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# International Standard



# 707

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Milk and milk products — Methods of sampling

*Lait et produits laitiers — Méthodes d'échantillonnage*

First edition — 1985-02-15

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UDC 637.1/.3 : 620.11

Ref. No. ISO 707-1985 (E)

Descriptors : agricultural products, dairy products, milk, sampling, sampling equipment, storage.

## 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.

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

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

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

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

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# Milk and milk products — Methods of sampling

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### 0 Introduction

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

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

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

### 1 Scope and field of application

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

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

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

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

### 2 References

ISO 3918, *Milking machine installations — Vocabulary*.

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

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

### 3 Definitions

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

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

1) At present at the stage of draft.

## Section one : General considerations

### 4 Administrative arrangements

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

#### 4.1 Sampling personnel

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

#### 4.2 Representation of the parties concerned

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

#### 4.3 Sealing and labelling of samples

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

#### 4.4 Replicate samples

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

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

#### 4.5 Preparation of a sampling report

Samples shall be accompanied by a report, signed by the authorized sampling person<sup>1)</sup> and countersigned by any witnesses present. The report shall give the following particulars :

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

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

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

### 5 Apparatus

#### 5.1 Sampling equipment

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

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

##### 5.1.1 Sampling for microbiological examination

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

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

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

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

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

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

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

### 5.1.2 Sampling for chemical and/or physical analysis

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

### 5.1.3 Sampling for sensory examination

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

## 5.2 Sample containers

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

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

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

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

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

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

## 6 Sampling technique

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

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

The sample container shall be closed immediately after sampling.

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

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

## 7 Preservation and storage of samples

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

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

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

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

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

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

## 8 Dispatch of samples

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

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

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

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

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## Section two : Methods of taking samples

### 9 Milk and liquid milk products

#### 9.1 Applicability

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

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

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

#### 9.2 Apparatus<sup>1)</sup> (see also 5.1)

##### 9.2.1 Plungers and agitators

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

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

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

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

##### 9.2.2 Dippers and extractors

###### 9.2.2.1 Dippers

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

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

###### 9.2.2.2 Extractors

###### 9.2.2.2.1 Sample extraction cylinder

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

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

###### 9.2.2.2.2 Chamber probe

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

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

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

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

###### 9.2.2.2.3 Probe or partial sample extractor

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

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

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

### 9.2.3 Sample containers

See 5.2.

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

## 9.3 Procedure

### 9.3.1 General

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

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

The equipment described in 9.2.2.3 may be used.

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

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

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

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

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

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

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

### 9.3.2 Sampling for microbiological examination

Take a sample of not less than 200 ml.

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

Sterilize the sampling equipment and containers as described in 5.1.1.

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

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

#### 9.3.3.1 Sample size

Take a sample of not less than 200 ml.

#### 9.3.3.2 Whole milk

##### 9.3.3.2.1 Milk from individual animals

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

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

##### 9.3.3.2.1.1 Hand milking

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

##### 9.3.3.2.1.2 Machine milking

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

The following shall be noted, according to the case :

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

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

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

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

#### 9.3.3.2.2 Sampling from small vessels — Milk buckets and cans

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

#### 9.3.3.2.3 Sampling from a weighbowl

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

#### 9.3.3.2.4 Sampling from refrigerated farm milk tanks or vats

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

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

#### 9.3.3.2.5 Sampling a divided bulk

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

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

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

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

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

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

#### NOTES

1 When clean compressed air is recommended, it is necessary to use filtered compressed air from which all contaminants (including oil, water and dust) have been excluded. The possibility of microbiological contamination should not be forgotten.

2 Minimal air pressure and volume should be used to prevent the development of rancid flavour.

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

#### 9.3.3.3 Skimmed milk and whey

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

#### 9.3.3.4 Buttermilk, fermented milk, flavoured milk

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

#### 9.3.3.5 Cream

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

### 9.4 Preservation, storage and dispatch of samples

See clauses 7 and 8.

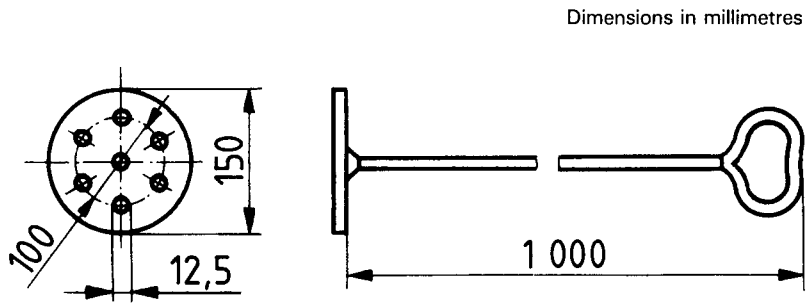


Figure 1 – Recommended plunger for cans and buckets

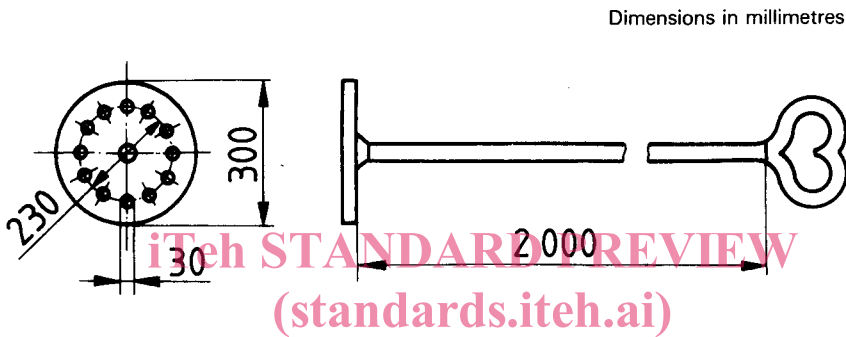


Figure 2 – Suitable plunger for road, farm and rail tanks

<https://standards.iteh.ai/catalog/standards/sist/9f1f2a3d-704b-456a-9f23-4e50e163f79c/iso-707-1985>

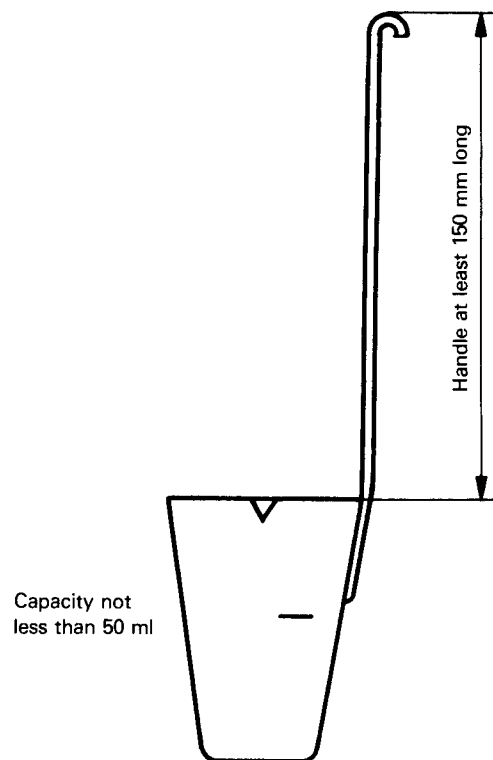


Figure 3 – Suitable dipper for liquids