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Milk and milk products — Guidance on sampling

Lait et produits laitiers — Lignes directrices pour l'échantillonnage



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 707|IDF 50 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

This third edition of ISO 707|IDF 50 cancels and replaces the second edition (ISO 707:1997), which has been technically revised.

Foreword

IDF (the International Dairy Federation) is a non-profit organization representing the dairy sector worldwide. IDF membership comprises National Committees in every member country as well as regional dairy associations having signed a formal agreement on cooperation with IDF. All members of IDF have the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO in the development of standard methods of analysis and sampling for milk and milk products.

Draft International Standards adopted by the Action Teams and Standing Committees are circulated to the National Committees for voting. Publication as an International Standard requires approval by at least 50 % of the IDF National Committees casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. IDF shall not be held responsible for identifying any or all such patent rights.

ISO 707|IDF 50 was prepared by the International Dairy Federation (IDF) and Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*. It is being published jointly by IDF and ISO.

All work was carried out by the Joint ISO-IDF Action Team on *Sampling and sample preparation* of the Standing Committee on *Quality assurance, statistics of analytical data and sampling* under the aegis of its project leader, Mr. T. Berger (CH).

This edition of ISO 707|IDF 50 cancels and replaces IDF 50:1995, which has been technically revised.

Introduction

Sampling is an operation that requires most careful attention; emphasis cannot be too strongly laid on the necessity of obtaining a properly representative sample. Written sampling procedures are demanded by ISO/IEC 17025^[6] if sampling is performed by laboratories. Written procedures are also required for subsampling steps in the laboratory, e.g. the preparation of test portions. The sampling procedure is part of the measurement procedure, but not of the measurement itself. It therefore does not contribute to the measurement uncertainty. Variations resulting from sampling procedures handled by the laboratory contribute to the uncertainty of the reported result and have therefore to be added to the measurement uncertainty. Reference [10] is a guidance document on this issue.

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. Whenever special requirements are given for sampling and/or arise from a specific analysis to be performed, these requirements should be followed.

Heterogeneity in cheese provides particular challenges for sampling. Sampling uncertainty is mainly influenced by the heterogeneity of the sample, the sample size and the sampling method.

There are significant consequences for both microbiological as well as for chemical analyses in cheese. Normally the cheese curd is moulded into a specific shape and dimensions and this can affect the development. During ripening of the moulded cheese curd under regular conditions or in environments in which the humidity, temperature, and possibly composition of the atmosphere are controlled, the outside of the cheese will develop into a semi-closed layer with a lower moisture content, the rind, often initiated by brining. Due to the influence of the salt gradient in the brine, of oxygen, of drying out and of other reactions, the rind successively becomes of a somewhat different composition than the interior of the cheese.

Rennet and microorganisms, added as selected cultures or naturally available, by enzymatic and microbiological activity, change the structure and composition of the inner zone of the cheese. Moreover, microorganisms are often not homogeneously distributed throughout the cheese.

Ripening is influenced by storage temperature, time, humidity, and salt gradients. During or after ripening, the cheese rind can be treated or can be naturally colonized with desired cultures of microorganisms. The resulting layer, in the latter case referred to as smear, will have further influence on the ripening of the border zone. To be able to make correct decisions on the sampled material, specific knowledge of cheese ripening is necessary. Depending on the desired conclusion, it has to be decided where a sample is to be taken and how many samples are necessary.

For these reasons, ISO 707|IDF 50 has been written in the form of guidance rather than as an “imperative” standard.

The test samples obtained by the methods described in this International Standard are “laboratory samples” as defined in ISO 78-2:1999^[1], 3.1. The “test portion” obtained by the methods described is also defined in ISO 78-2:1999^[1], 3.3.

Milk and milk products — Guidance on sampling

1 Scope

This International Standard gives guidance on methods of sampling milk and milk products for microbiological, chemical, physical and sensory analysis, except for (semi)automated sampling.

NOTE See also Reference [9].

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7002, and the following, apply.

3.1

laboratory sample

sample as prepared for sending to the laboratory and intended for inspection or testing

[ISO 78-2:1999^[1], 3.1]

3.2

test portion

quantity of material drawn from the laboratory sample on which the test or observation is actually carried out

[Adapted from ISO 78-2:1999^[1], 3.3]

NOTE It is possible that test portions of milk and milk products may require further processing, e.g. removal of parts that impair the test result, aseptic extraction of parts or grating.

4 General arrangements

This International Standard is not suitable as a basis for formulating legal obligations between contracting parties. In such cases, additional written requirements are necessary.

The number of units to be selected for sampling by inspection by attributes may be determined according to ISO 5538|IDF 113^[3]. Sampling for inspection by variables may be determined according to ISO 8197 (IDF 136A)^[5].

The following instructions are not necessarily applicable for routine sampling:

- a) the parties concerned or their representatives should be given the opportunity to be present when sampling is performed;
- b) whenever special requirements are given for the sampling and/or arise from a specific analysis to be performed, these requirements should be followed.

4.1 Sampling personnel¹⁾

An authorized person, properly trained in the appropriate technique, e.g. for microbiological purposes, and free from any infectious disease, shall perform sampling.

4.2 Sealing and labelling of samples

Samples should be sealed (if this is a legal requirement or if agreed between the parties concerned) and a label attached, reproducing integrally the identification of product, the nature of the product and, at least, the identification number, name and signature (or initials) of the authorized person (4.1) 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 and the condition of product and storage conditions at the moment of sampling.

4.3 Replicate samples

Samples should be taken in duplicate, or in greater numbers, if this is a legal requirement or if agreed between the parties concerned.

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

4.4 Sampling report

Samples should be accompanied by a report, signed or initialled by the authorized sampling personnel (4.1) and countersigned — as far as necessary or agreed by the parties concerned — by witnesses present.

The report should include at least the following information:

- a) the place, date and time of sampling (the time only being required when agreed between the parties concerned);
- b) the names and designations of the authorized sampling personnel and of any witnesses;
- c) the precise method of sampling, including sample preparation and homogenization techniques;
- 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) if necessary, the place to which the samples are to be sent;
- h) if possible, the name and address of the producer or trader or of the persons responsible for packing the product.

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

When appropriate, the report should also include any relevant conditions or circumstances (e.g. 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, e.g. difficulty in achieving homogeneity of the product.

Test portion size and handling vary according to the test(s) intended and are found under the appropriate headings in the individual International Standards specifying the tests.

Sampling also includes preparation of the laboratory sample. Therefore, the sampling report or a separate laboratory report should clearly state how the laboratory samples were prepared. Sampling reports are transmitted to the appropriate authority together with the test report. The example of a sampling report for cheese is given in Annex D (see also 16.3).

5 Apparatus

5.1 Sampling equipment

5.1.1 General

Sampling equipment should be made of stainless steel, or other suitable material of adequate strength, which does not bring about a change in the sample which could affect the results of subsequent examinations.

All surfaces should be smooth and free from crevices. All corners should be rounded except in the case of method D mentioned in 5.1.2. The equipment should be dry prior to use.

5.1.2 For microbiological examination

Sampling equipment for microbiological examination should be clean and sterilized prior to use. Disposable plastics equipment should be sterile.

If solder is used in the manufacture of the equipment, it should be capable of withstanding a temperature of 180 °C. If possible, sterilization should be performed by one of the three following methods:

- a) Method A: Exposure to hot air at 170 °C for at least 1 h or equivalent (see ISO 7218^[4]);
- b) Method B: Exposure to steam in an autoclave set at 121 °C ± 1 °C for at least 15 min (see ISO 7218^[4]);
- c) Method C: Exposure to a sufficient dose of γ -radiation.

After sterilization by one of methods A, B or C, the sampling equipment should be stored under conditions to ensure sterility until ready to sample.

If, in a particular situation, sterilization by methods A, B or C is impossible, one the following alternative methods might be used provided that the sampling equipment is used immediately after treatment. However, these methods should be regarded as secondary methods only.

- d) Method D: Exposure of all working surfaces of the sampling equipment to a suitable flame;
- e) Method E: Immersion in ethanol of at least 70 % volume fraction (see 5.5.1) followed by 5 min drying time;
- f) Method F: Ignition with ethanol of 96 % volume fraction (see 5.5.2).

After treatment by either method D or method F, the sampling equipment should be cooled under appropriate conditions to maintain sanitation before sampling.

5.1.3 For chemical and physical analysis and for sensory examination

Sampling equipment should be clean and dry and should not influence the properties, such as odour, flavour, consistency, and composition, of the product. In some cases, equipment treated as described in 5.1.2 is required to avoid microbial contamination of the product.

The marking of samples should not influence the properties or composition of the product. Odourless marking equipment should be used, e.g. odourless permanent ink or felt-tip pens.

5.2 Sample containers

Sample containers and closures should be of material and construction that 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. Appropriate materials include glass, some metallic materials (e.g. stainless steel) and some plastics (e.g. polypropylene).

The containers should preferably be opaque. If necessary, transparent filled containers should be stored in a dark place. Containers and closures should be dry, clean and either sterile or suitable for treatment by one of the methods described in 5.1.2. The use of glass containers for sampling inside production areas should be avoided.

The shape and capacity of the containers should be appropriate to the particular requirements for the product to be sampled. Single-service plastic containers as well as aluminium foil of adequate strength (sterile and non-sterile) and suitable plastic bags, with appropriate methods of closure, may also be used.

Sample containers other than plastic bags should be securely closed either by using a suitable stopper or a screw cap of metallic or plastics material. The latter should have, if necessary, a liquid-tight plastic liner which is insoluble, non-absorbent and greaseproof and which will not influence the composition, properties or the odour and flavour of the sample. If stoppers are used, they should be made from, or covered with, non-absorbent, odourless and flavourless material. Sample containers need to be airtight/sealed to prevent contamination and air ingress.

Containers for samples for microbiological examinations should not be closed with cork stoppers or caps with cork seals, even if provided with liners. Containers for solid, semi-solid or viscous products should be wide-mouthed.

Small retail containers are considered as sample containers; the sample should consist of the contents of one or more intact, unopened containers.

Requirements for insulated containers for transport of cooled, frozen or quick-frozen samples are given in Annex B.

5.3 Sample preparation equipment

The technical equipment for sample preparation should be described in the specific method of analysis.

5.4 Thermometer specifications

Thermometers used in the sampling procedure should be validated and of sufficient accuracy.

5.5 Ethanol

5.5.1 Ethanol, undenatured, with a volume fraction of 70 %.

5.5.2 Ethanol, undenatured, with a volume fraction of 96 %.

CAUTION — This solution is hygroscopic and may change its concentration over a long period of time. Use freshly prepared solutions.

6 Sampling

Sampling should be done in such a way as to get representative samples of the product.

If laboratory samples for microbiological, chemical and physical analyses and sensory examinations are taken separately, those for microbiological examinations should be taken first using aseptic techniques and sterilized equipment and containers (see 5.1.2).

Care should be taken to ensure that when taking samples for sensory examinations, the flavour of the samples is not adversely affected by the use of sampling equipment or sampling cocks, e.g. method E or F (5.1.2).

The precise method of sampling and the mass or volume of product to be taken varies according to the nature of the product and the purpose for which samples are required.

For details of the requirements, see Clauses 9 to 16. If products contain coarse particles, it may be necessary to increase the minimum sample size. The sample container should be closed immediately after sampling.

For small retail containers, the sample consists of one or more unopened containers.

If necessary, a further sample is taken for temperature control during transportation to the testing laboratory.

