.1.1 Hand milking

Place all the milk from the animal, including strippings but excluding foremilk, in one container and thoroughly mix before sampling.

9.3.3.2.1.2 Machine milking

Admit air through the teat cups at the conclusion of milking the animal to ensure the transfer to the receiving container of all the milk trapped in the claw-piece, long milk tubes, etc.

The following shall be noted, according to the case :

a) bucket milking and direct-to-can milking : add any hand strippings from the animal to the rest of the milk and thoroughly mix the whole by transfer, stirring or plunging prior to sampling;

b) recorder jar : transfer the whole of the milk from the recorder jar to a bucket, add any hand strippings, and take a sample as in the bucket system. Where hand stripping is not practised, and a recorder jar is fitted with a sampling attachment, the milk and machine strippings may be mixed by allowing clean air to bubble through the milk at a sufficient rate to ensure thorough mixing. This may be achieved either by a continuous airbleed at a relatively slow rate throughout milking, or alternatively by vigorous aeration at the end of milking for a minimum period of 10 s, care being taken to

avoid loss of milk from the recorder jar. Withdraw any milk in the vicinity of the sampling point which may not have thoroughly mixed before taking the sample;

c) milk meter : a sample representative of the milk may be taken from the proportion of the milk retained in the meter by emptying the metering tube into a suitable container and mixing the contents by stirring. This method shall not be used when hand stripping is practised. Furthermore, as only a proportion of the yield is available for sampling, this method may be less reliable than other methods and it is necessary to verify for the particular milk meter, under the particular conditions of use, that a representative sample is obtained.

9.3.3.2.2 Sampling from small vessels — Milk buckets and cans

Thoroughly mix the milk, either by transfer, stirring or plunging.

9.3.3.2.3 Sampling from a weighbowl

It is essential for the milk to be adequately mixed in the weighbowl if a representative sample is to be obtained. Some degree of mixing will occur when milk is tipped into the weighbowl, the extent being determined by the design of the weighbowl and the manner of tipping. It is essential to supplement this by sufficient hand or mechanical agitation to ensure even distribution of fat. The amount of additional mixing shall be determined by experiment. The samples shall normally be taken from the weighbowl itself. When the volume of the consignment to be sampled exceeds the capacity of the weighbowl, a sample representative of the whole consignment shall be obtained in accordance with 9.3.3.2.5.

9.3.3.2.4 Sampling from refrigerated farm milk tanks or vats

Mechanically agitate the milk until sufficient homogeneity is obtained (at least 5 min). If the tank is equipped with a periodical, time-programmed agitation system, sampling may be carried out after only a short duration of agitation (1 to 2 min).

If the volume of milk is less than 15 % of the capacity of the tank, perform the agitation manually.

9.3.3.2.5 Sampling a divided bulk

Where the quantity of milk to be sampled is in more than one container, take a representative quantity from each container after mixing the contents, and note the quantity of milk which each sample relates. Unless the samples from each container are to be tested individually, mix portions of these representative quantities in amounts which are proportional to the quantity in the container from which each was taken. Take sample(s) from these bulked proportionate amounts after mixing.

9.3.3.2.6 Sampling from large vessels — Storage, rail and road tanks

In each case, thoroughly mix the milk by an appropriate method before sampling, for example by mechanical agitation.

The extent of mixing shall be appropriate to the period of time over which the milk has been at rest. The efficiency of the method of mixing applied in any particular circumstances shall be demonstrated as being adequate for the purposes of the analysis envisaged; the criterion of mixing efficiency is the repeatability of analytical results from samples taken either from different parts of the consignments, or from the outlet of the tank at intervals during discharge. A method of mixing shall be considered efficient if the difference in fat content between two samples, taken under these conditions, is less than 0,1 %.

Mixing of the contents of large vessels or storage, rail and road tanks can be performed

- by a mechanical agitator built into the tank and driven by an electric motor
- by a propeller or agitator driven by an electric motor and placed on the manhole with the agitator suspended in the milk
- in the case of rail or road tankers by recirculation of the milk through the transfer hose attached to the tanker unloading pumps and inserted through the manhole
- in the case of vertical tanks by clean compressed air.

NOTES

- 1 When clean compressed air is recommended, it is necessary to use filtered compressed air from which all contaminants (including oil, water and dust) have been excluded. The possibility of microbiological contamination should not be forgotten.
- 2 Minimal air pressure and volume should be used to prevent the development of rancid flavour.

In a large vessel, with a bottom discharge outlet, there may be, at the discharge point, a small quantity of milk which is not representative of the whole contents even after mixing. Accordingly, samples should preferably be taken through a manhole. If samples are taken from the discharge outlet, discharge sufficient milk to ensure that the samples are representative of the whole.

9.3.3.3 Skimmed milk and whey

Use the method described for whole milk in 9.3.3.2.2, 9.3.3.2.5 and 9.3.3.2.6.

9.3.3.4 Buttermilk, fermented milk, flavoured milk

Use the method described for whole milk in 9.3.3.2.2, 9.3.3.2.5 and 9.3.3.2.6 and take a sample before fat or other solid matter has had time to separate.

9.3.3.5 Cream

When using the plunging method for mixing cream, use the plunger in such a manner that the whole of the cream at the bottom of the container is thoroughly agitated and mixed with the upper layer. To avoid foaming, whipping or churning of the cream, do not raise the disc of the plunger above the surface of the cream during plunging. The equipment described in 9.2.1 (see figures 1 and 2) may be used.

9.4 Preservation, storage and dispatch of samples

See clauses 7 and 8.

