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Animal and vegetable fats and oils — Sampling

Corps gras d'origines animale et végétale — Échantillonnage

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Contents

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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 5555 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Sub-Committee SC 11, *Animal and vegetable fats and oils*.

This second edition cancels and replaces the first edition (ISO 5555:1983), which has been technically revised.

Annexes A, B and C of this International Standard are for information only.

Introduction

Practically all fats are marketed on the basis of the result of analysis of a sample of the fat. Disputes are invariably settled by reference to this sample. Therefore careless or inaccurate sampling could lead to misunderstandings, delays and unwarranted financial adjustments.

Correct sampling is a difficult procedure and one that requires the most careful attention. Emphasis cannot therefore be too strongly laid on the necessity of obtaining properly representative samples for analysis.

The sampling procedures given in this International Standard are recognized as good practice and it is strongly recommended that they be followed whenever practicable. It is recognized that it is difficult to lay down fixed rules which can be followed in every case; particular circumstances may render desirable some modification of the methods specified.

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Animal and vegetable fats and oils — Sampling

1 Scope

This International Standard describes methods of sampling crude or processed animal and vegetable fats and oils, referred to as fats hereafter whatever the origin and whether liquid or solid. It also describes the apparatus used for this process.

NOTE 1 Methods of sampling milk and milk products, including milk fats, are specified in ISO 707.

2 Definitions

For the purposes of this International Standard, the following definitions apply.

2.1 consignment: The quantity of fat delivered at one time and covered by a particular contract or shipping document. It may be composed of one or more lots or parts of lots.

2.2 lot: An identified quantity of fat, presumed to be of uniform characteristics.

2.3 increment: A quantity of fat taken at one time from one place in a lot.

2.4 bulk sample: The quantity of fat obtained by combining the various increments from a lot in amounts proportional to the quantities they represent.

NOTE 2 The bulk sample should be representative of the lot and take account of any contractual requirements.

2.5 laboratory sample: The quantity of fat, obtained from the bulk sample after suitable homogenization and reduction in size, which is representative of the lot and intended for laboratory examination.

2.6 conventional mass per volume sample; "litre weight in air" sample: The quantity of fat taken for the mass of fat to be calculated from the volume.

3 General

The object of sampling and of preparing samples is to obtain from a consignment (which may be in lots) a manageable quantity of the fat, the properties of which correspond as closely as possible to the properties of the consignment sampled.

The methods of taking samples described below are intended for the guidance of experts and can be used for

a) consignments in bulk, e.g. in land tanks, ships' tanks, tank wagons and tank cars; and

b) consignments consisting of a number of packages, e.g. barrels, drums, cases, tins, bags and bottles.

4 Apparatus

4.1 General

For a particular purpose, the choice of sampling instruments and their suitability depend on the skill of the sampler in following the recommended procedures.

In all circumstances, it shall be borne in mind whether the sample is intended for preliminary inspection, for analysis, or for the determination of conventional mass per volume ("litre weight in air").

4.2 Materials

Sampling instruments, ancillary apparatus and sample containers (including caps) shall be made of materials which are chemically inert to the fat being sampled and they shall not catalyse chemical reactions.

For sampling instruments, stainless steel is the most suitable material. Aluminium may be used only when the acidity is low but not for the storage of samples.

Plastics, copper and copper alloys shall not be used nor any toxic material.

WARNING — If glass apparatus is used for a particular reason, great care shall be taken to avoid breakages.

4.3 Examples of types of sampling instruments

4.3.1 General

Many forms and types of sampling instruments exist, and the instruments described in this International Standard are only examples of those commonly used.

The instruments are all simple, robust and easily cleaned. They can be used for all the sampling operations described in this International Standard with all types of fats commonly found in commerce.

Certain basic requirements are common to all sampling instruments; e.g. they shall be capable of taking a representative sample from a required level or area and of preserving the integrity of the sample until it can be transferred to a sample container. Ease of cleaning, practical size and ability to withstand rough usage are other essential characteristics.

Alternative designs of instruments to those described in this International Standard may be used, e.g. to meet the needs of individual users.