7 Preservation of samples

Preservatives should normally not be added to samples intended for microbiological or sensory examination but 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, and testing of texture and flavour should not be performed;
- c) the nature and quantity of preservative are stated in the sampling report and, preferably, indicated on the label;
- d) the safety instructions for the preservative used are followed.

In certain cases, the preservative will interfere with the analyte. In such cases, a suitable correction should be used.

8 Storage and transport of samples

Storage and dispatch of the samples should be such that the state of the sample at the time of sampling remains essentially unaltered until the time of starting the test procedure.

During transport, where necessary, precautions should be taken to prevent exposure to off-odours, direct sunlight and other adverse conditions. If cooling is necessary, the minimum requirements to be met are the temperature ranges which are either legally requested or prescribed by the manufacturer. The storage temperature after sampling should be attained as quickly as possible. The time and temperature should be considered in combination and not independently.

Storage temperatures are given in Table 1.

Samples should be dispatched immediately after sampling to the testing laboratory. The time for dispatch of the samples to the testing laboratory should be as short as possible, preferably within 24 h. If requested, samples should be dispatched as instructed by the testing laboratory.

After preparation of the test portion, analysis should be carried out immediately.

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

Sampling according to Clause	Product	Preservation permitted for samples intended for chemical and physical analysis	Storage temperature ^a also used before and during transport °C	Minimum recombined sample size ^b
9	Non-sterilized milk and liquid milk products	Yes	1 to 5	100 ml or 100 g
9	Sterilized milk, UHT milk and sterilized liquid milk products in original, unopened containers	No	Ambient, max. 30	100 ml or 100 g
9	Sterilized milk, UHT milk and sterilized liquid milk products after sampling from the production line or from one or more original pack(s)	Yes	1 to 5	100 ml or 100 g
10	Evaporated milk, sweetened condensed milk, milk concentrates, and sterilized concentrates	No	Ambient, max. 30	100 g
11	Semi-solid and solid milk products except butter and cheese	No	1 to 5	100 g
12	Edible ices and semi-finished ice products	No	≤ -18	100 g
13	Dried milk and dried milk products	No	Ambient, max. 30	100 g
14	Butter and butter products	No	1 to 5 (in the dark)	50 g
15	Butterfat (butter oil and similar products)	No	1 to 5 (in the dark)	50 g
16	Fresh cheese	No	1 to 5	100 g
16	Processed cheese	No	1 to 5	100 g
16	Other cheeses	No	1 to 5	100 g

^a These temperatures are meant as general guidelines (see ISO 7218^[4]). For specific analytical purposes, other temperatures can be more appropriate. It may be, under certain practical conditions, not always easy or even impossible to maintain the "ideal" or desirable temperatures specified here especially during transportation. It is therefore recommended to use suitable containers in all cases where it is necessary (see also Annex B) and to monitor and record temperatures in a suitable way.

^b In certain cases, it will be necessary to take a number of samples to produce a composite of corresponding minimum sample size. A larger sample size for laboratory samples may be necessary according to the tests required and the type of product. Smaller sample sizes are possible if no analytical and statistical arguments are against it. For the measurement of zonal differences, e.g. in cheese, it may even be necessary to take smaller sample sizes.

9 Milk and liquid milk products

9.1 Applicability

The instructions given in this clause are applicable to raw and heat-treated milk, whole, partly skimmed and skimmed milk, flavoured milk, cream, fermented milk, buttermilk, liquid whey and similar products.

9.2 Sampling equipment

Sampling equipment should comply with the requirements of Clause 5.

9.2.1 Apparatus for manual mixing

Apparatus for mixing liquids in bulk should have a surface sufficient to produce adequate disturbance of the products. In view of the different shapes and sizes of containers, no specific design of apparatus can be recommended for all purposes, but they should be designed in such a way as to avoid damage of the inner surface of the container during mixing.

9.2.1.1 Apparatus for manual mixing in small vessels

For mixing liquids in small vessels (e.g. in buckets and cans) a stirrer (plunger) of the design and dimensions shown in Figure A.1 is suitable. The length should be adjusted to the depth of the vessel.

9.2.1.2 Apparatus for manual mixing in large vessels

A stirrer (plunger) of the design and dimensions shown in Figure A.2 is suitable for use for larger vessels (e.g. road and farm tanks).

9.2.2 Apparatus for mechanical agitation

9.2.2.1 Built-in agitators

The product to be stirred in the tank or vessel determines the technical data and construction of built-in agitators. Various types of agitators are used but no attempt has been made to describe any of them, within this International Standard.

9.2.2.2 Removable agitators

Removable agitators are mostly provided with a propeller and are introduced into transport, road and rail tanks through the inspection port. Best stirring results are achieved at a depth corresponding to 0,7 of the filling height. It is recommended that the agitator be inclined by 5° to 20° as this provides a horizontal as well as vertical component to the resultant stirring motion of the liquor liquid.

9.2.3 Apparatus for taking samples

9.2.3.1 Apparatus for sampling

A dipper of the shape and size shown in Figure A.3 is suitable for sampling. The tapered form of the cup permits nesting of the dippers.

9.2.3.2 Sample containers

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

9.2.3.3 Thermally insulated transport container

See Annex B.

9.3 Sampling

Thoroughly mix all liquids, by inverting, stirring, by pouring to and from one product container to another of the same volume, until sufficient homogeneity is obtained but avoid foaming. The equipment described in 9.2.1 and 9.2.2 may be used.

Take the sample immediately after mixing. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

9.3.1 Sampling for microbiological examination

Samples for microbiological examination should always be taken first using aseptic techniques. Whenever possible, they should be taken from the same product containers as those for chemical and physical analysis and for sensory examination.

Treat the sampling equipment and sample containers as described in 5.1.2.

Proceed as described in 9.3.2 but using aseptic techniques.

9.3.2 Sampling for chemical and physical analysis and sensory examination

In certain cases, sampling equipment and sample containers should be treated as described in 5.1.2 for chemical and physical analysis and sensory examination.

9.3.2.1 Small vessels, milk buckets and cans

Thoroughly mix the milk, e.g. by transfer, stirring or plunging (plunger).

9.3.2.2 Milk tanks or vats

Mechanically stir the milk for at least 5 min until sufficient homogeneity is obtained. 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 min to 2 min). In those instances where the propeller of the agitator is close to the surface of the milk, the agitator should not be used, as that is likely to lead to the formation of foam.

9.3.2.3 Weighing bowl

To obtain a representative milk sample, it is essential that the milk be adequately mixed in the weighing bowl. A sufficient degree of mixing is not achieved when the milk being tipped into the weighing bowl varies and does not allow proper sampling. In that case, it is essential to supplement the mixing by additional agitation. The amount of additional agitation should be determined by experiment. When the volume of milk to be sampled exceeds the capacity of the weighing bowl, a sample representative of the whole consignment should be obtained.

9.3.2.4 Large vessels, storage, rail and road tanks

In each case, thoroughly mix the milk by an appropriate method before sampling, e.g. by mechanical agitation, by stirring with clean compressed air without foaming or by the use of a plunger. When compressed air is used, any adverse influence on the product to be mixed should be avoided.

The extent of mixing should be appropriate to the period of time over which the milk has been at rest.

An agitator propeller too close to the surface of the milk is likely to lead to the formation of foam. Do not use an agitator if foam production cannot be avoided.

Mixing by using a plunger or a removable agitator in road, rail tanks or vessels of similar size should be performed as follows.

- a) When samples are taken within 30 min after filling the container, the milk should be mixed for at least 5 min by plunging or stirring with an agitator. When the milk has been stored in the tank for a longer period of time, mixing should be extended to at least 15 min.
- b) When the tank is completely filled as is normally the case with transport, road and rail tanks, proper mixing of milk showing pronounced creaming phenomena can only be achieved by mechanical agitation.

In a large vessel with a bottom discharge outlet or a sampling cock installed at another place, there may be, at the discharge outlet, a small quantity of milk which is not representative of the whole content even after mixing. Accordingly, samples should preferably be taken through the inspection port. If samples are taken from the discharge outlet valve or the sampling cock, discharge sufficient milk to ensure that the samples are representative of the whole.

The efficiency of the method of mixing applied in any particular circumstances should 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 whole, or from the outlet of the tank at intervals during discharge.

9.3.2.5 Containers of different design

To take samples from shallow containers, special equipment is required.

9.3.2.6 Subdivided quantities

Unless a part of the bulk is tested individually, a representative quantity should be taken from each container after mixing the content and the quantity and container related to the sample noted in the sampling report (4.4). Mix portions of each representative quantity in amounts which are proportional to the quantity of the container from which they are taken. Samples from the aggregate sample thus obtained should be taken after mixing.

9.3.2.7 Sampling from closed systems

For taking samples from closed systems (e.g. UHT plants, aseptic techniques) and in particular for microbiological analysis, the working instructions for the installed sampling equipment should be observed.

9.3.2.8 Retail containers

The content of intact and unopened containers constitute the sample.

9.3.3 Applicability to products other than milk

9.3.3.1 Buttermilk, fermented milk, flavoured milk

Choose a suitable method from those described for milk and take a sample before fat or other solid matter has had time to separate. If the latter has occurred, proceed to ensure a representative sample from a homogeneous product as described in 9.3.1.

9.3.3.2 Cream

When using a plunger or a mechanical agitator for mixing cream thoroughly, mix the cream at the bottom of the container with the upper layers.

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 A.1 and A.2) may be used. When mechanical agitators are used, the incorporation of air should be avoided.

9.3.3.3 Whey

Choose a suitable method from those described for milk.

9.4 Preservation, storage and dispatch of samples

See Clauses 7 and 8.

10 Evaporated milk, sweetened condensed milk and milk concentrate

10.1 Applicability

The instructions given in this clause are applicable to evaporated milk, sweetened condensed milk and milk concentrates and similar products.

10.2 Sampling equipment

See 5.1.

10.2.1 Apparatus for manual mixing, see 9.2.1 and 9.2.2.

10.2.2 Stirrers, broad bladed, of sufficient length to reach the bottom of the product container and having one edge shaped to the contour of the container (see Figure A.4).

10.2.3 Dippers, see 9.2.3.1.

10.2.4 Rods, of length about 1 m and diameter about 35 mm.

10.2.5 Containers, for subsampling, of capacity 5 l, with a wide mouth and meeting the requirements of 5.2.

10.2.6 Spoons or spatula, broad bladed.

10.2.7 Sample containers, see 5.2.

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

10.3 Sampling evaporated milk

Take the sample immediately after mixing, avoid foaming. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

10.3.1 Sampling for microbiological examination

Samples for microbiological examination should always be taken first by using aseptic techniques. Whenever possible, they should be taken from the same product containers as those for chemical and physical analysis and for sensory examination.

Treat the sampling equipment and sample containers as described in 5.1.2.

Proceed as described in 10.3.2, but using aseptic techniques.

10.3.2 Sampling for chemical and physical analysis and for sensory examination

In certain cases, the sampling equipment and sample containers should also be treated as described in 5.1.2 for chemical and physical analysis and sensory examination.

10.3.2.1 Large vessels (e.g. 2 kg and 4 kg)

Thoroughly mix the evaporated milk by plunging or stirring using a manual stirrer, by mechanical agitation, by pouring from one container to another, until sufficient homogeneity is obtained. Care should be taken to minimize foam formation; excess foaming can lead to changes in the physical and sensory characteristics of the product being sampled.

However, in most cases, sufficient distribution of fat is only obtained if the containers have been left standing in water at a temperature not greater than 45 °C for a maximum of 30 min before mixing as described above.

If it proves difficult to obtain sufficient homogeneity, take subsamples from different locations of the product container, collected with the same sampling apparatus to obtain a representative laboratory sample.

Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

Note on the label and in the sampling report (4.4) if the sample is a mixture of subsamples.

10.3.2.2 Very large vessels (containers) of 500 kg and more, and road tanks

Mixing is, in principle, performed in the same manner as described for milk (9.3.2.4). Intensity of mixing is dependent on the degree of concentration.

10.3.2.3 Retail containers

The content of an unopened container of size greater than the minimum sample size constitutes the laboratory sample.

Where the size of a single unopened retail container does not meet the minimum sample size, a composite from multiple unopened retail containers constitutes the laboratory sample. The composite should be created as described in 10.3.2.1.

Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

If a sample is taken from retail containers, it should be preheated beforehand as described in 10.3.2.1.

10.4 Sampling sweetened condensed milk and milk concentrate

The sample should be taken immediately after mixing, avoid foaming. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

10.4.1 General

The sampling of bulk containers 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, 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 (see 10.2.4) 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.

Since sweetened condensed milk is frequently stored at ambient temperature, it is recommended that the content of bulk containers is brought to a feasible temperature. Crystallized concentrate in storage tanks cannot be sampled representatively unless the tank is designed for and equipped with a power driven agitator.

When the product is not homogeneous and particularly when crystals are not evenly distributed, note this fact in the sampling report (4.4). Perform sampling immediately after mixing.

10.4.2 Sampling for microbiological examination

Samples for microbiological examination should always be taken first using aseptic techniques. Whenever possible, they should be taken from the same product containers as those for chemical and physical analysis and for sensory examination.

Treat the sampling equipment and sample containers as described in 5.1.2.

Proceed as described in 10.4.2.1, but using aseptic techniques.

10.4.2.1 Bulk containers

Thoroughly clean, treat as described in 5.1.2 and rinse with cold sterile water, the outside end of the product container, or of the drum, if it is an end-opening type (bunghole), before opening the container or removing the end-cover (bung). For sterilization the surface can, repeatedly if required, be flamed using alcohol (see 5.1.2).

Proceed as described in 10.4.3, but using aseptic techniques.

In the case of condensed milk, which flows readily and is of uniform consistency, drums with a bung hole are turned. While the product is allowed to drain, the sample should be taken. Bung holes with screw caps are difficult to disinfect, so particular care should be taken. When the product has become viscous, the surface layer has to be removed to a depth of 20 mm to 30 mm by using a spoon treated as described in 5.1.2 and then the sample should be taken.

When surface samples are taken sampling should be performed according to special instructions corresponding to the particular purpose.

Note the type of bulk container in the sampling report (4.4).

10.4.3 Sampling for chemical and physical analysis and for sensory examination

In certain cases, sampling equipment and sample containers should also be treated as described in 5.1.2 for chemical and physical analysis and sensory examination.

10.4.3.1 Open-ended containers (drums with cover).

Thoroughly clean and dry one end of the container before opening to prevent foreign matter falling into the container during the opening process. The content should be mixed by using a stirrer (see Figure A.4). The blade round the sides and the bottom of the container should be scraped to remove any adhering product.

Thoroughly mix the content using a combination of rotary and vertical movement with the stirrer inclined diagonally while taking care to avoid incorporation of air into the sample. The stirrer should be withdrawn and the condensed milk adhering to it be transferred into a 5 l container (10.2.5) by using a spatula or spoon. The mixing and withdrawal should be repeated until 2 l to 3 l have been collected. The volume should be mixed until homogeneous before taking the sample.

10.4.3.2 Enclosed containers, (drums) with outlet (bung) at one end or at the side.

For reasons described in 10.4.1, sampling through the outlet (bung hole) is suitable only with condensed milk which flows readily and is of uniform consistency. Inserting a rod through the bung hole should mix the content while agitating and stirring as far as possible in all directions.

Withdraw the rod and proceed as described in 10.4.3.1 (sampling with a stirrer).

10.4.3.3 Bulk container, of capacity 500 l with inspection port.

The procedure is, in principle, the same as for milk (see 9.3.2.5).

10.4.3.4 Retail containers.

The content of the intact, unopened containers constitute the test sample. One or more containers should be taken to obtain a total sample. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

10.5 Preservation, storage and dispatch of samples

See Clauses 7 and 8.

11 Semi-solid and solid milk products except butter and cheese

11.1 Applicability

The instructions given in this clause are applicable to puddings, desserts and milk products of the fermented or not fermented, semi-solid, solid or foamed type, with or without addition of stabilizers, binding agents, fruits, nuts or other ingredients as well as other products, the semi-solid or solid texture being the common property.

11.2 Sampling equipment

See 5.1.

11.2.1 Apparatus for mixing

See 9.2.1.

11.2.2 Apparatus for sampling

See 9.2.3.1.

11.2.3 Sample containers

See 5.2. The capacity of the sample containers should be such that they are almost completely filled by the sample and allow proper mixing of the content before testing.

11.3 Sampling

The sampling of semi-solid and solid milk products from large containers may be a matter of extreme difficulty, particularly when the product is highly viscous or if it contains particular non-homogenously distributed constituents. Mixing should, therefore, be adjusted to the particular requirements of the product.

If possible, preference should be given to retail container lots. In special cases, the instructions given in 11.3.2.1 and 11.3.2.2 should be adjusted to the specific properties of the product.

Take the sample immediately after mixing, while avoiding foaming. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

11.3.1 Sampling for microbiological examination

Samples for microbiological examination should always be taken first using aseptic techniques. Whenever possible, they should be taken from the same product containers as those for chemical and physical analysis and for sensory examination.

Treat the sampling equipment and sample containers as described in 5.1.2.

Proceed as described in 11.3.2, but using aseptic techniques.

11.3.2 Sampling for chemical and physical analysis and for sensory examination

In certain cases sampling equipment and sample containers should also be treated as described in 5.1.2 for chemical and physical analysis and for sensory examination.

The required product type and subsequent examination are the decisive factors for the sampling technique to be employed.

11.3.2.1 Containers or tanks

The product should be mixed by plunging or stirring by mechanical agitation until sufficient homogeneity is ensured. Mixing should be done gently as to avoid foaming, whipping, whey-separation, and disruption of lumpy ingredients (see also 9.2.1).

If it proves difficult to obtain sufficient homogeneity, samples should be taken from different portions of the product container to obtain a representative total sample. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

Note on the label and in the sampling report if the sample is a mixture of subsamples (see 4.4).

11.3.2.2 Retail containers

The content of intact, unopened containers constitutes the sample. One or more containers should be taken to obtain a total sample. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

Large containers from which portions are taken for selling or consumption should be taken as a whole.

11.4 Preservation, storage and dispatch of samples

See Clauses 7 and 8. During transit precautions should be taken to prevent exposure to vibration because of demixing and thixotropic effects that could negatively influence homogeneity or water deposition.

12 Edible ices, semi-processed (semi-finished) ices and other frozen milk products

12.1 Applicability

The instructions given in this clause are applicable to edible ices, semi-processed ices and other frozen products.

12.2 Sampling equipment

See 5.1.

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

12.2.2 Spoon, knife or spatula, or ice scoop.

12.2.3 Sample containers, see 5.2.

The sample containers should be placed in an appropriate thermally insulated transport container (see 9.2.3.3) which has been suitably refrigerated (e.g. with solid carbon dioxide) for not less than 30 min before use.

12.3 Sampling

Sampling from containers from which portions are to be taken can best be performed at product temperatures between -12°C and -18°C .

If the consistency of the product is too firm for sampling, the whole container constitutes the sample.

Refer to Table 1 for minimum sample size.

12.3.1 Sampling for microbiological examination

Samples for microbiological examination should always be taken first by using aseptic techniques. Whenever possible, they should be taken from the same product containers as those for chemical and physical analysis and for sensory examination.

Treat the sampling equipment and sample containers as described in 5.1.2.

The treated spoon, knife or spatula (12.2.2) should be used to remove the surface layer of the product in the centre of the container from the sampling area to a depth of at least 10 mm. A sample of adequate size should be taken with a treated instrument from the removed area. If required, a “surface sample” should be obtained by uniformly scraping the product surface to be tested by using a treated spoon or spatula to a minimum depth.

When the microbiological condition of the product as presented to the customer has to be examined, the retailer's vending operations normally used for dispensing should be applied for the purpose of sampling.

The sample should be transferred as quickly as possible into the treated sample container which should be closed immediately. The container should immediately be placed in precooled transport containers (12.2.3).

Proceed as described in 12.3.2, but using aseptic techniques.

12.3.2 Sampling for chemical and physical analysis and for sensory examination

In certain cases, sampling equipment and sample containers should also be treated as described in 5.1.2 for chemical and physical analysis and for sensory examination.

After sampling, the sample should immediately be transferred into the precooled transport container.

Only original packages should be taken for physical analysis.

12.3.2.1 Retail containers

Retail containers include small packages, ice lollipops, multilayered ices, and marbled ices.

The samples should be collected and dispatched in their original containers while keeping the samples deep-frozen until analysed.

12.3.2.2 Soft ice

Soft ice is ice directly sold from the freezer. When the condition of the product as presented to the retail customer has to be examined, the retailer's vending operations normally used for dispensing should be applied for the purpose of sampling.

When information is required regarding the condition of the product in the freezer, the sample should be taken directly from the freezer. To this end, the outlet should be thoroughly cleaned and disinfected first as described in 5.1.2.

A sufficient amount of the product should be let out. The requisite number of sample containers should be filled in succession from the freezer while it is continuing to operate.

12.3.2.3 Semi-processed ices

Sampling of semi-processed ices (e.g. as concentrates and powders for the production of edible ices) is performed as described in Clauses 9 and 13.

12.4 Preservation, storage and dispatch of samples

See Clauses 7 and 8. The storage and transport temperature may vary according to the purposes of the product and analysis envisaged. The temperature should be $-18\text{ }^{\circ}\text{C}$ or, in certain cases, even lower.

13 Dried milk and dried milk products

13.1 Applicability

The sampling instructions given in this clause are applicable to such products as milk powder with different fat contents, dried whey, milk protein products and their derived products, co-precipitates and other powders with high milk protein contents. The instructions are also applicable to lactose in powder form.

The sampling instructions in Clause 13 are not suitable for powders in large bulk containers (silo). From such containers, a number of small samples should be taken during its loading or unloading to allow access to the entire consignment (batch).

Special attention should be paid to exclude the influence of atmospheric moisture.

13.2 Sampling equipment

See 5.1.

13.2.1 Borers, of sufficient length, to reach any desired point of the product container.

The borer should be made entirely of polished stainless steel. Borers suitable for sampling containers up to 30 kg are shown in Figure A.5. Dimensions for guidance are given in Table A.1.

The protruding borer edge and point of type A should be sufficiently sharp to serve as a scraper and to facilitate sampling.

13.2.2 Scoop, spoon or broad-bladed **spatula**.

13.2.3 Sample containers (see 5.2).

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

13.3 Sampling

Precautions should be taken to ensure 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 should be securely closed again after sampling.

Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

13.3.1 Sampling for microbiological examination

Samples for microbiological examination should always be taken first by using aseptic techniques. Whenever possible, they should be taken from the same product containers as those for chemical and physical analysis and for sensory examination.

The sampling equipment and sample containers should be sterilized as described in 5.1.2 using method A, B or C. Pre-sterilized (disposable) equipment may also be used.

The sterilized spoon or spatula (13.2.2) should be used to remove the surface layer of the product from the sampling area. The sample should be taken with a sterilized borer, if possible, from near the centre of the container by using the technique described in 13.3.2. The sample should be transferred as quickly as possible into a sterilized sample container, which should be closed immediately, taking aseptic precautions.