Dimensions in millimetres

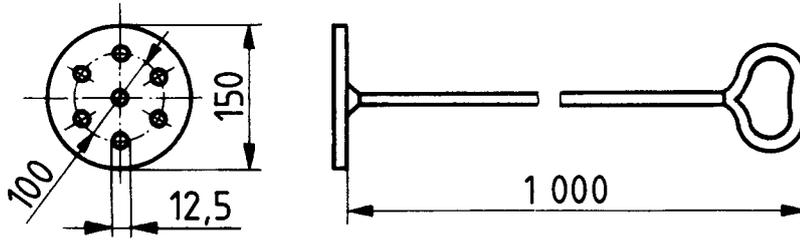


Figure 1 – Recommended plunger for cans and buckets

Dimensions in millimetres

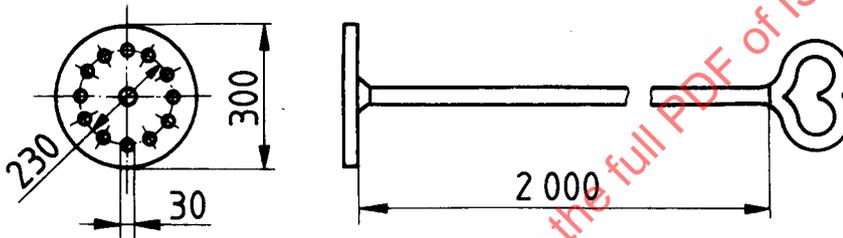


Figure 2 – Suitable plunger for road, farm and rail tanks

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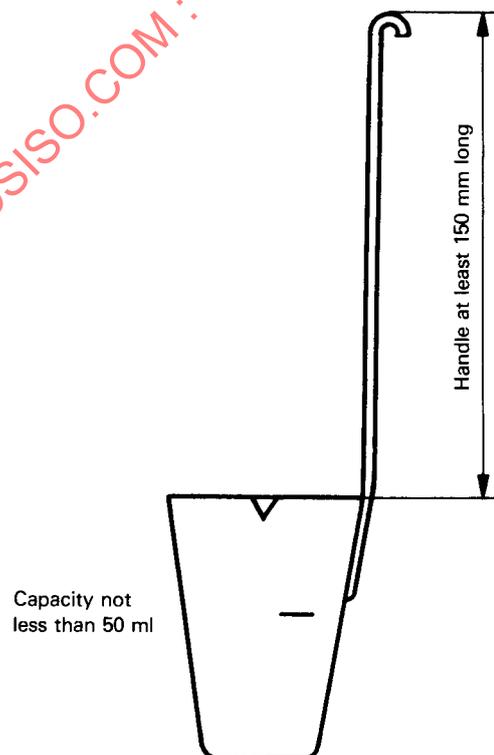


