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Milk and milk products — Guidance on sampling

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Foreword

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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.

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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 personnel[1)]

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.

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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	$\leqslant -18$	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.