The instruments can be of various sizes according to the quantity of sample required and the accessibility of the fat.

The types of apparatus mentioned in 4.3.2, 4.4.1, 4.4.2 and 4.4.5 are described in annex B.

4.3.2 Sampling instruments

4.3.2.1 Simple weighted sample can, see B.1 and figure B.1.

4.3.2.2 Weighted cage for sample bottle, see B.2 and figure B.2.

4.3.2.3 Valve sampling cylinder (sinker sampler), see B.3 and figure B.3.

4.3.2.4 Bottom samplers, see B.4 and figure B.4.

4.3.2.5 Sampling tubes, see B.5 and figure B.5.

4.3.2.6 Sampling scoops, see B.6 and figure B.6.

4.4 Ancillary apparatus

4.4.1 Water-finding rule, see B.7 and figure B.7.

4.4.2 Ullage rule, see B.8 and figure B.8.

4.4.3 Labels, adhesive or tie-on and **sealing apparatus**, see also clause 7.

4.4.4 Thermometers.

4.4.5 Measuring tape and **weight**, see B.9.

4.5 Sample containers

4.5.1 Sample containers, made of the materials specified in 4.2.

NOTE 3 Glass containers are recommended.

5 Sampling technique

5.1 All sampling operations shall be performed by an operator with clean hands or wearing gloves (clean plastics or cotton gloves may be used).

5.2 The apparatus and sample containers shall be clean and dry prior to initial use. During the sampling of similar fats, the same sampling apparatus may be used provided that it is adequately flushed with the fat to be sampled to ensure that none of the previous sample remains.

5.3 Sampling shall be carried out in such a manner as to protect the samples, the fat being sampled, the sampling instruments and the sample containers from adventitious contamination with rain, dust, etc.

5.4 All extraneous material shall be removed from the outside of the sampling instruments before the instruments are emptied.

5.5 If heating is necessary to facilitate sampling, it is important that fats are not overheated. It is recommended, in accordance with usual practice, that the temperature of a bulk of fat in a storage tank should not be raised by more than 5 °C per day.

The area of heating coils should be large in relation to the volume of fat and their temperature kept as low as possible to avoid local overheating. Steam, at a maximum pressure of 150 kPa (1,5 bar) gauge reading (128 °C) or hot water (only if the heating coils are self-draining) should be used. Care is required to prevent contamination of the fat by steam or water.

The temperature of the fat during sampling should be within the range indicated in annex A.

5.6 After samples have been taken as specified in 6.1 to 6.8, as appropriate, laboratory samples shall be prepared as specified in 6.9.

6 Methods of sampling

6.1 General

6.1.1 Containers for transport and storage of fats

A distinction is made between the following types of containers from which samples are taken and which may affect the method of sampling to be used:

a) vertical cylindrical land tanks (see 6.2);

b) ships' tanks (see 6.3);

c) tank wagons or cars (see 6.4);

d) horizontal cylindrical tanks (see 6.4);

e) weigh tanks (see 6.5);

f) pipelines during transfer (see 6.6);

g) packages, e.g. barrels, drums, cases, tins, bags and bottles (see 6.8).

The procedure is also given for sampling for the determination of conventional mass per volume ("litre weight in air") (see 6.7).

6.1.2 Water

Water may be present as free water at the bottom (i.e. separated water), as an emulsion layer or as water in suspension in the fat in any of the containers described in 6.1.1, but during usual operations the fat is unlikely to remain static for sufficient time in weigh tanks and pipelines for the water to settle to the bottom.

Measurement of water is mostly conducted in vertical storage tanks (see 6.2), but the same principles apply to the containers listed other than pipelines.

The presence of water may be detected with a bottom sampler (B.4) and free water may be measured with a water-finding rule (B.7) and water-finding paste or paper, or by electronic means.

Whichever method is used, accurate determination of water content is often difficult because of the indistinct separation of free water and the emulsion layer and water in suspension, in the lower layers of the fat.

It may also be useful to determine whether the water is fresh or sea water.