If there is the likelihood of dispute concerning the microbiological condition of the top layer of powders in the product container, a special sample from this layer should be taken first.

13.3.2 Sampling for chemical and physical analysis and for sensory examination

In certain cases, the sampling equipment and sample containers should also be treated as described in 5.1.2 for chemical and physical analysis and for sensory examination.

The clean dry borer (13.2.1) should be passed into or through the product, if necessary, with the container laid on its side, the slit oriented downwards and an even rate of penetration being used.

When the borer reaches the desired point in the container, it should be rotated through 180° and withdrawn. Its contents should be discharged into the sample container.

According to the purpose of testing envisaged, a sample may also be taken with a scoop.

Close the sample container immediately once sampling is completed.

13.3.3 Retail containers

The content of the intact and unopened containers constitute the sample. One or more containers should be taken to obtain a sample. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

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, butter with additives, half-cream milk and half-fat butter, and similar products.

14.2 Sampling equipment

See 5.1.

14.2.1 Butter triers, of sufficient length, to pass diagonally to the bottom of the product container and of a dimension suited for the purpose envisaged (see Figure A.7). When used, the butter trier temperature should be equal to the butter to be sampled.

14.2.2 Spatula, broad bladed.

14.2.3 Knife, of sufficient size.

14.2.4 Sample containers (see 5.2).

The capacity of the sample containers should be such that it is adequately proportioned to the size of the sample.

The use of opaque sample containers is recommended. If required for the tests to be performed, wrap the container or core in aluminium foil (e.g. to preserve from photo-oxidation).

Carton boxes should be used for samples of 2 kg.

In some cases it is essential that the sample containers be completely filled or provided with inert gas and have an airtight closure, e.g. when fat indices are to be determined.

14.2.5 Sample containers, for sensory examination (see 5.2).

Suitable containers include carton boxes, which can be adequately closed and be provided with a sufficiently large piece of aluminium foil or plastic coated parchment paper on the inside.

The capacity of the boxes should be such that they are almost completely filled by the sample.

14.3 Sampling

Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

14.3.1 Sampling for microbiological examination

Samples for microbiological examination should always be taken first using aseptic techniques. Whenever possible, they should be taken from the same product containers as those for chemical and physical analysis and for sensory examination.

Treat the sampling equipment and sample containers as described in 5.1.2.

The spatula (14.2.2) should be used to remove the surface layer of the product from the sampling area to a depth of not less than 5 mm. Proceed as described in 14.3.2 but using aseptic techniques. A treated trier should each time be used for taking a core of the product.

For microbiological examination of the surface, sampling should be performed according to special instructions depending on the purpose envisaged.

14.3.2 Sampling for chemical, physical analysis and for sensory examination

A sample of sufficient size should be taken for the number of sensory examinations and physical analyses.

In certain cases sampling equipment and sample containers should be treated as described in 5.1.2 for chemical, physical analysis and for sensory examination.

14.3.2.1 Retail containers, with content of 1 kg or less.

The content of the intact and unopened container constitute the sample. One or more containers should be taken to obtain a sample. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

14.3.2.2 Products in bulk or packets, with content of more than 1 kg.

A butter trier of suitable size (14.2.1) should be passed from the edge diagonally through the product ensuring that the trier does not penetrate the bottom surface. The trier should be rotated through a half turn and be withdrawn with the core.

The upper 25 mm of the core should be discarded to avoid taking a non-representative portion of the butter due to moisture loss at the surface.

The rest of the core should be removed from the trier by using a spatula and be transferred either directly or after wrapping in aluminium foil to the container. The temperature of the butter, the sampling room and the used butter trier should be about the same.

Sampling of butter stored under deep-freezing conditions requires special care and experience (see 14.3.2.4).

14.3.2.3 Large containers, for sample sizes of more than 2 kg.

For sampling from a large container or sample sizes of more than 2 kg, a knife (14.2.3) should be used to cut a block of the product that fits into the sample box. The block should be wrapped in aluminium foil and be placed in the box. Any deformation of the product should be avoided during cutting and wrapping.

14.3.2.4 Frozen butter.

If the block of butter to be sampled is frozen (i.e. below 0 °C), the temperature of the butter should be raised to enable samples to be taken by using a butter trier (14.2.1).

CAUTION — Removing pieces of butter from corners or the sides of the frozen block results in unrepresentative sampling, because of moisture loss from the sides of the blocks during storage.

The temperature of the butter can be raised by storing the butter in a temperature-controlled conditioning room for a period of time. The minimum conditioning time required will depend on the temperature and size of the block as well as the temperature to which the block is to be conditioned (typically 0 °C to 5 °C).

When conditioning, the block of butter should be removed from its outer cardboard packaging to facilitate heat transfer. However, the butter should remain covered by the plastic inner wrap to prevent surface moisture changes by evaporation or condensation during conditioning. The conditioning room should have a suitable airflow and the ambient temperature should typically be in the range 5 °C to 10 °C. A purpose-built microwave unit may be used as an alternative to a temperature-controlled conditioning room.

If no conditioning room is available samples may also be conditioned at room temperature.

NOTE 1 Standard industrial microwaves are not suitable, as they are apt to create “hot spots” in the block and hence local melting of the butter.

NOTE 2 Assuming a temperature of 5 °C to 10 °C in the conditioning room, the time required for a 25 kg block frozen at –18 °C to condition to 0 °C to 5 °C is typically in the range 24 h to 48 h.

14.4 Preservation, storage and dispatch of samples

See Clauses 7 and 8.

15 Butterfat (butter oil) and related products

15.1 Applicability

The instructions given in this clause are applicable to anhydrous milk fat, butterfat, butter oil and similar products.

15.2 Sampling equipment

See 5.1.

15.2.1 Butter triers, of sufficient length to pass diagonally to the bottom of the product container and of a dimension suited for the purpose envisaged (see Figure A.7).

15.2.2 Spatula, broad bladed.

15.2.3 Apparatus for manual mixing (plunger), described in 9.2.1.

15.2.4 Dipper, of capacity 25 ml to 100 ml.

15.2.5 Sample containers (see 5.2).

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

In some cases, it is essential that the sample containers be completely filled or provided with inert gas and have an airtight closure, e.g. when fat indices are to be determined.

15.3 Sampling

Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

15.3.1 Sampling for microbiological examination

Samples for microbiological examination should always be taken first by using aseptic techniques. Whenever possible, they should be taken from the same product containers as those for chemical and physical analysis and for sensory examination.

Treat the sampling equipment and sample containers as described in 5.1.2.

The spatula (15.2.2) should be used to remove the surface layer of the product from the sampling area to a depth of not less than 5 mm. Proceed as described in 15.3.2 but using aseptic techniques.

15.3.2 Sampling for chemical and physical analysis and for sensory examination

In certain cases, sampling equipment and sample containers should also be treated as described in 5.1.2 for chemical, physical analysis and for sensory examination.

15.3.2.1 Retail containers, with content of 1 kg or less.

The content of intact and unopened containers constitute the sample. One or more containers should be taken to obtain a sample. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

15.3.2.2 Product in bulk.

15.3.2.2.1 Liquid products.

The product should be thoroughly mixed by plunging or by mechanical agitation until sufficient homogeneity is obtained.

15.3.2.2.2 Solid products.

Take a sample as described in 14.3.

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 cheese (e.g. hard, extra hard cheese, semi-hard, semi-soft, soft cheese, fresh cheese, acid curd cheese, cheese in brine, prepacked cheese, processed cheese, processed cheese preparations, flavoured processed cheese and cheese products).

16.2 Sampling equipment and chemicals

See 5.1.

16.2.1 Cheese triers, of shape and size appropriate to the cheese to be sampled (see Figure A.6).

16.2.2 Knife, with a pointed blade and a smooth surface.

16.2.3 Spatula.

16.2.4 Cutting wire, of sufficient size and strength.

16.2.5 Sample containers (see 5.2).

16.3 Sampling

Sampling is performed, depending upon the shape, mass and type, by taking an entire cheese, packed or prepacked portions or a sector, slices or cores as shown in Figures A.8 to A.25.

The heterogeneity of the product should be taken into account when collecting samples. Refer to Table 1 for minimum sample size and acceptable sampling temperatures.

Immediately after sampling, the samples (cores, slices, sectors, entire small cheese etc.) should be placed in a sample container of suitable size and shape. The sample may be cut into pieces for insertion into the container, but it should not be compressed or ground.

Storage of cheese samples tightly wrapped in aluminium foil, waxed paper (cheese paper) or closable plastic bags inside or even outside a sample container is an additional measurement to prevent moulding of the cheese surface.

Unless otherwise specified and whatever the method of sampling used, the sample should include any surface layer of the cheese (such as mould and rind).

If it is necessary to examine the surface layer (e.g. examine surface flora), special sampling instructions should be observed according to the purpose envisaged.

Often samples are collected in the same way as being consumed. Consumer habits are different from region to region. Therefore, the sample has to be described precisely in the sampling report (see 4.4).

An example of a sampling report for cheese is given in Annex D.

16.3.1 Sampling for microbiological examination

Samples for microbiological examination should always be taken first using aseptic techniques. Whenever possible, they should be taken from the same cheese or product (core) as those for chemical and physical analysis and for sensory examination.

The amount of sample taken for surface samples may be smaller than the minimum sample size referred to in Table 1.

The sampling equipment and containers should be treated as described in 5.1.2.

Proceed as described in 16.3.2 by using, however, aseptic techniques.

16.3.2 Sampling for chemical and physical analysis and sensory examination

16.3.2.1 Sampling of cheese other than fresh cheese and cheese sold in brine, oil etc.

16.3.2.1.1 Sampling by taking an entire cheese or cheese in prepacks

This method is normally used for small cheeses, small portions of cheese or prepacked cheeses.

A sufficient number of packets or portions should be taken to obtain the sample. Refer to Table 1 for minimum sample size and acceptable sampling temperatures. The sample should be placed in the original packaging in the sample container (plastic bags etc.).

16.3.2.1.2 Sampling by cutting sectors or slices

Any outer wrapping should be removed from the cheese. Inner wrapping, e.g. wax or plastic film, should not be removed.

The sample should be cut by using a knife (16.2.2) of sufficient size or a cutting wire (16.2.4). The sectors or slices should be of sufficient thickness.

16.3.2.1.3 Sampling by taking cores

Any outer wrapping should be removed from the cheese. Inner wrapping, e.g. wax or plastic film, should not be removed.

The cores should be wrapped in aluminium foil, special waxed paper (cheese paper) or closable plastic bags before placing them in the sample container if analysis is not performed immediately after sampling.

16.3.2.1.3.1 Sample including surface layer

A cheese trier (16.2.1) of sufficient length should be inserted into the cheese. The trier should be rotated through one complete turn and be withdrawn with the core.

By using the knife (16.2.2), the entire core should be transferred to a sample container (16.2.5). This procedure should be repeated until the sample is obtained. The core hole should be sealed with a suitable sealing compound.

16.3.2.1.3.2 Sample excluding surface layer

A cheese trier (16.2.1) of a larger diameter than the sampling trier should be inserted to a depth of approximately 25 mm into the cheese. The trier should be rotated through one complete turn. This short core should not be withdrawn, be kept separately and used later for closing the core hole.

A smaller sampling trier of sufficient length should be inserted through the inner surface of the cheese exposed by the above core hole. The trier should be rotated through one complete turn and be withdrawn with the core. With the aid of a knife (16.2.2) transfer the entire core to a sample container (16.2.5).

