
**Milk and milk products — Guidance on
sampling**

Lait et produits laitiers — Lignes directrices pour l'échantillonnage

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ISO 707:1997

<https://standards.iteh.ai/catalog/standards/sist/1ffa691d-1b97-4738-9355-8910d898a8df/iso-707-1997>



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Printed in Switzerland

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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.

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.

International Standard ISO 707 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 5, *Milk and milk products*, in collaboration with the International Dairy Federation (IDF) and AOAC INTERNATIONAL, and will also be published by these organizations.

This second edition cancels and replaces the first edition (ISO 707:1985), which has been technically revised.

Annexes A and B form an integral part of this International Standard. Annexes C and D are for information only.

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INTRODUCTION

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

The procedures described in this International Standard are recognized as good practice to be followed whenever practicable. However, it is impossible to lay down fixed rules to be followed in every case, and, even if 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 the sampling and/or arise from a specific analysis to be performed, these requirements should be followed.

The samples obtained by the methods described in this International Standard are "laboratory samples" as defined in ISO 78-2.

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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 sampling of ex-farm milk from individual animals and sampling of milk within quality payment schemes [1]. It is not applicable to the selection of a number of units from a consignment, nor does it apply to subsequent operations in the laboratory.

NOTE - The number of units to be selected for sampling by inspection by attributes may be determined according to ISO 5538:1987 [2]. Sampling for inspection by variables may be determined according to ISO 8197:1988 [3].

2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 78-2:—¹⁾, *Chemistry — Layouts for standards — Part 2: Methods of chemical analysis.*

ISO 7002:1986, *Agricultural food products — Layout for a standard method of sampling from a lot.*

3 DEFINITIONS

For the purposes of this International Standard, the definitions given in ISO 7002 and the following definition apply.

3.1 laboratory sample: Sample as prepared for sending to the laboratory and intended for inspection or testing.

4 GENERAL ARRANGEMENTS

The following instructions are not necessarily applicable for routine sampling.

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

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

1) To be published. (Revision of ISO 78-2:1982)

4.1 Sampling personnel

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

4.2 Sealing and labelling of samples

Samples shall be sealed (in the case of a legal requirement or an agreement between the parties concerned) and a label attached, reproducing integrally the identification of the product, the nature of the product and, at least, the identification number, name and signature (or initials) of the person responsible for taking the samples. If necessary, additional information may be included, such as the purpose of sampling, the mass or volume of the sample, the unit from which the sample was taken and the condition of the product and storage conditions at the moment of sampling.

4.3 Replicate samples

Samples shall be taken in duplicate, or in plural in the case of a legal requirement or an agreement 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 Preparation of a sampling report

Samples shall be accompanied by a report, signed or initialled by the authorized sampling personnel²⁾ and countersigned - as far as necessary or agreed by the parties concerned - by witnesses present. The report shall give the following particulars:

- a) the place, date and time of sampling (mentioning the time of sampling is only required when agreed by the parties concerned);
- b) the names and designations of the sampling personnel and of any witnesses;
- c) the precise method of sampling, if this differs from the instructions given in this International Standard;
- d) the nature and number of units constituting the consignment, together with their batch code markings, where available;
- e) the identification number and any code markings of the batch from which the samples were taken;
- f) the number of samples duly identified as to the batches from which they were taken;
- g) 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.

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

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

5 APPARATUS

5.1 Sampling equipment

5.1.1 General

Sampling equipment shall 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 shall be smooth and free from crevices. All corners shall be rounded. The equipment shall be dry prior to use.

5.1.2 Sampling for microbiological examination

Sampling equipment shall be clean and sterilized prior to use. Disposable plastic equipment shall be sterile. If solder is used in the manufacture of the equipment, it shall be capable of withstanding a temperature of 180 °C. If possible, sterilization shall be performed by one of the two following methods:

Method A: Exposure to hot air at 170 °C to 175 °C for not less than 2 h.

Method B: Exposure to steam at 121 °C ± 1 °C for not less than 20 min in an autoclave.

After sterilization by method A or method B, sampling equipment shall be stored under sterile conditions prior to use.

If, in a particular situation, sterilization by method A or method B is impossible, the following alternative methods, which shall be regarded as secondary methods only, can be used, provided that the sampling equipment is used immediately after sterilization:

Method C: Exposure to a suitable flame so that all working surfaces of the sampling equipment come into contact with the flame;

Method D: Immersion in at least 70% (V/V) ethanol solution;

Method E: Ignition with 96 % (V/V) ethanol.

CAUTION 96 % ethanol is hygroscopic and may change its concentration over a period of time.

Method F: Exposure to a sufficient dose of γ -radiation.

After sterilization by method C, D or E, sampling equipment shall be cooled under sterile conditions or, in the case of method D, be rinsed with the ethanol solution before sampling.

5.1.3 Sampling for chemical and physical analysis and sensory examination

Sampling equipment shall be clean and dry and shall not influence the properties, such as odour, flavour or consistency or the composition of the product. In some cases sterile equipment is required to avoid microbial contamination of the product.

5.2 Sample containers

Sample containers and closures shall be of materials and construction which adequately protect the sample and which do not bring about a change in the sample which could affect the results of subsequent analyses or examinations. Materials which are appropriate include glass, some metals (e.g. stainless steel) and some plastics (e.g. polypropylene). The containers should preferably be opaque. If necessary, transparent filled containers shall be stored in a dark place. Containers and closures shall be dry, clean and either sterile or suitable for sterilization by one of the methods described in 5.1.2.