6.2 Sampling from vertical cylindrical land tanks

6.2.1 Preliminary operations

6.2.1.1 Sediment, emulsion and free water

Determine whether there is sediment or an emulsion layer or free water at the bottom of the tank by means of a bottom sampler and/or water detectors as described in 6.1.2.

The careful application of heat followed by standing assists the water in suspension to settle out (see 5.5).

It is desirable, so far as possible, to run off free water before sampling, subject to contractual requirements and agreement of contract parties, and to measure the amount removed.

6.2.1.2 Homogenizing

Before sampling begins, it is essential that the whole of the product is as homogeneous and as nearly liquid as possible.

Check the fat in the tank for uniformity by examining increments taken from various levels using a simple weighted sample can (B.1), a weighted cage for sample bottle (B.2) or a valve sampling cylinder (B.3) and from the bottom using a bottom sampler (B.4).

If layers of different composition are present, homogeneity can, in most cases, be obtained by heating as described in 5.5.

If heating is not permissible because of the nature of the fat, or if it is not necessary, or if heating has to be avoided for any other reason, the fat can be made homogeneous by blowing nitrogen through it.

NOTE 4 If a fat is know to be inhomogeneous and nitrogen is not available, the parties may agree to blowing dry air through the product, although this process is to be deprecated especially in the case of marine oils, because it may cause deterioration of the fat by oxidation. Details of such operations should be included in the sampling report sent to the laboratory.

6.2.2 Procedure

6.2.2.1 General

Sample each tank separately.

6.2.2.2 Inhomogeneous fats

If the contents of the tank are not and cannot be made homogeneous, a simple weighted sample can (B.1), a weighted cage for sample bottle (B.2) or a valve sampling cylinder (B.3) is generally used for sampling, plus a bottom sampler (B.4).

Take increments at depths of every 300 mm, from top to bottom, until the layer of different composition is reached. In this layer, take more increments (for example at depths of every 100 mm). Also take a bottom sample.

Mix appropriate increments to give

a) a sample of the clear oil;

b) a sample of the separated layer.

Prepare a bulk sample by mixing samples a) and b) in proportion to the respective sizes of the two layers, taking care to ensure that the proportions are as exact as possible.

Prepare the number of bulk samples given in table 1, preparing at least one bulk sample for each tank.

Table 1 — Number of bulk samples to be taken from each ship's tank or land tank

Mass of tank contents tonne	Number of bulk samples for each tank
⩽ 500	1
> 500 ⩽ 1 000	2
> 1 000	1 for every 500 t or part thereof

6.2.2.3 Homogeneous fats

If the contents of the tank are homogeneous, use one of the same sampling instruments as in 6.2.2.2, but in this case take at least three increments, "top", "middle" and "bottom".

NOTE 5 The "top" increment should be taken at a level of one-tenth of the total depth from the surface, the "middle" increment should be taken at a level of one-half of the total depth and the "bottom" increment should be taken at a level of nine-tenths of the total depth.

Prepare a bulk sample by mixing in the proportions one part from each of the top and bottom increments and three parts from the middle.

Prepare the number of bulk samples given in table 1, preparing at least one bulk sample for each tank.

6.3 Sampling from ships' tanks

The shape and disposition of ships' tanks make sampling more difficult than in vertical cylindrical land tanks. Usually, sampling is carried out during transfer as described in 6.6. If samples are to be taken from ships' tanks, use (as far as possible) the procedure described in 6.2, including the preliminary operations, such as heating.

Sample each tank separately. Prepare the number of bulk samples indicated in table 1. In preparing the bulk sample from increments taken from a tank, make allowance for the shape of the tank by mixing, as far as possible, the increments in the corresponding proportions.

Barge tanks should preferably be sampled as soon as they have been filled.

6.4 Sampling from tank wagons or cars and horizontal cylindrical tanks

Samples should preferably be taken as soon as the tanks have been filled, i.e. before settling occurs possibly leading to fractionating or layering.