Repeat this procedure until the sample is obtained. Refer to Table 1 for minimum sample size and acceptable sampling temperatures. Close the core hole by reinserting the first outer core.

16.3.2.2 Sampling of fresh cheese

For sampling of fresh cheese the containers should be intact and unopened. The containers should not be opened until immediately before analysis.

A sufficient number of sample containers should be taken to obtain the sample. Refer to Table 1 for minimum sample size and acceptable sampling temperatures. Containers from which portions are taken should be taken as a whole.

16.3.2.3 Sampling of cheese sold in brine, oil etc.

This kind of cheese should be sampled by taking fragments of minimum sample size each (without brine, oil etc.) as referred to Table 1.

During storage in brine in particular, the composition of the cheese will change, depending on time and temperature. The testing laboratory should specify whether the sample should include brine, oil etc. or not. Normally brine, oil etc. are included. Whenever possible, the original ratio of cheese and liquid should be maintained and the latter should completely cover the cheese.

If brine is included, a sufficient amount of brine should be taken so as to cover the cheese completely. If brine is not included, the cheese or cheese fragments should be dried with filter paper and be placed in the sample container.

The testing laboratory can prescribe the temperature at which the sample should be stored or dispatched.

NOTE Indicate in the sampling report whether the sample has been taken with or without brine, oil etc.

16.4 Preservation, storage and dispatch of samples

See Clauses 7 and 8.

Annex A (informative)

Examples of sampling equipment and shapes of sampling

A.1 Examples of sampling equipment

A.1.1 Stirrers (plungers)

See Figures A.1 and A.2.

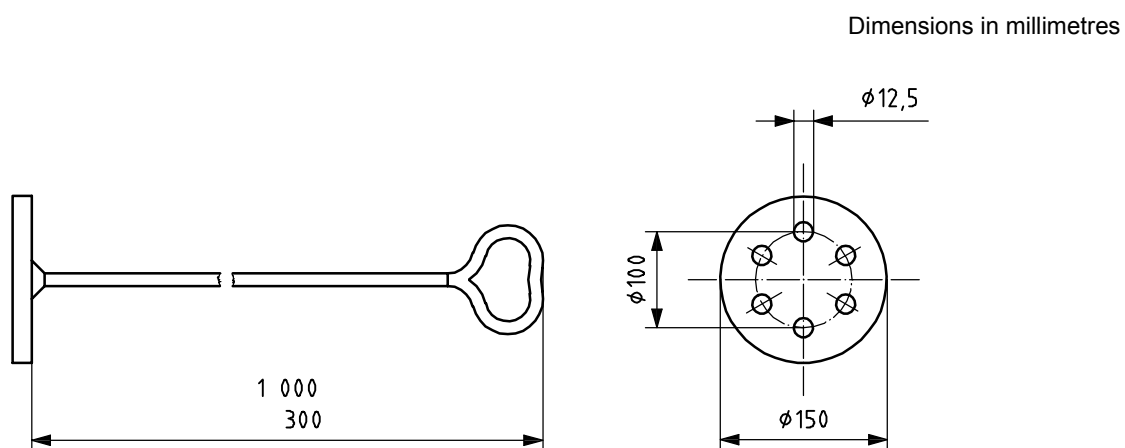


Figure A.1 — Recommended stirrer (plunger) for cans and buckets

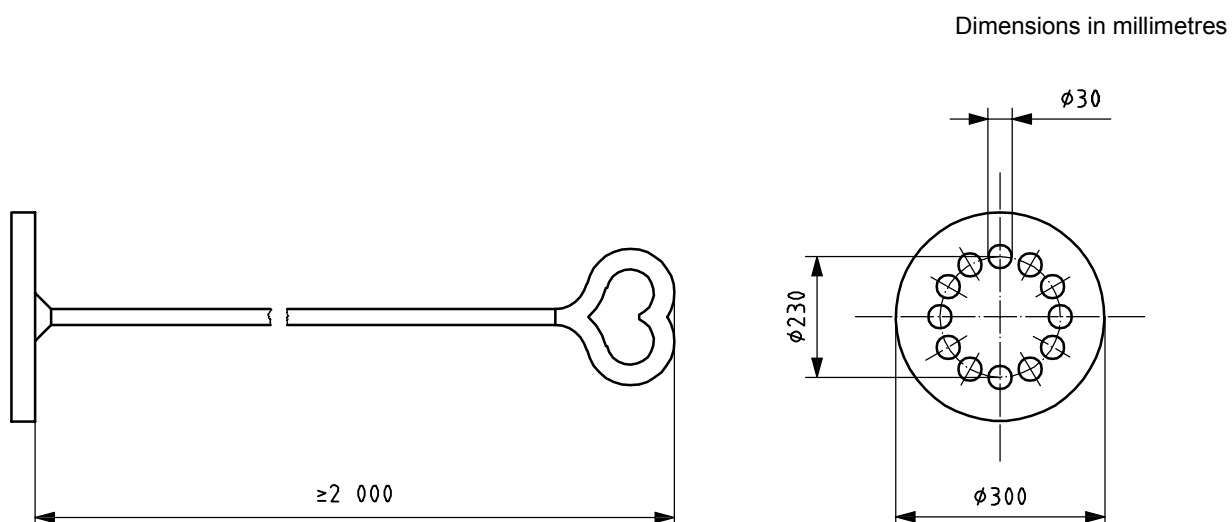


Figure A.2 — Suitable stirrer (plunger) for road, rail and farm tanks

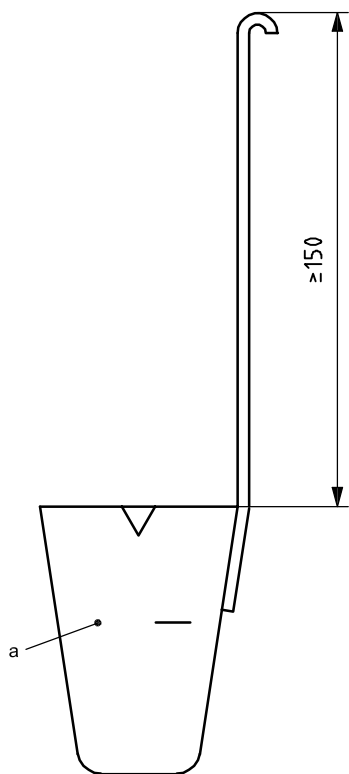
A.1.2 Dippers

See Figure A.3.

A.1.3 Stirrers

See Figure A.4.

Dimensions in millimetres



a ≥ 50 ml

Figure A.3 — Suitable dipper for liquids

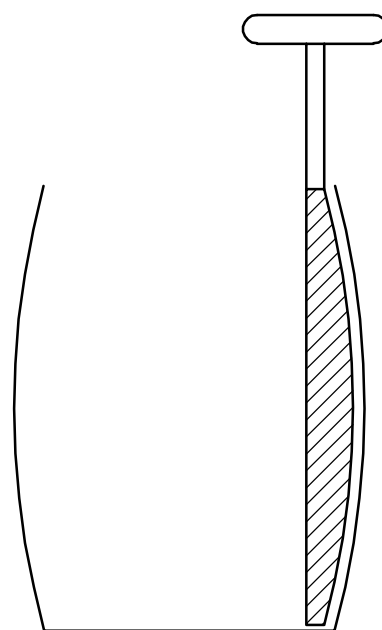


Figure A.4 — Suitable stirrer for mixing sweetened condensed milk in barrels

A.1.4 Borers

A.1.4.1 Sampling of dried milk

See Figure A.5 and Table A.1.

Dimensions in millimetres

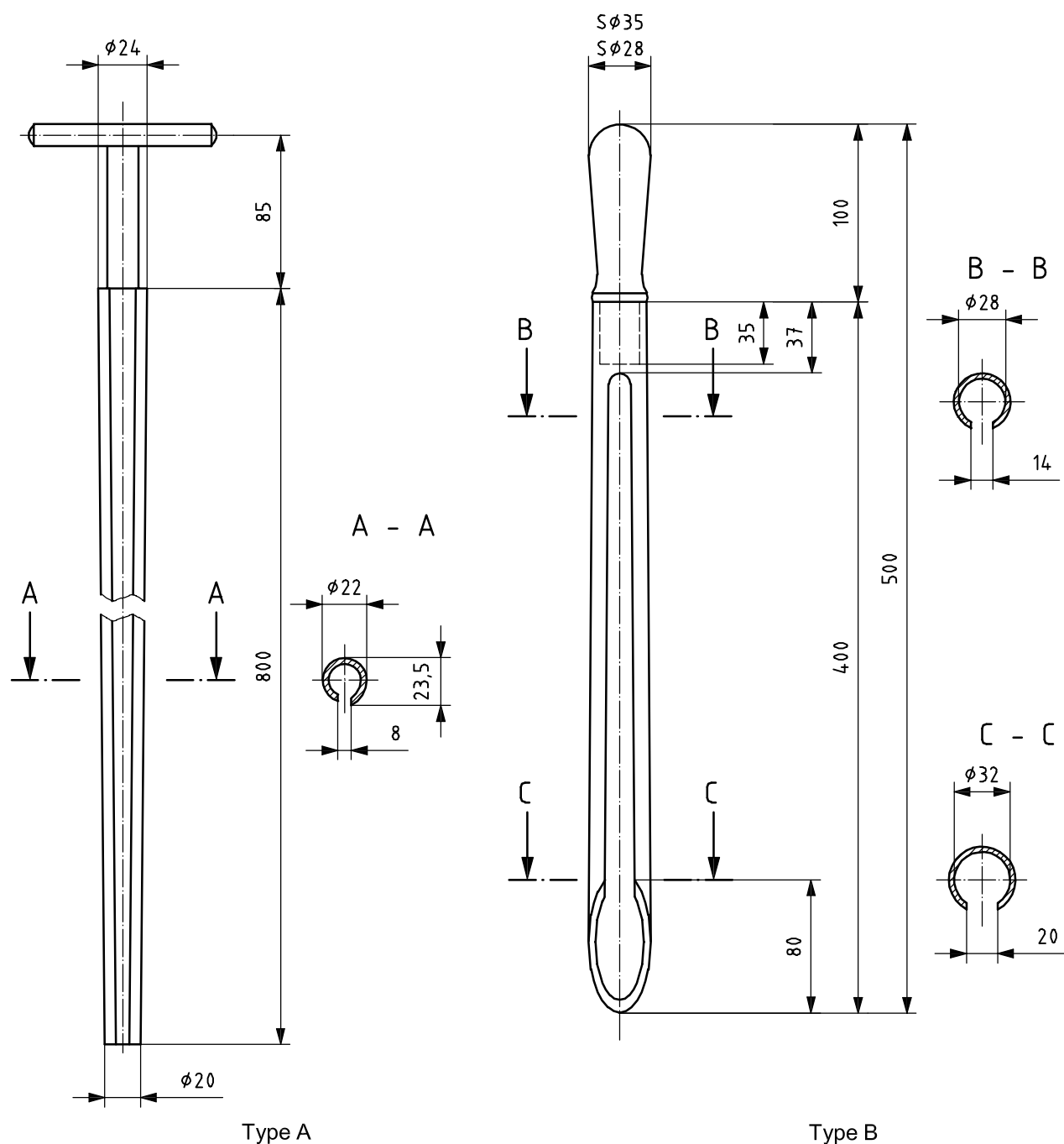


Figure A.5 — Dried milk borers (see Table A.1)

Table A.1 — Borers

Dimension	Type A (long) mm	Type B (short) mm
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	22	28
Slit width at point	4	20
Slit width at grip	14	14

A.1.4.2 Sampling of cheese

See Figure A.6 and Table A.2.