Figure 3 – Suitable dipper for liquids

Dimensions in millimetres

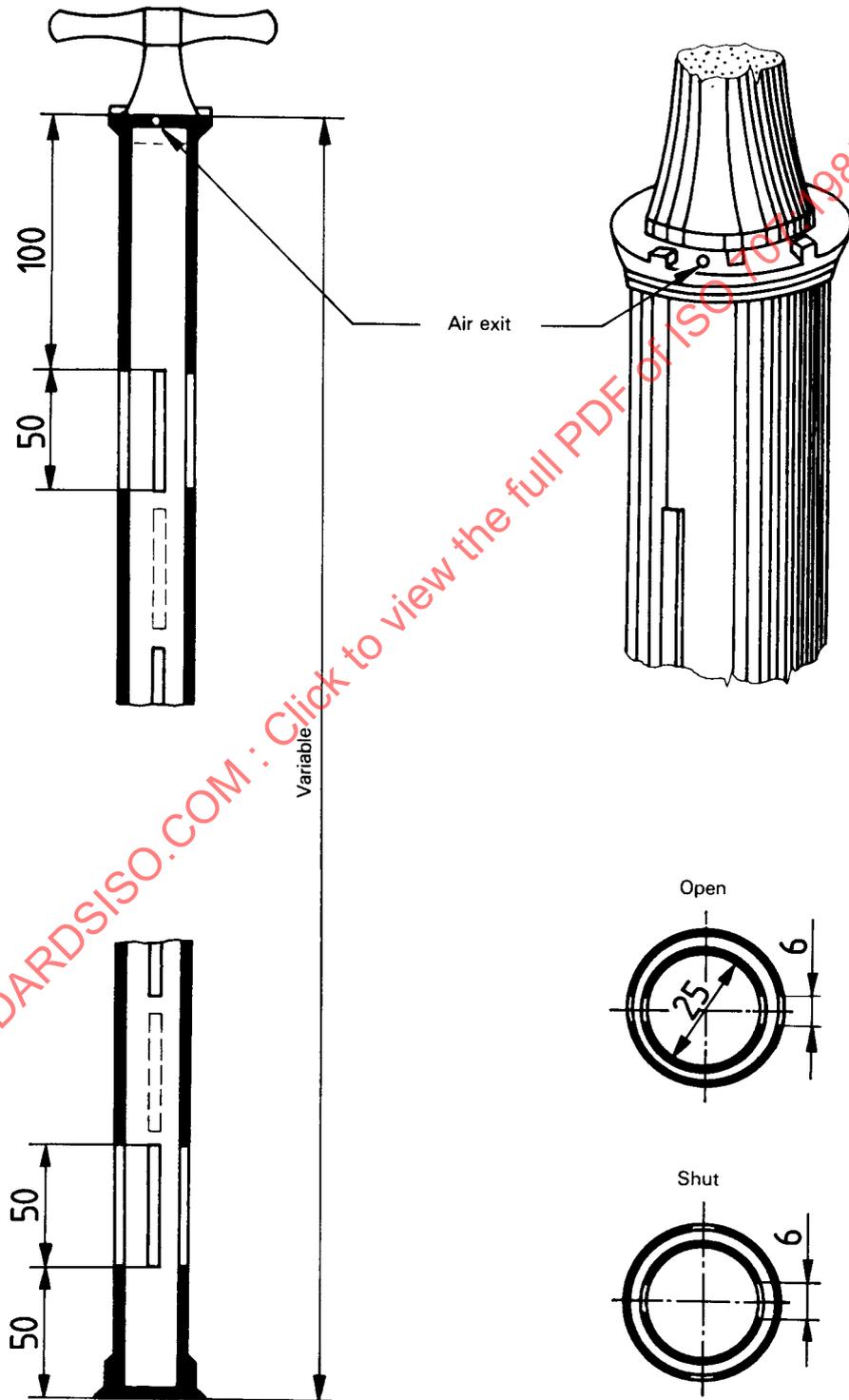


Figure 4 — Sample extraction cylinder

Dimensions in millimetres

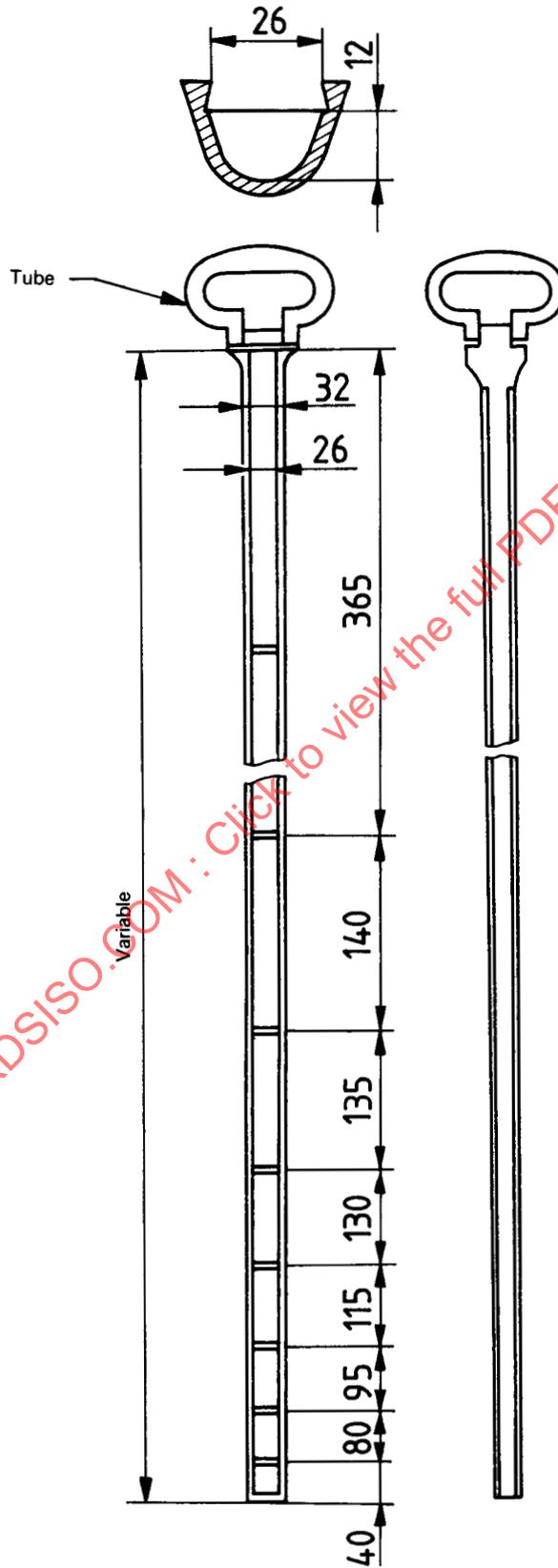


Figure 5 – Chamber probe

Dimensions in millimetres

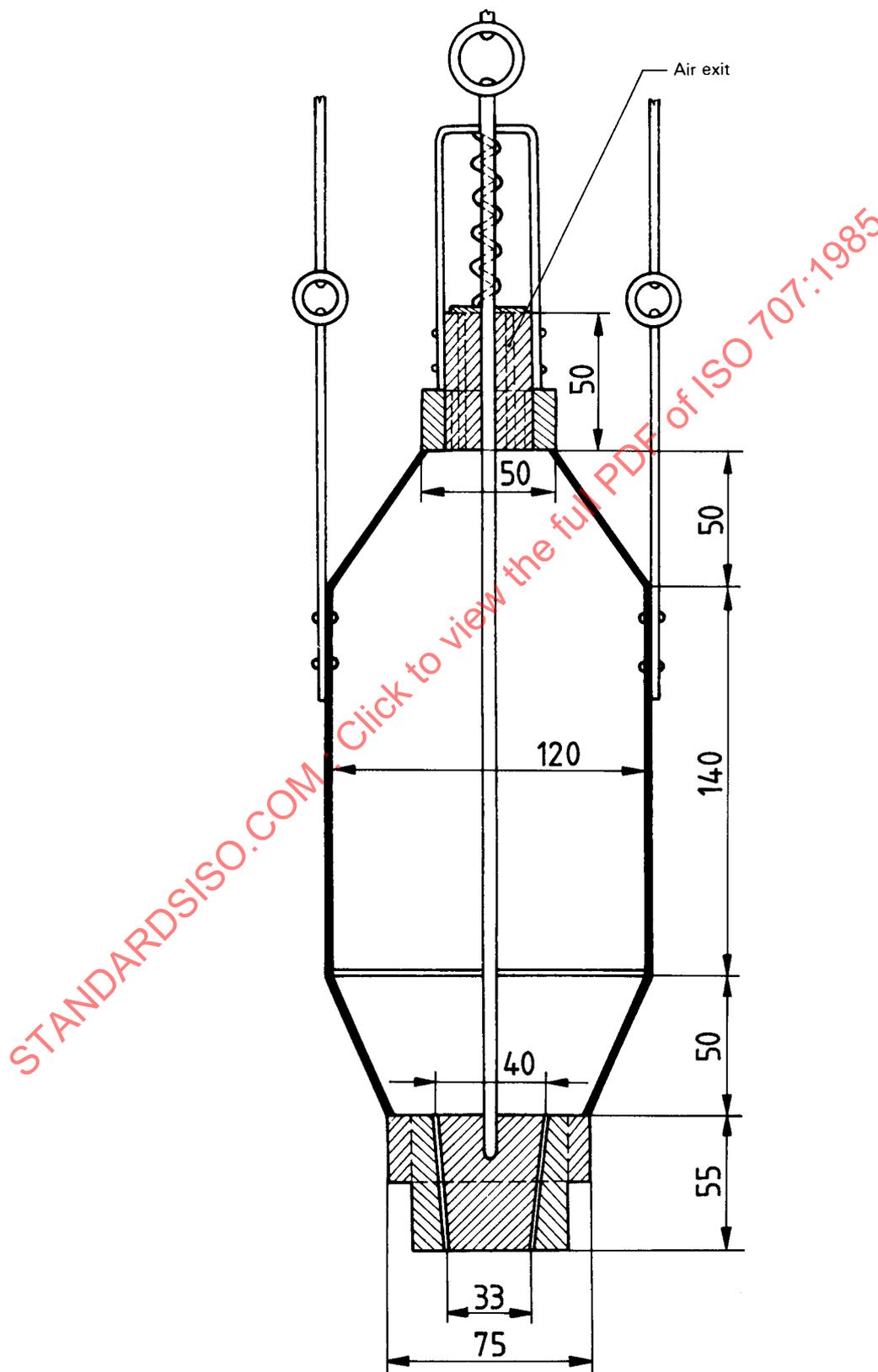


Figure 6 — Probe or partial sample extractor

10 Evaporated milk, sweetened condensed milk and concentrated whey products

10.1 Applicability

The instructions given in this clause are applicable to all types of evaporated milks, all types of sweetened condensed milks and all types of concentrated whey products.

10.2 Sampling equipment (see also 5.1)

10.2.1 Plungers and agitators (see 9.2.1).

10.2.2 Stirrers, broad bladed, of sufficient length to reach the bottom of the product container, and preferably having one edge shaped to the contour of the container. (A suitable stirrer is shown in figure 7.)

10.2.3 Dippers (see 9.2.2.1).

10.2.4 Rods, round, about 1 m long, of diameter about 35 mm.

10.2.5 Containers for subsampling, of capacity 5 l, wide-mouthed.

10.2.6 Spoons or spatulas, broad-bladed.

10.2.7 Sample containers.

See 5.2.

The capacity of the sample containers shall be such that they are almost completely filled by the sample and allow proper mixing of the contents before testing.

10.3 Procedure

10.3.1 Evaporated milk

10.3.1.1 Sampling for microbiological examination

Take a sample of not less than 200 g.

Take samples for microbiological examination first and, whenever possible, from the same product containers as those taken for chemical and physical analysis and for sensory examination.

Sterilize the sampling equipment and containers as described in 5.1.1.

Proceed as described in 10.3.1.2 using aseptic techniques.

10.3.1.2 Sampling for chemical and/or physical analysis and for sensory examination

Take a sample of not less than 200 g.