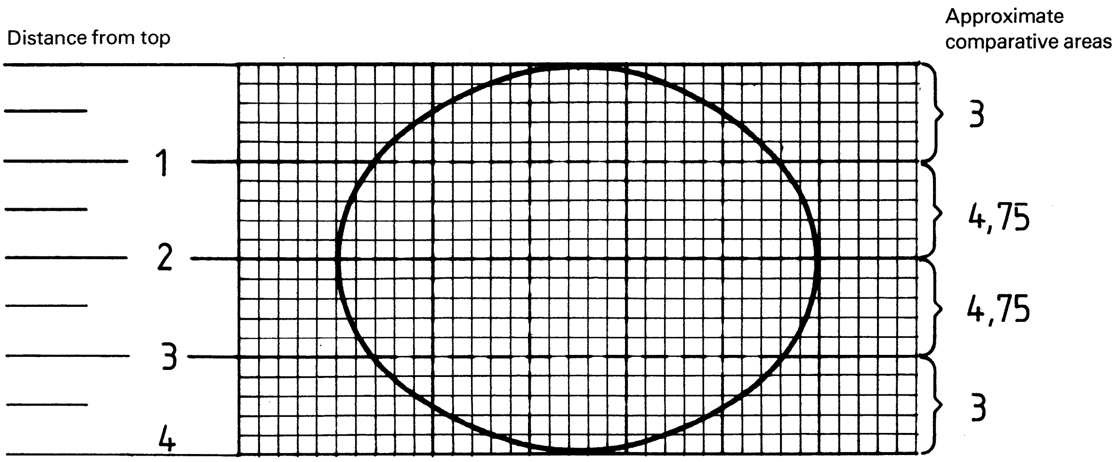
Take the increments by means of a simple weighted sample can (B.1), a weighted cage for sample bottle (B.2) or a valve sampling cylinder (B.3) by the procedure described in 6.2.2.

If the increments cannot be taken immediately after the tanks have been filled, perform a preliminary test for the presence of free water as a bottom layer. If free water is present, and with the agreement of the parties concerned, remove it by opening the bottom tap, measure the amount of water removed and report this to the buyer and seller or to their representatives.

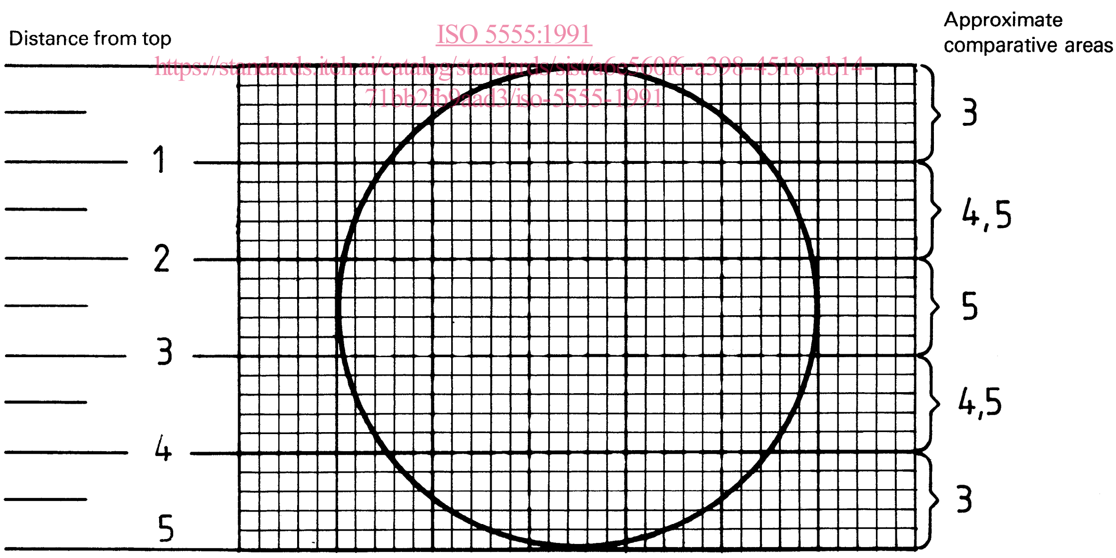
Then make the contents sufficiently homogeneous by blowing nitrogen[1] through and/or by heating until they are entirely liquid, provided that the particular fat being sampled will not suffer from such treatment.