Dimensions in millimetres

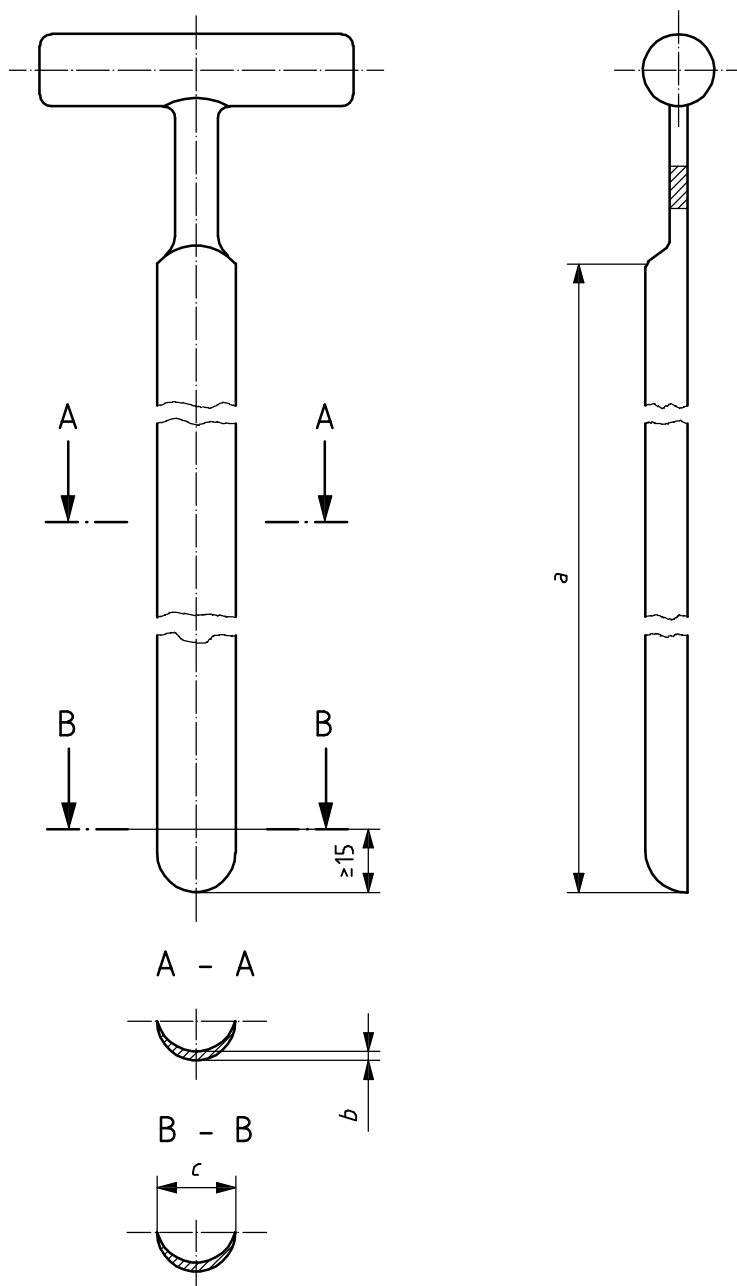


Figure A.6 — Cheese trier (see Table A.2)

Table A.2 — Cheese triers

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

A.1.4.3 Sampling of butter

See Figure A.7 and Table A.3.

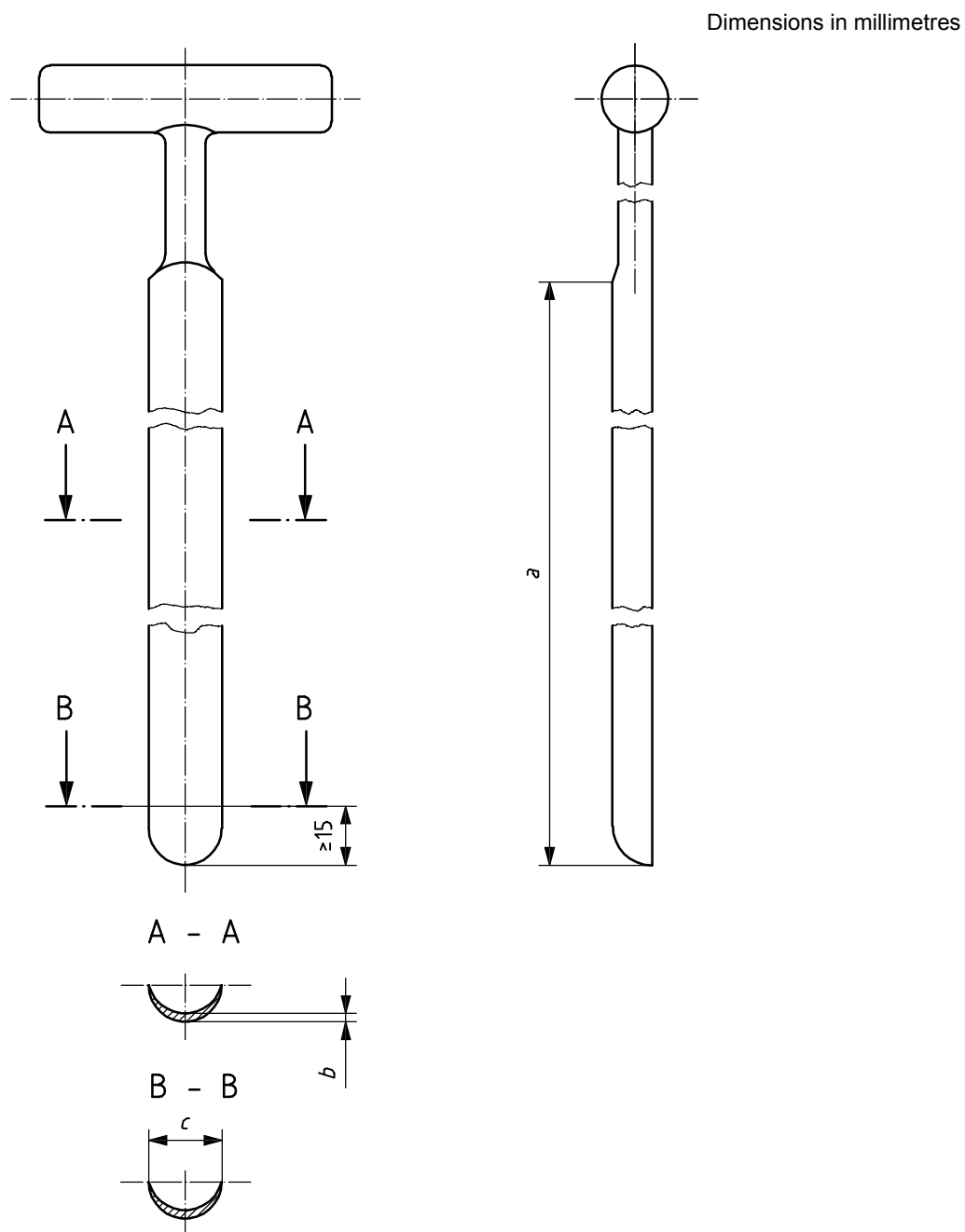


Figure A.7 — Butter trier (see Table A.3)

Table A.3 — Butter triers

Dimension	Type A (long) mm	Type B (medium) mm	Type C (short) mm
Length of blade, <i>a</i>	540	220 to 260	125
Minimum thickness of metal in middle of blade, <i>b</i>	1,8	1,5	1,0
Minimum frontal breadth at 15 mm from end of blade, <i>c</i>	17	17	11

NOTE Normally borers of type B are used. In particular cases type A (long) and type C (short) may also be used.

A.2 Sampling figures

Shapes of samples are shown in Figures A.8 to A.25.

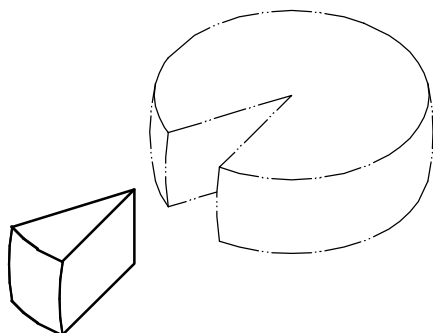


Figure A.8 — Sampling a flat cylindrical shaped cheese by cutting out one sector

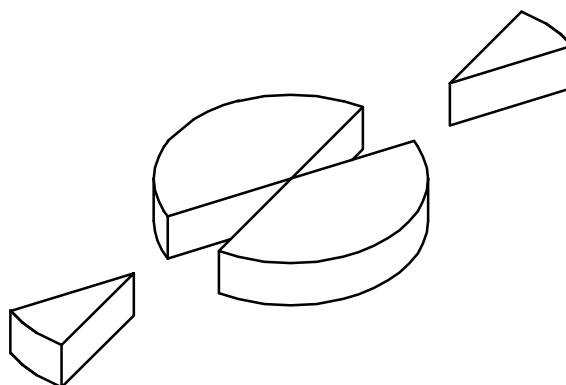


Figure A.9 — Sampling by cutting two sectors

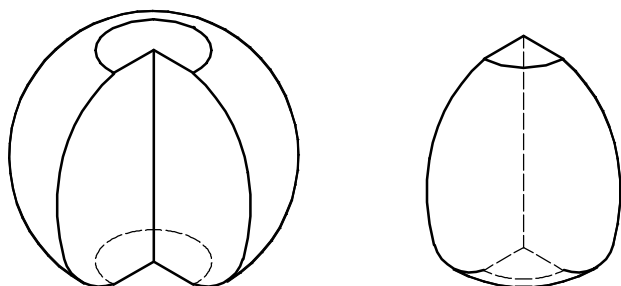


Figure A.10 — Sampling a spherical cheese, also with flattened sides, by cutting out a sector

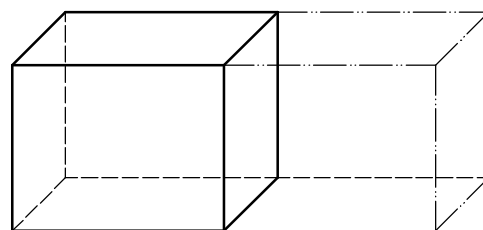


Figure A.11 — Sampling by cutting out a piece from a block-shaped or loaf-shaped cheese of 3 kg to 5 kg mass and in which the largest face is rectangular but not square

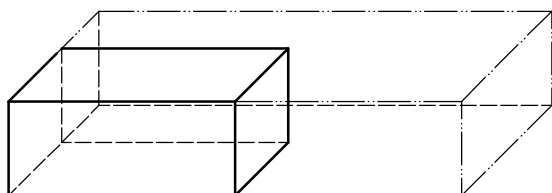


Figure A.12 — Sampling by cutting out a piece from a block-shaped or loaf-shaped cheese of 10 kg to 20 kg mass and in which the largest face is rectangular but not square

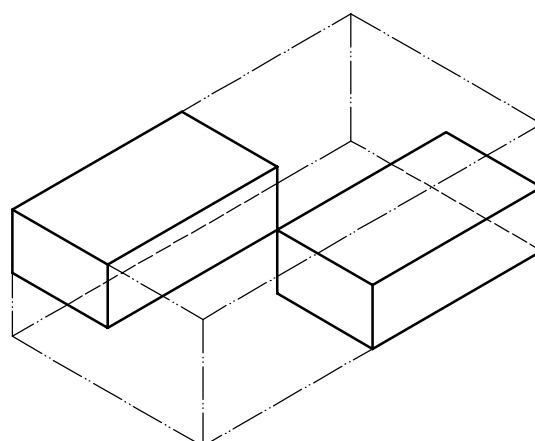


Figure A.13 — Sampling by cutting out a piece from a block-shaped or loaf-shaped cheese in which the largest face is rectangular but not square