Thoroughly mix the product by plunging or stirring, by mechanical agitation, by pouring from one container to another, or by the use of clean compressed air (see the notes to 9.3.3.2.6), until sufficient homogeneity is obtained.

Take the sample immediately after mixing by means of a dipper (10.2.3). If it proves difficult to obtain sufficient homogeneity, take samples from different portions of the product container until not less than 200 g is obtained.

Note on the label and in the sampling report if the sample is a mixture of subsamples.

10.3.1.3 Sampling products packed in small retail containers

The contents of the intact and unopened containers constitute the sample. Take one or more containers to provide a sample of not less than 200 g, if not otherwise prescribed by the testing laboratory.

10.3.2 Sweetened condensed milk and concentrated whey products

10.3.2.1 Sample size

Take a sample of not less than 200 g.

10.3.2.2 General

The sampling of bulk containers of sweetened condensed milk may be a matter of extreme difficulty, particularly when the product is not homogeneous and is highly viscous. Problems of sampling may arise through the presence of large crystals of sucrose or lactose, or through precipitation of various salts which may occur throughout the body of the product or adhere to the walls, or through the presence of lumpy matter. Such conditions will become apparent when a sampling rod is introduced into the product container and is withdrawn after exploring as large a volume of the container as possible. Provided that the size of sugar crystals is not larger than 6 μm , difficulties in sampling should not be experienced from this cause. When the product is not homogeneous, note this fact on the label and in the sampling report. Since sweetened condensed milk is frequently stored at atmospheric temperature, it is recommended that, in order to obtain a representative sample, the contents are brought to a temperature of not less than 20 °C.

Crystallized concentrated whey in storage tanks cannot be sampled representatively unless the tank is designed for, and equipped with, a power driven agitator. Sampling shall not take place until the lactose crystals have been dispersed.

10.3.2.3 Sampling for microbiological examination

See 10.3.1.1.

Thoroughly clean, sterilize and rinse with cold sterile water the outside end of the product container or of the drum, if it is an end-opening type, or the locality adjacent to and including the bung hole of an enclosed type of drum, before opening the container or removing the end-cover or bung.

Proceed as prescribed in 10.3.2.4 using aseptic techniques.

10.3.2.4 Sampling for chemical and physical analysis and for sensory examination

10.3.2.4.1 In the case of open-ended containers, remove one end of the container, which shall have been previously thoroughly cleaned and dried to prevent foreign matter falling into the bulk during the opening process. Mix the contents by means of a stirrer (see figure 7). Scrape the blade round the sides and bottom of the container to remove any adhering product and thoroughly mix the contents by a combination of rotary and vertical movements, with the stirrer inclined diagonally, taking care to avoid incorporation of air in the sample. Withdraw the stirrer and transfer the condensed milk or whey adhering to it into a container (10.2.5) by means of a spatula or spoon (10.2.6). Repeat the mixing and withdrawal until 2 to 3 l have been collected. Mix this volume until homogeneous, and take a sample of not less than 200 g.

10.3.2.4.2 In the case of enclosed drums with bungs at the end, or at the side, sampling through the bung hole is suitable only with condensed milk which flows readily and is of uniform consistency (see 10.3.2.2). Mix the contents by inserting a rod (10.2.4) through the bung hole, and after exploring and stirring as far as possible in all directions, withdraw the rod and prepare a sample as described in 10.3.2.4.1.

Alternatively, allow the contents to run into a suitable container, taking care that as much of the contents as possible are recovered from the drum. After stirring with a stirrer, collect the sample as described in 10.3.2.4.1.

10.3.2.5 Sampling products packed in small retail containers

The contents of the intact and unopened containers constitute the sample. Take one or more containers to provide a sample of

not less than 200 g, unless otherwise instructed by the testing laboratory.

10.4 Preservation, storage and dispatch of samples

See clauses 7 and 8.

11 Jellified milk products and "set" fermented milk products

11.1 Applicability

The instructions given in this clause are applicable, among others, to milk puddings and fermented milk products of the "set" or semi-liquid type, with or without addition of stabilizers, starch, fruits, nuts or other ingredients, the jellified texture being the common property.

11.2 Sampling equipment (see also 5.1)

11.2.1 Plungers or agitators (see 9.2.1).

11.2.2 Dippers (see 9.2.2.1).

11.2.3 Sample containers

See 5.2 and 11.3.1.

The capacity of the sample containers shall be such that they are almost completely filled by the sample and allow proper mixing of the contents before testing.

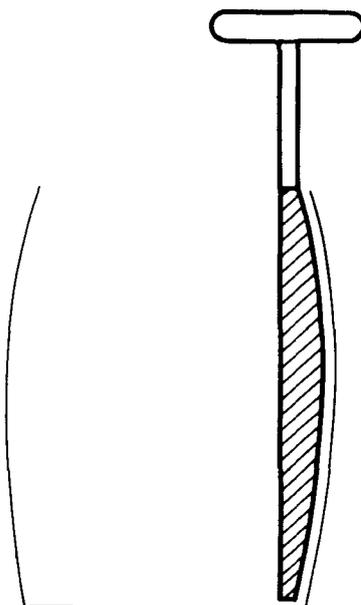


Figure 7 — Suitable stirrer for mixing sweetened condensed milk in barrels

11.3 Procedure

11.3.1 General

The sampling of jellified products from large containers may be a matter of extreme difficulty, particularly when the product is highly viscous or if various kinds of constituents, such as fruits which may settle at the bottom of the container, have been added.

In most cases, however, sampling will have to be carried out from lots which consist of small retail containers.

11.3.2 Sampling for microbiological examination

Take a sample of not less than 200 g.

Take samples for microbiological examination first and, whenever possible, from the same product containers as those taken for chemical and physical analysis and for sensory examination.

Sterilize the sampling equipment and containers as described in 5.1.1.

Proceed as described in 11.3.3 using aseptic techniques.

11.3.3 Sampling for chemical and/or physical analysis and for sensory examination

Take a sample of not less than 200 g.

For physical and sensory examination, do not stir the contents of small retail containers. If the product is contained in a large container, for example more than 2 kg, thoroughly mix the contents of the container by plunging or stirring or by mechanical agitation until sufficient homogeneity is obtained. Avoid foaming, whipping, whey-separation and settling of ingredients.

Take the sample immediately after mixing by means of a dipper (11.2.2). If it proves difficult to obtain sufficient homogeneity, take samples from different portions of the product container until a quantity of not less than 200 g is obtained.

Note on the label and in the sampling report if the sample is a mixture of subsamples.

11.3.4 Sampling products packed in small retail containers

In most cases, sampling will have to be carried out from lots which consist of small retail containers, as the jellified texture will be formed only in the last stage of manufacture. In that case, the sample shall consist of the contents of one or more unopened containers, to provide a mass of not less than 200 g if not otherwise prescribed by the testing laboratory.