The shape and capacity of the containers shall be appropriate to the particular requirements of 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.

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

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

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

In the case of small retail containers, these are considered as sample containers; the sample shall consist of the contents of one or more intact, unopened containers.

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

6 SAMPLING TECHNIQUE

Sampling shall be carried out in such a way as to obtain representative samples of the product.

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

Care shall be taken to ensure that when taking samples for sensory examinations the flavour of the samples is not adversely affected by sterilization of the sampling equipment or sampling cocks, e.g. flaming with ethanol.

The precise method of sampling and the mass or volume of product to be taken varies with the nature of the product and the purpose for which samples are required. 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 shall be closed immediately after sampling.

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

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

7 PRESERVATION OF SAMPLES

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

Preservatives 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 shall not be performed,

- c) the nature and quantity of preservative are stated in the sampling report and, preferably, indicated on the label.

8 STORAGE AND TRANSPORT OF SAMPLES

Storage and dispatch of the samples shall be such that the state of the sample at the time of sampling is not adversely affected to any considerable extent. 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 specified by the manufacturer. The storage temperature after sampling should be attained as quickly as possible. The time and temperature shall be considered in combination and not independently. Storage temperatures are given in table 1.

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

| Sam-pling according to clause | Product | Preservation permitted for samples intended for chemical and physical analysis | Temperature ¹⁾ before and during transport (°C) | Minimum sample size ²⁾ |
|-------------------------------|--|--|--|-----------------------------------|
| 9 | Non-sterilized milk and liquid milk | yes | 0 to 4 | 100 ml or g |
| 9 | Sterilized milk, UHT milk and sterilized liquid milk products in unopened containers | no | Ambient, max. 30 | 100 ml or 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 | 0 to 4 | 100 ml or g |
| 10 | Evaporated milk, sweetened condensed milk, and milk concentrates | no | Ambient, max. 30 | 100 g |
| 11 | Semi-solid and solid milk products except butter and cheese | no | 0 to 4 | 100 g |
| 12 | Edible ices and semi-finished ice products | no | - 18 or lower | 100 g |
| 13 | Dried milk and dried milk products | no | Ambient, max. 30 | 100 g |
| 14 | Butter and butter products | no | 0 to 4 (in the dark) | 50 g |
| 15 | Butterfat (butter oil and similar products) | no | 0 to 4 (in the dark) | 50 g |
| 16 | Fresh cheese | no | 0 to 8 | 100 g |
| 16 | Processed cheese | | Ambient, max. 30 | 100 g |
| 16 | Other cheeses | | 4 to 8 | 100 g |

1) The temperatures mentioned in the table are meant as general guidelines. For specific analysis purposes, other temperatures may be more appropriate. It may be, under certain practical conditions, not always easy or even impossible to maintain the "ideal" or desirable temperatures specified in this table. 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.

2) A larger sample size may be necessary according to the tests required and the type of product.

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

9 MILK AND LIQUID MILK PRODUCTS

9.1 Applicability

The instructions given in this clause are applicable to raw and heat-treated milk (except raw milk from individual animals and raw milk taken within quality payment schemes), whole, partly skimmed and skimmed milk, flavoured milk, cream, fermented milk, buttermilk, liquid whey and similar products.

9.2 Apparatus

Sampling equipment shall correspond to that given in clause 5.

9.2.1 Apparatus for manual mixing

Agitators for mixing liquids in bulk shall 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 agitators can be recommended for all purposes, but they shall 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 agitation in small vessels

For mixing liquids in small vessels (e.g. in buckets and cans) a stirrer (plunger) of the design and dimensions as shown in figure A.1 is suitable. The length shall 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 as 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 mixed in the tank or vessel determines the technical characteristics and construction of built-in agitators.

Various types of agitators are used but no attempt has been made to describe any of them in this International Standard.

9.2.2.2 Removable agitators

Removable agitators are usually provided with a propeller and are introduced into transport, road and rail tanks through the manhole.

Best stirring results are achieved at a depth corresponding to 0,7 of the filling height. It is recommended that the stirrer be inclined 5° to 20° as this allows vertical mixing of the liquor liquid as well as horizontal movement.

9.2.3 Apparatus for taking samples

9.2.3.1 Apparatus for sampling

A dipper of the shape and size as shown in figure A.3 is suitable to be used 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 shall be such that they are almost completely filled by the sample and allow proper mixing of the contents before testing, but avoid churning during transport.

9.2.3.3 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. The equipment described in 9.2.1 and 9.2.2 may be used.

Take the sample immediately after mixing. The size of the sample shall not be less than 100 ml.

9.3.1 Sampling for microbiological examination

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

Sterilize sampling equipment and sample containers as described in 5.1.2. Proceed as described in 9.3.2, however using aseptic techniques.

9.3.2 Sampling for chemical and physical analysis and sensory examination

In certain cases sampling equipment and sample containers shall be sterile for chemical and physical analysis and sensory examination.

9.3.2.1 Small vessels, milk buckets and cans

Thoroughly mix the milk, for example by transfer, stirring or plunging (plunger).

9.3.2.2 Milk tanks or vats

Mechanically agitate 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).