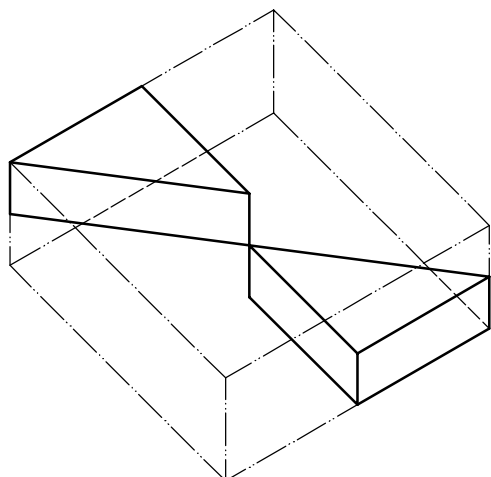


Figure A.14 — Sampling by cutting out a piece from a block-shaped cheese in which the largest face is square

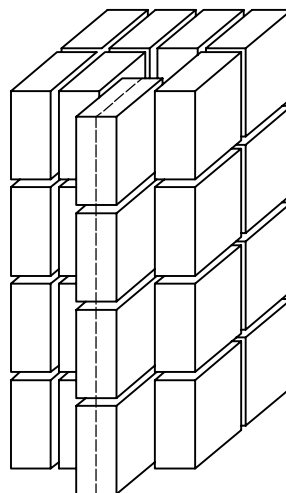


Figure A.15 — Sampling cheeses in brine from containers with more than four blocks of cheese

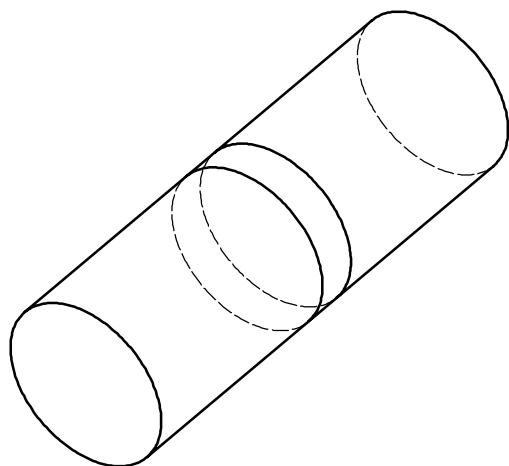


Figure A.16 — Sampling by cutting out one piece

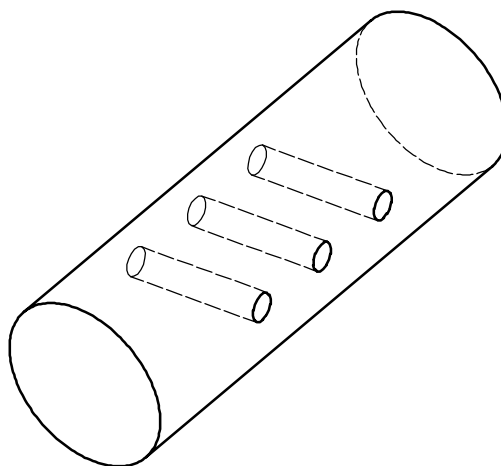


Figure A.17 — Sampling by means of a trier

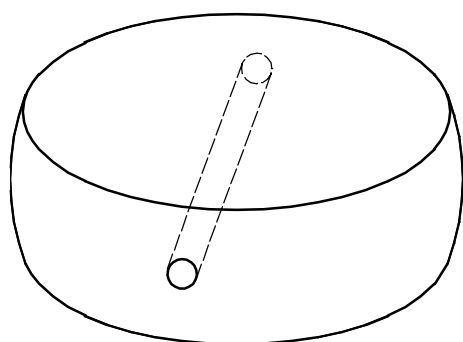


Figure A.18 — Sampling of a flat cylindrical cheese from the side by using a trier

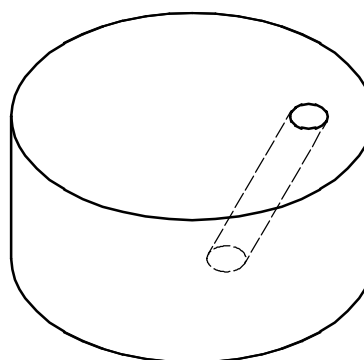


Figure A.19 — Sampling of a wide cylindrical cheese by using an inclined trier, passing from the top through the product

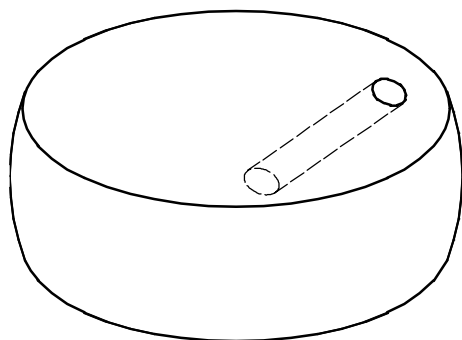


Figure A.20 — Sampling a large flat cylindrical cheese by using a trier

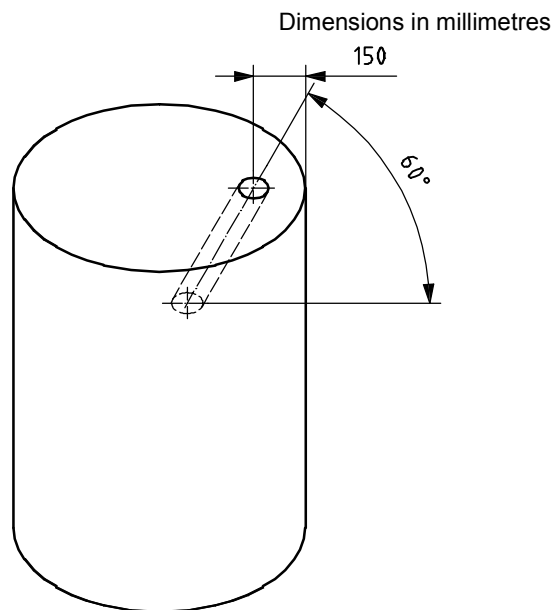


Figure A.21 — Sampling a tall cylindrical cheese by using a trier

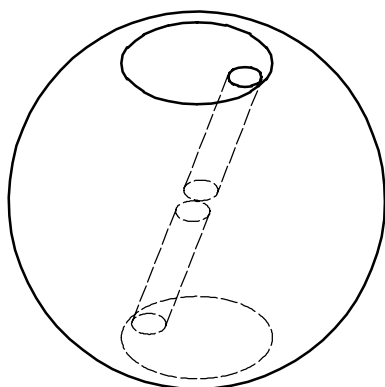


Figure A.22 — Sampling a spherical cheese, also with flattened sides, by using a trier

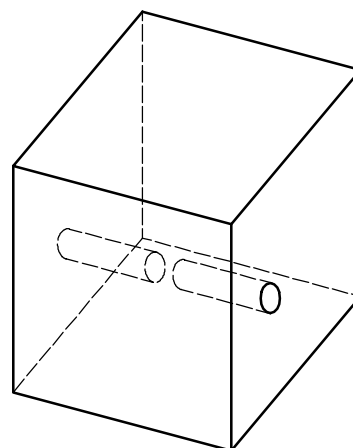


Figure A.23 — Sampling a cubic cheese by using a trier

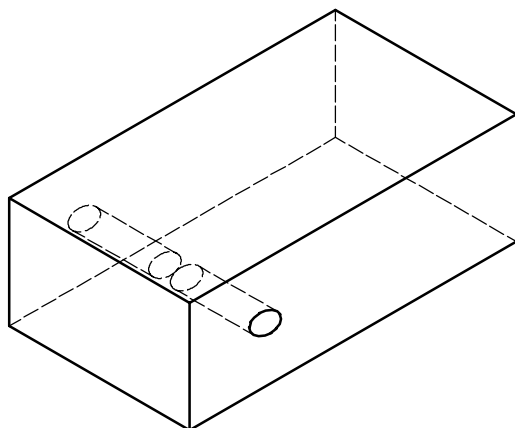


Figure A.24 — Sampling a block-shaped cheese by using a trier

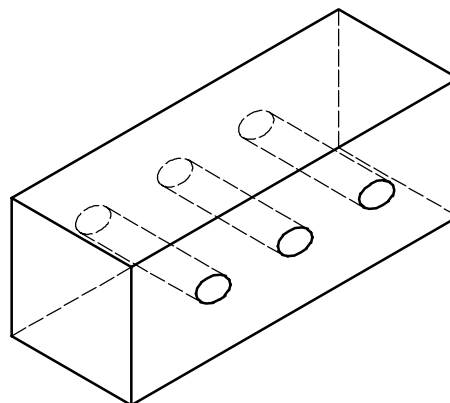


Figure A.25 — Sampling a block-shaped cheese (loaf-shaped) by using a trier

Annex B (informative)

Thermally insulated container for the transport of cooled, frozen and quick-frozen food samples

B.1 General

This annex gives recommendations for the planning and the design of thermally insulated containers which are intended for the storage of cooled, frozen and quick-frozen food samples during transport from place of sampling to testing laboratory in such a manner that the condition of the sample prevailing at the time of sampling will not be materially changed.

The test assessing the insulating effects of the described container is indicated in Clause B.3.

Users of insulated sample containers should regularly measure and record the course of the temperature of samples during transport under practical conditions using suitable equipment (e.g. a Pt 100 thermometer).

Using more technically complex cooling devices for connection to passenger car batteries with active cooling, such as devices using Peltier elements, should be an acceptable alternative to the use of an insulated container.

B.2 Requirements

NOTE An ambient temperature of 30 °C has been chosen for the test conditions of a suitably insulated transport container. This is only a convention for the testing. If this transport container is used in areas with higher ambient temperatures, a higher test temperature should be chosen. The ratio of the amount of the food samples to the refrigerant has to be adapted to these conditions.

B.2.1 Groups of products

B.2.1.1 Group A, with an initial temperature range of 0 °C to + 4 °C.

During storage over 24 h in an insulated transport container (ambient temperature 30 °C ± 1 °C) the temperature of samples of product group A should not drop below 0 °C and should not rise above 5 °C.

B.2.1.2 Group B, which have been precooled to –18 °C or below.

After storage over 24 h in an insulated transport container (ambient temperature 30 °C ± 1 °C) the temperature of samples of product group B should not exceed –18 °C.

B.2.2 Transport container

It may not be necessary to meet all requirements for a container designed only for certain products and/or certain types of analysis.

B.2.2.1 Materials

The container material should:

- a) not transfer to the sample any substances that might be detrimental to health;
- b) not affect the odour and flavour of samples;

NOTE This criterion may be tested with a test food sample (e.g. butter) in accordance with ISO 22935-2 | IDF 99-2^[7] and ISO 22935-3 | IDF 99-3^[8].

- c) be resistant to corrosion, with the exception of the insulating material;
- d) be opaque;
- e) since packaging might be damaged during transport, the container material should:
 - 1) with the exception of the insulating material, be resistant to abrasion,
 - 2) for materials coming into contact with the samples, be resistant to saturated steam and temperatures down to $-20\text{ }^{\circ}\text{C}$,
 - 3) for the materials of the inner surface and the insulating material of type A and type B transport container, be resistant to temperatures down to $-60\text{ }^{\circ}\text{C}$,
 - 4) be resistant to the detergents and disinfectants normally employed in the food industry.