11.4 Preservation, storage and dispatch of samples

See clauses 7 and 8.

12 Edible ices and frozen products with milk constituents

12.1 Applicability

The instructions given in this clause are applicable to edible ices, frozen cream, frozen yogurt and frozen confections.

12.2 Sampling equipment (see also 5.1)

12.2.1 Borers, of sufficient length to reach the bottom of the product container.

The borers described in 12.2.1 (see figure 9) are suitable.

12.2.2 Spoon, knife or spatula, broad-bladed.

12.2.3 Boring device, for products which are deep-frozen in a large, solid block.

A boring device such as a bit or hollow tube driven by an appropriate electric or mechanical drill is suitable.

12.2.4 Sample containers

See 5.2.

The sample containers shall be placed in a thermally insulated transport box which has been suitably refrigerated (for example with solid carbon dioxide) for not less than 30 min before use. (A suitable box is shown in figure 8.)

Place sufficient refrigerating material in the transport box with the samples to ensure that the latter arrive at the laboratory in suitable condition.

12.3 Procedure

12.3.1 Sampling for microbiological examination

Take a sample of not less than 100 g.

Take samples for microbiological examination first and, whenever possible from the same product containers as those taken for chemical and physical analysis and for sensory examination.

Sterilize the sampling equipment and containers as described in 5.1.1.

Using a sterilized spoon, knife or spatula (12.2.2), remove the surface layer of the product from the sampling area to a depth of at least 10 mm. With a sterilized instrument [borer (12.2.1), boring device (12.2.3)], take a sample of not less than 100 g, if possible from near the centre of the container using the same technique as in 12.3.2 but taking aseptic precautions.

Transfer the sample as quickly as possible into the sterile sample container which should be immediately closed, taking aseptic precautions. Place the container immediately in the thermally insulated, suitably refrigerated transport box (12.2.4).

If required, obtain a "surface sample" of the product by lightly scraping the entire surface of the product container using a sterilized spoon or spatula to a minimum uniform depth to give a sample of not less than 100 g.

When information is required regarding the microbiological condition of the product as presented to the customer, or the care used in vending operations on the part of the retailer, use the server normally used for dispensing for the purpose of sampling.

When sampling individual products, proceed as described in 12.3.2.2 using sterile sampling equipment and containers, and using aseptic techniques.

12.3.2 Sampling for chemical and/or physical analysis and for sensory examination

12.3.2.1 General

Take a sample of not less than 200 g.

Pass the clean and dry borer through the product, the slit being oriented downwards and an even rate of penetration being used. When the borer reaches the bottom of the container, rotate it through 180°, withdraw it and discharge the contents into the sample container.

Use one or more borers to provide a sample of not less than 200 g. Immediately close the sample container, once sampling is completed, and place it in the refrigerated transport box.

12.3.2.2 Individual products

12.3.2.2.1 Products in packages, tubs, etc.

If the sample is to be subdivided, take the required number of equal parts of the product. Transfer one part from each product container into a separate sample container. Repeat this operation using further units until each sample container contains the required amount of sample.

12.3.2.2.2 Edible ices in small packages, individual portions

If possible, collect and dispatch the samples in their original containers, keeping the samples deep-frozen at all times until analysed. Otherwise, follow the general procedure.

12.3.2.2.3 Multi-layered ices, ripple ices and ices containing nuts, fruits, chocolate chips, etc.

The sample unit shall be the complete unit offered for sale as it is not possible to subdivide many of the ices of more complex composition representatively.

12.3.2.2.4 Stick confections

Place the complete unit offered for sale in the sample container together with the wrapper and stick, if provided, or, alternatively, after having discarded the wrapper and having cut the stick nearest to the confection.

12.3.2.2.5 Soft ice

Soft ice is freshly frozen ice which is normally sold directly from the freezer. When a sample is taken from the freezer, fill the requisite number of sample containers in succession from the outlet of the freezer while it is continuing to operate.

12.4 Preservation, storage and dispatch of samples

See clauses 7 and 8.

13 Dried milk and dried milk products

13.1 Applicability

The instructions given in this clause are applicable to dried milk with different fat contents, dried whey, casein, caseinates, coprecipitates and other powders with high milk protein contents. The method described is also applicable to cheese powder and to lactose in powder form.

The methods described are not suitable for powders in large bulk containers. Samples of powder in such containers should be obtained by bulking a number of small samples taken during loading or unloading of the container to allow access to the entire consignment.

13.2 Sampling equipment (see also 5.2)

13.2.1 Borers, of sufficient length to reach the bottom of the product container. Borers suitable for sampling containers up to approximately 50 kg, for example sacks, are shown in figure 9. Dimensions for guidance are given in table 2.

Table 2 — Dimensions of borers

Dimensions in millimetres

	Type A long	Type B short
Length of blade	800	400
Thickness of metal of blade	1 to 2	1 to 2
Inner diameter of blade at point	18	32
Inner diameter of blade at grip or stem	22	28
Slit width at point	4	20
Slit width at grip or stem	14	14

The blade and stem should preferably be made of polished stainless steel.

The protruding edge of the blade of type A shall be sufficiently sharp to serve as a scraper and the point of the blade shall be sufficiently sharp to facilitate sampling.