If circumstances require that static liquid has to be sampled in a tank wagon or horizontal cylindrical tank, without mixing as indicated above, the greatest care is necessary in taking the correct proportion of sample relative to the liquid depth.

1) See note 4 in 6.2.1.2.



a) Elliptical cross-section horizontal tank



b) Cylindrical cross-section horizontal tank

Figure 1 — Cross-sections of typical tanks

If a valve sampling cylinder is used to sample every 300 mm of depth of the tank wagon, reference should be made to figure 1 to determine the proportions of the increments, from each 300 mm level, that should be mixed to form the bulk sample. This fairly simple method (of drawing to scale, on graph paper, the cross-section of tanks of any shape or size) can be used to indicate the proportions of increments for mixing.

Inclined tanks shall be sampled by the methods described in 6.3 for ships' tanks. The tank-shape corrections described above are not applicable to inclined or irregular tanks.

Prepare bulk samples from the increments in proportion to the cross-sections of the tanks.

6.5 Sampling from weigh tanks

Weigh tanks should be sampled immediately after they have been filled, before settling occurs.

Take the sample by allowing a sampling instrument to sink to the middle and fill. If unavoidable delay occurs, which may result in the settling of sediment to the bottom of the tank, agitate the contents before sampling, or carry out sampling at depths of every 300 mm.

If the tank is closed, sample from a horizontal drip tap (as described in 6.6.2) immediately after filling.

Prepare bulk samples from the increments in proportion to the cross-sections of the tanks.

6.6 Sampling from pipelines during transfer

6.6.1 General

This method shall be used only if the fat is entirely liquid and contains no components which could block a tap or dripcock. Any water-containing emulsion, for example fore-pump oil, shall be drawn off, stored, sampled and weighed separately.

Samples from very large bulk quantities may be taken during transfer by means of frequent removal of increments from the flow at regular intervals when the tank is being emptied. This method is particularly easy to apply if the oil is transferred from a tank fitted with a weigh tank meter.

Alternatively, sampling may be carried out by means of a side or secondary stream tapped from the main stream, but it is difficult to ensure accurate sampling by this method.

6.6.2 Taps or dripcocks

The tap or dripcock shall be fed from a nozzle of diameter not less than 9,5 mm, capable of being inserted in the centre or at one-third diameter of the main discharge pipeline and facing the flow of liquid. Taps let into the side or bottom of the pipeline are not acceptable. The tap or dripcock shall be introduced, if possible, into a horizontal section of the main pipeline, as far from elbows and T-joints as possible, and preferably within 10 m to 50 m of the pressure side of the pump. A petcock is not recommended. The sampling line shall be of diameter not less than 9,5 mm and shall fall continuously to its outlet. The tap or dripcock shall be of such design as to be easily and quickly cleaned in case of blockage.

To allow the clearing of a pipeline blockage and the pigging of the main flow line, a means of withdrawing the small bore pipes should be provided.

Heating and insulation should be provided for fats of high viscosity or high melting point.

6.6.3 Procedure

Regulate the rate of flow in the main pipeline to ensure sufficient turbulence to mix completely the product in the pipeline. Maintain the rate of flow as constant as possible.

A cover shall be fitted over the whole apparatus and the sample containers to prevent adventitious contamination.

Carefully and immediately mix all the sample taken from the dripcock, after completion of the discharge, to form the bulk sample from which the laboratory samples are to be taken.

In view of the possibility of blockage of the dripcock etc. by pieces of dirt, and of variations that inevitably occur in the flow, it is essential that an experienced sampler is present constantly throughout the sampling operation.

6.6.4 Minimum size of bulk sample

Prepare bulk samples during transfer from each tank of the minimum size specified in table 2.

Table 2 — Minimum size of bulk sample when sampling from pipelines

Mass of tank contents tonne	Minimum size of bulk sample litre
⩽ 20	1
> 20 ⩽ 50	5
> 50 ⩽ 500	10