B.2.2.2 Design and construction

Design and construction should ensure that the transport container is:

- a) break-proof and dimensionally stable under conditions of general cargo shipment;
- b) odour-tight as well as liquid-tight;
- c) provided with exterior and interior surfaces that are smooth, free of cracks and gaps, non-porous, easy to clean and easy to disinfect;
- d) depending on the product group, provided with insulation meeting the requirements of the tests specified in Clause B.3;
- e) provided with insulating material that is replaceable;
- f) not subject to pressure rise above that of the atmosphere when dry ice is used.

B.2.3 Refrigerants

The use of crushed ice as a refrigerant is admissible if negative influence on samples can be excluded.

B.2.3.1 Cooling elements

Dimensionally stable cooling elements made of plastics, filled with sodium chloride (NaCl) solution. The surface should be resistant to the detergents and disinfectants normally employed in the food industry and should not affect the odour and flavour of the samples (no extraneous odour absorption).

To avoid local overcooling of the food, small cooling elements of about 200 g have been found to be more advantageous than larger cooling elements. Commercially available cooling elements for household and camping applications normally meet the requirements of this International Standard. Their suitability shall be verified in preliminary tests.

B.2.3.2 Dry ice (solid CO_2)

Dry ice in the form of slabs or cubes should first be crushed to permit more precise metering.

NOTE Dry ice is commercially available. With the aid of appropriate ancillary equipment, dry ice can also be produced from carbon dioxide pressure cylinders with ascension pipes.

B.3 Testing the insulating effect of insulated transport containers

B.3.1 Product group A

B.3.1.1 Apparatus

B.3.1.1.1 Drying cabinet or oven, of adequate size to accommodate the transport container, capable of operating at $30\text{ °C} \pm 1\text{ °C}$.

B.3.1.1.2 Temperature-measuring equipment, consisting of suitable sensors (e.g. Pt 100 thermometer) arranged in the core and marginal areas of the test food and a measuring instrument with a recorder connected to these sensors for recording the measured values. At temperatures from 0 °C to 20 °C , the temperature-measuring equipment should permit measurements which are accurate to within $\pm 0,5\text{ °C}$.

B.3.1.1.3 Cooling elements (see B.2.3.1).

B.3.1.1.4 Test food (e.g. yogurt and milk), taken from similar packages to those containing samples.

NOTE Test packages to ISO 5155^[2] are also suitable for this purpose.

B.3.1.2 Procedure

In the test food, which has been precooled to between 0 °C and 4 °C , the sensors should be arranged in such a manner that the core temperature and the temperature in the area immediately adjacent to the interior wall of the packing can be measured. The sensors should be connected to the recorder. Cooling elements (see B.2.3.1), which have been precooled to approx. -18 °C , should then be added to the test food.

The amount of refrigerant used should be selected to suit the amount of test food and the size of the interior space (see Clause C.1).

The transport container should be closed and transferred to the drying cabinet or oven specified in B.3.1.1.1, which shall be maintained at $30\text{ °C} \pm 1\text{ °C}$. The container should stay there for 24 h. The temperature should be measured and recorded continuously throughout the test duration.

Throughout the test, the temperature of the test food should not drop below 0 °C . If the temperature does drop below this value, the test should be aborted and repeated with a different ratio of test food to refrigerant.

B.3.2 Product group B

B.3.2.1 Apparatus

B.3.2.1.1 Drying cabinet or oven (see B.3.1.1.1).

B.3.2.1.2 Temperature-measuring equipment, comprising one point-measuring sensor and one indicator suitable for measuring temperatures from -50 °C to -10 °C accurately to within $\pm 1\text{ °C}$ or another suitable apparatus with a comparable efficiency.

B.3.2.1.3 A drill or similar tool, for penetrating into the frozen test food in order to permit temperature measurements to be carried out in the interior.

B.3.2.1.4 Dry ice (see B.2.3.2).

B.3.2.1.5 Test food. The packages usually employed for sampling purposes should be used as test food (e.g. ice cream or quick-frozen food).

B.3.2.1.6 Insulating material, or enclosures to prevent direct contact between dry ice and test food, e.g. expanded polystyrene panels, napped plastic sheeting.

B.3.2.2 Procedure

An amount of dry ice estimated to suit the amount of test food used should be introduced into the transport container.

The test food, which has been precooled below $-18\text{ }^{\circ}\text{C}$, should be provided with a bore of 10 mm depth and placed into the transport container in such a manner that it is insulated from the dry ice by a layer of insulating material in accordance with B.3.2.1.6 of at least 10 mm thickness.

The transport container should then be closed and transferred to the drying cabinet or oven (see B.3.1.1.1) which must have been adjusted to a temperature of $30\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$. In the drying cabinet or oven, the transport container should stay there for 24 h. After this period of time, the transport container should be removed from the drying cabinet or oven and opened, and the temperature prevailing in the bore provided in the test food should then be measured immediately, using the temperature-measuring equipment specified in B.3.2.1.2.

.....

Annex C (informative)

Additional information for the use of insulated transport containers

C.1 Food samples

The appropriate ratio of food samples to refrigerant depends on the transport conditions to be expected, such as:

- a) the degree of filling of the interior space;
- b) the transportation time;
- c) the expected mean ambient temperature;
- d) the nature of the food sample.

The ratio should be roughly estimated in preliminary tests.

The results of a test with a suitable transport container for which precooled cooling elements with a total mass of 1 800 g were necessary to meet the requirements of B.3.1 for 3 × 1 l milk (3,5 % mass fraction fat) packed in cartons can be taken as guidance.

C.2 Transport

For the transport of frozen and quick-frozen samples, preliminary tests should be carried out to make sure that an excessive amount of dry ice will not lead to heavy overcooling (e.g. below $-30\text{ }^{\circ}\text{C}$) of the food samples.

C.3 Test

During tests involving the storage of ice cream samples cooled with dry ice for a period of 1 week, no significant decrease of the pH value, which might affect microbiological examinations, could be observed. That means that it is not necessary to provide the samples with gas-tight packing prior to transportation.

Annex D
(informative)

Sampling report for cheese

This sampling report is for cheese only but can be used as a model for other dairy products

Sample

Identification no.

Sample description

Batch/code/markings

Date of expiry

Cheese variety/age

Cheese surface rindless rind rind with smear

(tick all that apply) coating type of coating

.....

No. samples

Sample mass (approximate)

Packaging prepacked aluminium plastic

(tick all that apply) vacuum modified atmosphere

Origin

Place

Date

Location

(name/address of producer/
trader/packaging)

Time of sampling (facultative)

Laboratory/place to be sent

Name of sampling person

Designation of sampling person

Signature

Name of witness

Designation of witness

Countersignature

Sampling

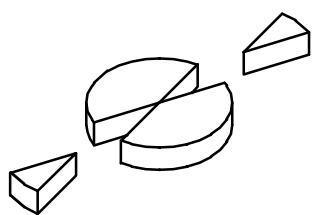
Relevant conditions / circumstances

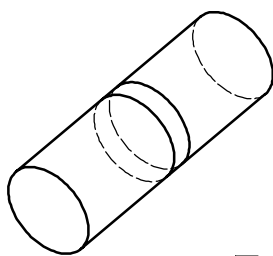
(temperature/humidity)

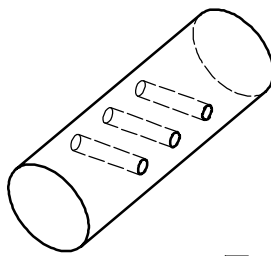
Preservative substance

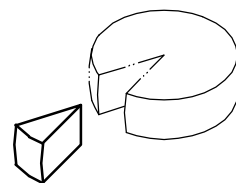
Sampling equipment sterilized by sampling person laboratory others

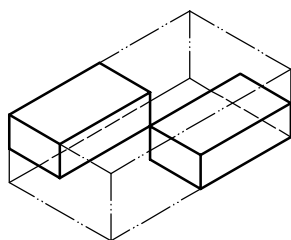
Sampling method (tick all that apply, note differences)

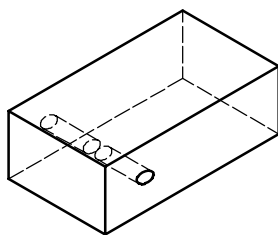


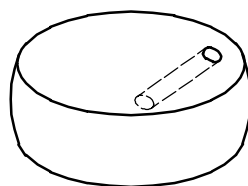


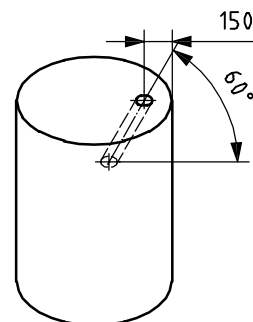


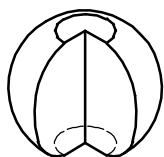


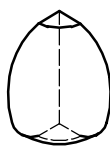


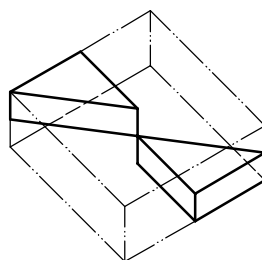


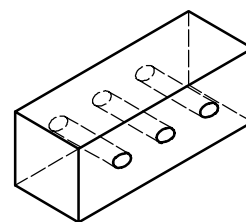


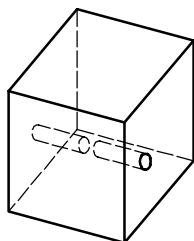


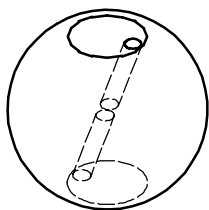


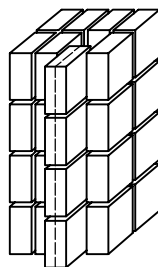












other

Sampling details

Did the samples include the smear? yes no

Did the samples include the rind? yes no

If no, how many millimetres of the rind were cut off?

Are the samples grated? yes no

Sample preparation (preparation of test portion)

How were the above-characterized samples prepared for analyses?

- Including the surface
- Excluding mm of the surface, this equals% mass removal of the original sample
- Grating the samples, using the following equipment:
- Other treatments, namely

Bibliography

- [1] ISO 78-2:1999, *Chemistry — Layouts for standards — Part 2: Methods of chemical analysis*
- [2] ISO 5155, *Household refrigerating appliances — Frozen food storage cabinets and food freezers — Characteristics and test methods*
- [3] ISO 5538|IDF 113, *Milk and milk products — Sampling — Inspection by attributes*
- [4] ISO 7218, *Microbiology of food and animal feeding stuffs — General requirements and guidance for microbiological examinations*
- [5] ISO 8197 (IDF 136A), *Milk and milk products — Sampling — Inspection by variables*
- [6] ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*
- [7] ISO 22935-2|IDF 99-2, *Milk and milk products — Sensory analysis — Part 2: Recommended methods for sensory evaluation*
- [8] ISO 22935-3|IDF 99-3, *Milk and milk products — Sensory analysis — Part 3: Evaluation of compliance with product specifications for sensory properties by scoring*
- [9] INTERNATIONAL DAIRY FEDERATION. Guidelines for sampling equipment and data collection on milk collecting tankers. *Bull. Int. Dairy Fed.*, 1990, (252), pp. 35-48
- [10] RAMSEY, M.H. and ELLISON, S.L.R., editors. *Measurement uncertainty arising from sampling — A guide to methods and approaches*. EURACHEM, Teddington, 2007. 102 p. (EURACHEM/CITAC Guide.) Available (2008-03-19) at: http://www.eurachem.org/guides/UfS_2007.pdf

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