Dimensions in millimetres

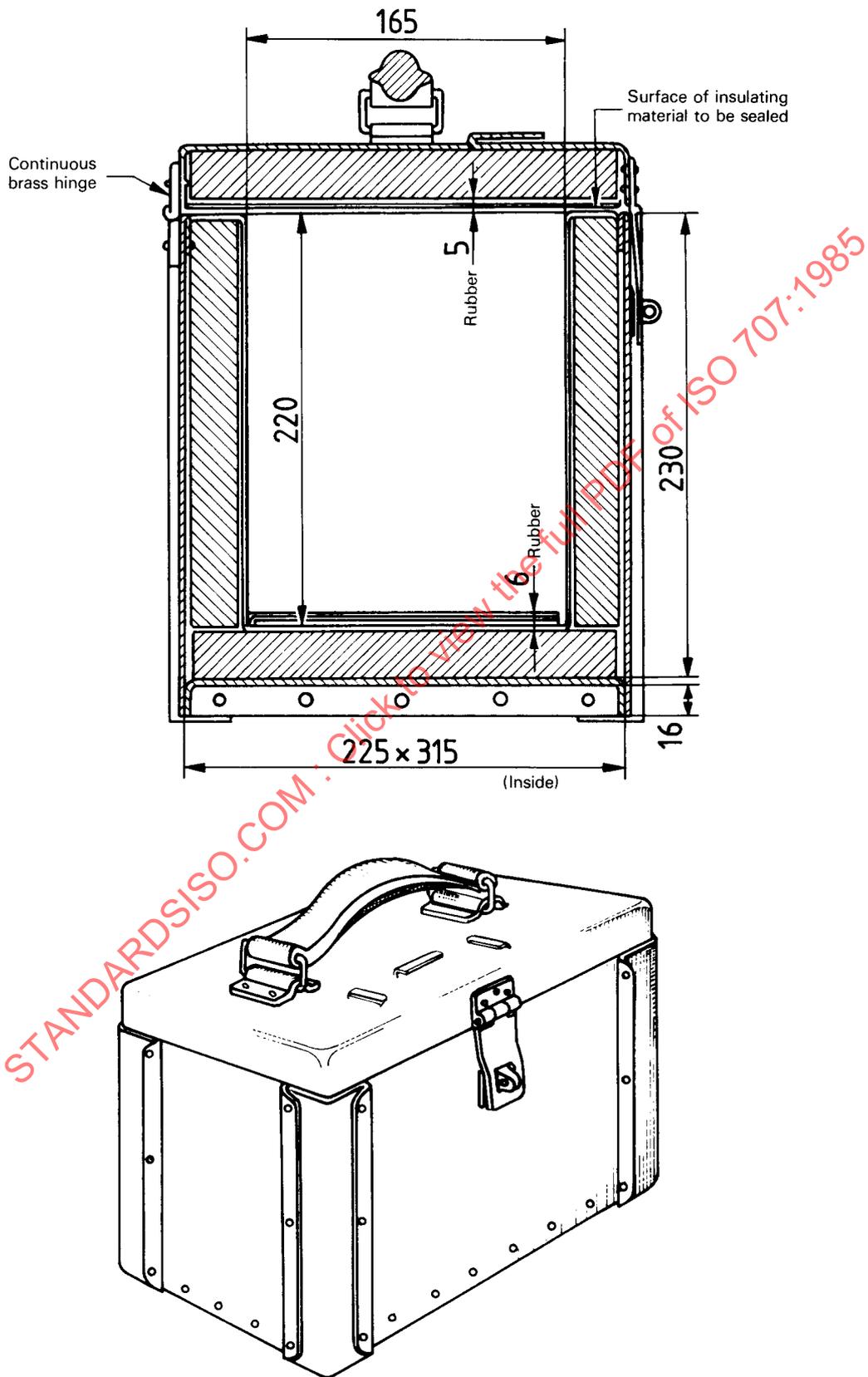


Figure 8 – Thermally insulated box suitable for the transport of samples of edible ices

13.2.2 Scoop, spoon or broad-bladed spatula.**13.2.3 Sample containers**

See 5.2.

The capacity of the sample containers shall be such that they are three-quarters filled by the sample and allow proper mixing of the contents by shaking before testing.

13.3 Procedure**13.3.1 General**

Care shall be taken to ensure that there is no uptake of atmospheric moisture by the contents of the product container during sampling for microbiological examination or in the period prior to sampling for chemical and physical analysis and for sensory examination. The product container shall be securely reclosed after sampling.

13.3.2 Sampling for microbiological examination

Take a sample of not less than 100 g.

Take samples for microbiological examination first and, whenever possible, from the same product containers as those taken for chemical and physical analysis and for sensory examination.

Sterilize the sampling equipment and containers as described in 5.1.1.

Remove the surface layer of the product by means of a sterilized spoon or spatula (13.2.2) from the sampling area, and take a sample of not less than 100 g by means of a sterilized borer (13.2.1) or scoop (13.2.2), if possible from near the centre of the container using the technique described in 13.3.3. Place the sample as quickly as possible into the sterilized sample container (13.2.3), which shall be closed immediately, taking aseptic precautions.

If there is the likelihood of dispute concerning the microbiological condition of the top layer of powder in the product container, first take a special sample from this layer.

13.3.3 Sampling for chemical and/or physical analysis and for sensory examination

Take a sample of not less than 200 g.

Pass the clean and dry borer through the product, if necessary with the container laid on its side, with the slit oriented downwards and an even rate of penetration being used. When the borer reaches the bottom of the container, rotate it through 180°, withdraw it and discharge the contents into the sample container. Use one or more borers to provide a sample of not less than 200 g. Alternatively, take a sample with a scoop (13.2.2). Immediately close the sample container once sampling is completed.

13.3.4 Sampling products packed in small retail containers

The contents of the intact and unopened containers constitute the sample. Take one or more containers to provide a sample of not less than 100 g for microbiological examination, and not less than 200 g for chemical and physical analysis and for sensory examination.

NOTE — Samples should always be taken in this way when it is desired to determine "instant" properties such as solubility, dispersibility, flowability and wettability.

13.4 Preservation, storage and dispatch of samples

See clauses 7 and 8.

14 Butter and related products**14.1 Applicability**

The instructions given in this clause are applicable to butter, whey butter, butter substitutes where the fat phase is a mixture of milkfat with other oils or fats, and low butterfat spreads.

14.2 Sampling equipment (see also 5.1)

14.2.1 Butter triers, of sufficient length to pass diagonally to the bottom of the product container. A suitable trier is shown in figure 10. Dimensions for guidance are given in table 3.

Table 3 — Dimensions of butter triers

Dimensions in millimetres

		Type A long	Type B medium	Type C short
a	Length of blade	540	225	125
b	Minimum thickness of metal in middle of blade	1,8	1,5	1,0
c	Minimum frontal breadth at 15 mm from end of blade	17	17	11

The grip, blade and stem should preferably be made of polished stainless steel.

The edges of the blade shall be sufficiently sharp to facilitate the sampling of hard butter.

14.2.2 Spatula, broad-bladed.**14.2.3 Knife**, of sufficient size.**14.2.4 Sample containers.****14.2.4.1** See 5.2.

Use opaque containers or keep the filled containers in the dark.

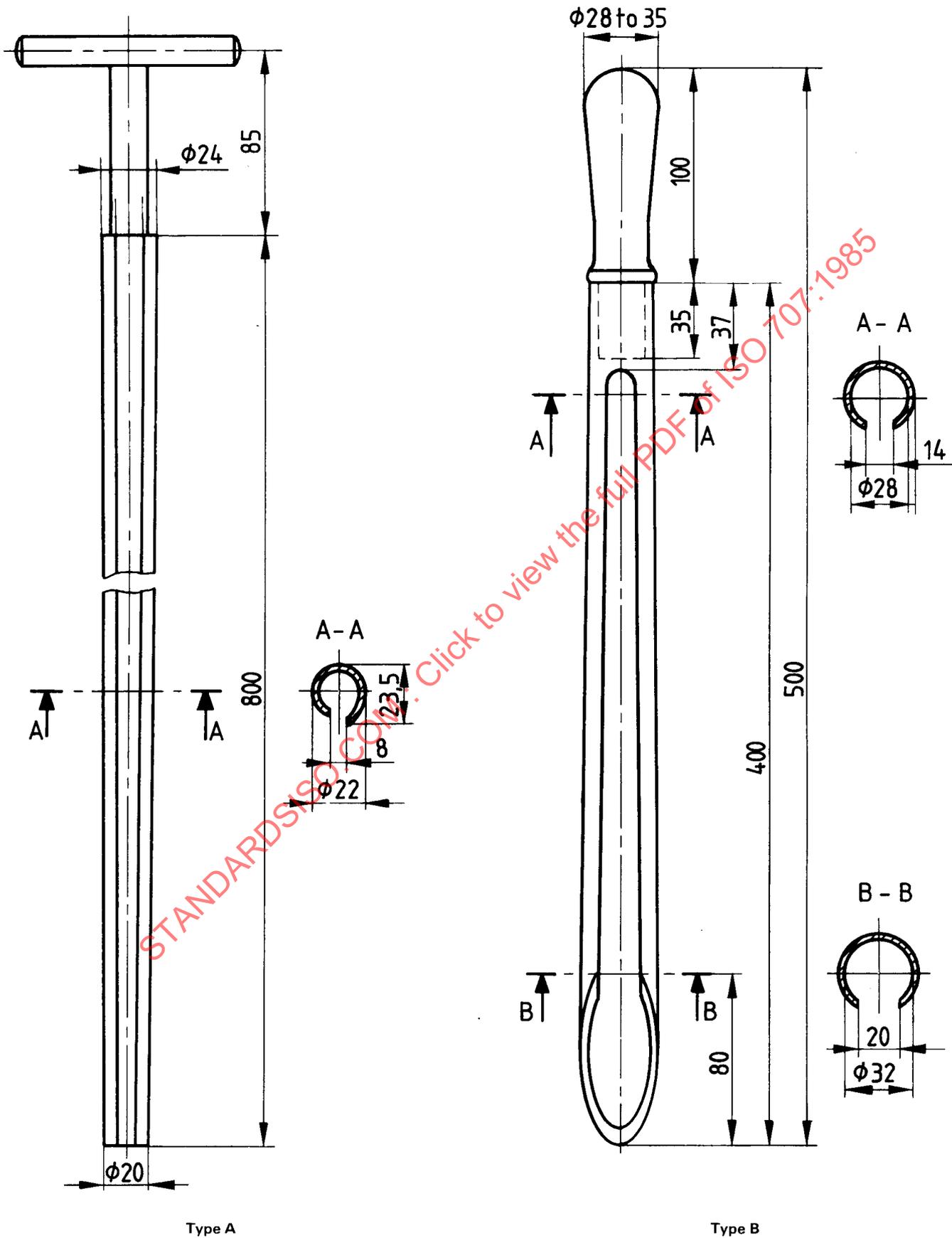


Figure 9 — Dried-milk borers

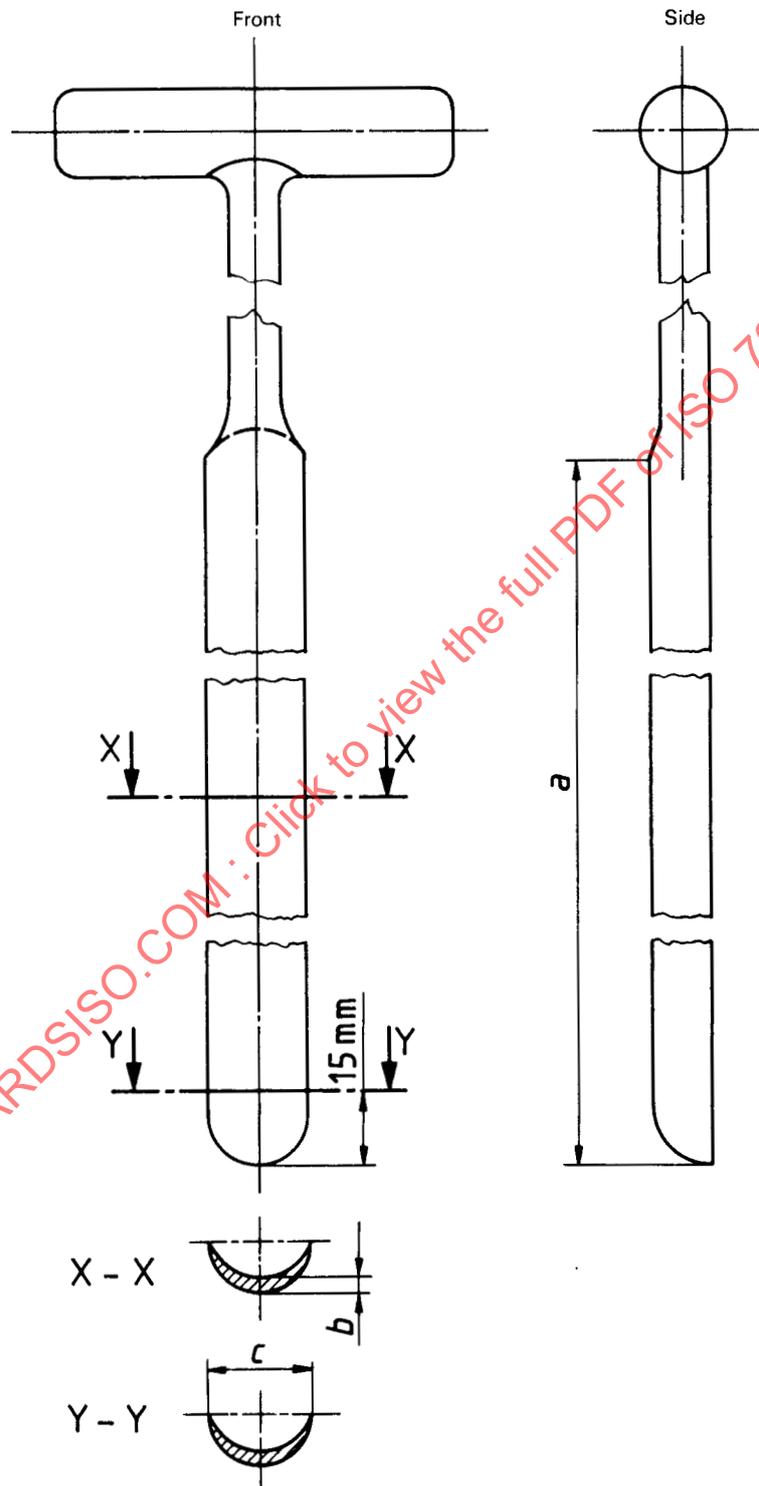


Figure 10 – Butter trier

14.2.4.2 Containers for samples for microbiological examination and for chemical and/or physical analysis

The capacity of the sample containers shall be such that they are more than half, but not more than three-quarters, filled by the sample. Alternatively, for chemical and/or physical analysis the sample may be wrapped in aluminium foil.

NOTE — In some cases, for example, if the sample is intended for the determination of peroxide value, it is essential that the containers be completely filled and have an airtight closure.

14.2.4.3 Containers for samples for sensory examination

Suitable containers include carton boxes, which can be adequately closed and which are provided on the inside with a sufficiently large piece of parchment paper. The capacity of the boxes shall be such that they are almost completely filled by the sample.

14.3 Procedure

14.3.1 Sampling for microbiological examination

Take a sample of not less than 100 g.

Take samples for microbiological examination first and, whenever possible, from the same product containers as those taken for chemical and physical analysis and for sensory examination.

Sterilize the sampling equipment and containers as described in 5.1.1.

Remove the surface layer of the product from the sampling area by means of a sterilized spatula (14.2.2) to a depth of not less than 5 mm. Proceed as described in 14.3.2 using aseptic techniques. Use a sterilized trier (14.2.1) each time for taking cores of the product.

14.3.2 Sampling for chemical and/or some physical analyses

14.3.2.1 Sampling from the product in bulk or from a container or a packet with a content of more than 1 kg

Take a sample of not less than 100 g.

If necessary, keep the container or packet, from which the sample is to be taken, for a sufficient time at a temperature of 8 to 10 °C, until the product has obtained the desired firmness.

Pass the best fitting butter trier from the edge diagonally through the product ensuring that the trier does not penetrate the bottom surface. Rotate the trier through one complete turn and withdraw a full core. Hold the point of the trier over the opened sample container and transfer the core of product to the container using a spatula. Leave about 25 mm of the upper part of the core using it to plug the hole in the product. Take one or more cores to obtain sufficient sample.

Do not include moisture condensed on the surface of the butter or adhering to the outside of the trier. Remove such moisture with absorbent paper.

Clean and dry the trier before each use.

14.3.2.2 Sampling from a container or a packet with a content of 1 kg or less

The contents of intact and unopened containers or packets constitute the sample. Take one or more containers or packets to provide a sample of not less than 100 g.

14.3.3 Sampling for sensory examination and/or for some physical analyses

14.3.3.1 Sampling from the product in bulk or from a container with a content of more than 2,5 kg

Take a sample of not less than 2 kg.

Carefully cut with a knife (14.2.3), or other suitable device, a block of the product which will fit into the sample box (14.2.4.3), wrap the block in parchment paper, and place it in the box. Avoid deformation of the product during cutting and wrapping.

14.3.3.2 Sampling from a container or a packet with a content of 2,5 kg or less

The contents of intact and unopened containers or packets constitute the sample. Take one or more containers or packets to provide a sample of not less than 200 g.

Avoid deformation of the product during sampling.

NOTE — Samples for sensory examination can also be used for the determination of spreadability and water dispersion.

14.4 Preservation, storage and dispatch of samples

See clauses 7 and 8.

15 Anhydrous milkfat and related products

15.1 Applicability

The instructions given in this clause are applicable to anhydrous milkfat, anhydrous butteroil or anhydrous butterfat, butteroil or butterfat and ghee, as mentioned in IDF Standard 68 A. They are also applicable to fractionated anhydrous milkfat or butterfat products.

15.2 Sampling equipment (see also 5.1)

15.2.1 Butter triers, of sufficient length to pass diagonally to the bottom of the product container.

The triers described in 14.2.1 (see figure 10) are suitable.

15.2.2 Spatula, broad-bladed.

15.2.3 Ladle, spoon or dipper, of capacity 25 to 100 ml, for sampling liquid products.

15.2.4 Sample containers

See 5.2.

Use opaque containers or keep the filled containers in the dark.

The capacity of the sample containers shall be such that they are almost completely filled by the sample and allow proper mixing of the contents before testing.

NOTE — In some cases, for example, if the sample is intended for the determination of peroxide value, it is essential that the containers be completely filled and have an airtight closure.

15.3 Procedure

15.3.1 Sampling for microbiological examination

Take a sample of not less than 100 g.

Take samples for microbiological examination first and, whenever possible, from the same product containers as those taken for chemical and physical analysis and for sensory examination.

Sterilize the sampling equipment and containers as described in 5.1.1.

Proceed as described in 15.3.2.1 for liquid products and as described in 15.3.2.2 for partially melted products, using, in each case, aseptic techniques.

For sampling solid products, remove the surface layer of the product, by means of a sterilized spatula (15.2.2) from the sampling area to a depth of not less than 5 mm and proceed as described in 15.3.2.3 using aseptic techniques. Use a sterilized trier (15.2.1) each time for taking cores of the product.

15.3.2 Sampling for chemical and/or physical analysis and for sensory examination

Take a sample of not less than 100 g.

15.3.2.1 Liquid products

Carefully mix the liquid with the ladle, spoon or dipper (15.2.3) avoiding inclusion of air and oxidation.

Take the sample immediately after mixing, by means of the equipment used for mixing.

15.3.2.2 Partially melted products

Do not take the sample until the product has completely melted (the temperature of the product shall never exceed 40 °C). Proceed as described in 15.3.2.1, or if the product has completely congealed, proceed as described in 15.3.2.3.

If possible, keep the product before sampling at a temperature which will promote melting or congealing.

15.3.2.3 Solid products

Pass the best fitting butter trier through the product ensuring that the trier does not penetrate the bottom surface. Rotate the trier through one complete turn and withdraw a full core. Hold the point of the trier over the opened sample container (15.2.4

and transfer the core of the product to the container using a spatula. Leave about 25 mm of the upper part of the core, using it to plug the hole in the product. Take one or more cores to provide a sample of not less than 100 g.

15.3.3 Sampling products packed in small retail containers

The contents of intact and unopened containers constitute the sample. Take one or more containers to provide a sample of not less than 100 g.

15.4 Preservation, storage and dispatch of samples

See clauses 7 and 8.

16 Cheese

16.1 Applicability

The instructions given in this clause are applicable to hard, semi-hard and soft cheese, prepacked cheese sold in brine, fresh cheese, processed cheese products and whey cheese.

16.2 Sampling equipment (see also 5.1)

16.2.1 Cheese triers, of shape and size appropriate to the cheese to be sampled. A suitable trier for some purposes is shown in figure 11. Dimensions for guidance are given in table 4.

Table 4 — Dimensions of cheese triers

Dimensions in millimetres

		Type A long	Type B medium	Type C short
a	Length of blade	540	150	125
b	Minimum thickness of metal in middle of blade	1,5	0,9	0,7
c	Minimum frontal breadth at 15 mm from end of blade	17	14	11

The grip, blade and stem should preferably be made of polished stainless steel.

The edges and the point of the blade shall be sufficiently sharp to facilitate the sampling of hard cheese.

16.2.2 Scalpel or knife, with a pointed blade and a smooth surface, preferably carried in a sheath.

16.2.3 Cutting wire, of sufficient size and strength.

16.2.4 Sealing compounds, for example a mixture of paraffin, wax and beeswax, prepared by heating.

16.2.5 Sample containers

See 5.2.

Aluminium foil is also suitable except for cores intended for microbiological examination.