INTERNATIONAL STANDARD

ISO 19219

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Animal and vegetable fats and oils — Determination of visible foots in crude fats and oils

Corps gras d'origines animale et végétale — Détermination de la teneur en sédiments visibles dans des graisses et huiles brutes

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Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to produce 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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 19219 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

Annex A of this International Standard is for information only.

Animal and vegetable fats and oils — Determination of visible foots in crude fats and oils

1 Scope

This International Standard specifies a method for the determination in crude fats or oils of visible matter which can be separated by gravity.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 661:1989, *Animal and vegetable fats and oils — Preparation of test sample*

3 Terms and definitions

For the purposes of this International Standard, the following term and definition applies.

3.1
visible foots
insoluble matter in fats and oils, together with occluded oil, which settles out from oil or fat within 96 h at the temperature specified in this International Standard

NOTE 1 “Foots” is a term that was originally used to describe those impurities that precipitate from raw linseed oil during storage and subsequently settle to the bottom (foot) of a storage tank.

NOTE 2 Visible foots are quantified by storage of a sample of the homogenized fat or oil for a period of 96 h at 20 °C or 10 °C above the melting point, whichever is the higher.

4 Principle

A homogenized test portion of crude fat or oil is allowed to stand at a controlled temperature for a period of 96 h. The volume of separated material, called “visible foots”, is read off from the graduated vessel.

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Sediment tube, pear-shaped, used when the sediment is $\leqslant$ 1,5 ml per 100 ml.

See Figure 1.

5.2 Sediment tube, cone-shaped, used when the sediment is $>$ 1,5 ml per 100 ml.

See Figure 2.

Dimensions in millimetres

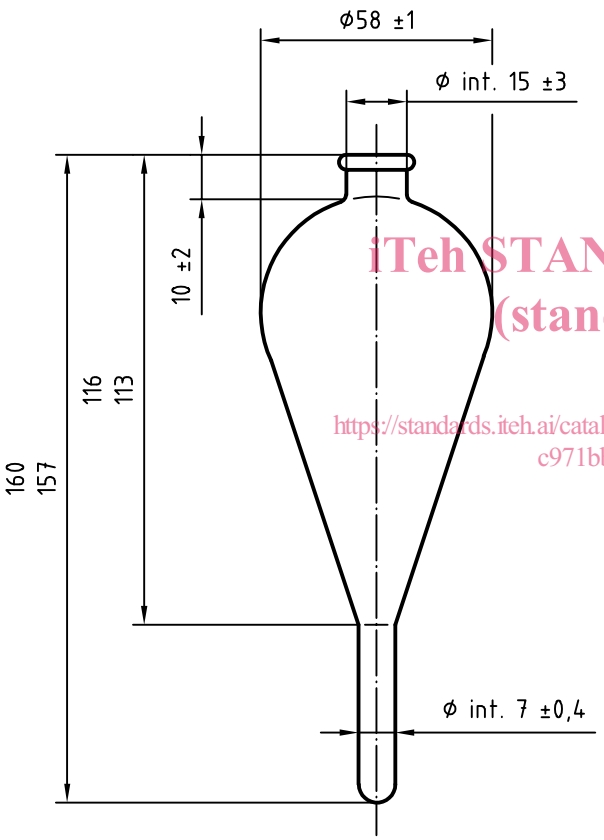

Figure 1 — Pear-shaped sediment tube

Dimensions in millimetres

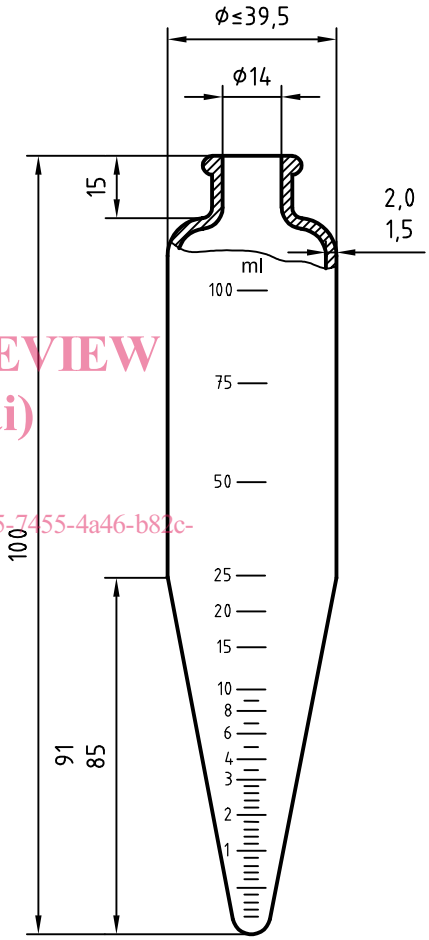

Figure 2 — Cone-shaped sediment tube

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555.

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transportation, sampling or storage.

Samples shall be stored in glass or polyethylene terephthalate (PET) bottles.

7 Preparation of test sample

Warm the fat or oil until it is fully liquid and mix (i.e. shaking the sample by hand) until any sediment from the bottom of the sample is redispersed in the fat or oil to ensure a sufficient homogeneous and representative sample. See ISO 661.

8 Procedure

8.1 Number of determinations

Carry out the test in duplicate.

8.2 Preparation of test portion

Cool the prepared test sample (clause 7), with constant stirring, until the temperature is 20 °C or 10 °C above the melting point, whichever is the higher.

8.3 Determination

Fill the sediment tube (5.1 or 5.2) to the 100 ml graduation mark, as quickly as possible, with the homogeneous fat or oil at the temperature of measurement. Allow it to stand in a vertical position for 96 h at 20 °C or 10 °C above the slip melting point, whichever is the higher. During the period of the test, the tube should remain undisturbed. Read the volume of the "foots" at the bottom of the tube after 96 h, to the nearest 0,1 ml.

9 Expression of results

Report the value of visible foots as a percentage by volume, at T °C, (where T °C is the temperature at which the test was carried out). State which sediment tube was used (5.1 or 5.2).

Calculate the mean of the results of the two tubes and report the results to the nearest

0,1 ml per 100 ml	for results $<$ 1 ml per 100 ml,
0,5 ml per 100 ml	for results from 1 ml to 3 ml per 100 ml,
1,0 ml per 100 ml	for results $>$ 3 ml per 100 ml.

10 Precision

10.1 Interlaboratory tests

Details of international interlaboratory tests on the precision of the method are summarized in annex A. The values derived from this test may not be applicable to concentration ranges and matrices other than those given.

10.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than:

level of foots found (ml per100 ml)	$<$ 1	1 to 3	$>$ 3
repeatability limit, r	0,1	0,3 to 0,5	1,0

10.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than:

level of foots found (ml per 100 ml)	< 1	1 to 3	> 3
reproducibility limit, R	1,0	3,0	5,0

11 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this International Standard;

— all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test results(s) obtained or, if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Interlaboratory tests

A.1 Results of interlaboratory tests

Tables A.1 and A.2 give the precision data for tests carried out in 1996 and 1997/1998.

Table A.1 — Visible foots (1996 study)

	Sample No.					
	1	2	3	4	5	6
No. of participating laboratories	12	13	12	13	13	13
No. of acceptable results	11	10	9	12	12	10
Mean value	1,39	2,49	0,42	3,96	5,72	6,73
Repeatability standard deviation, s_r	0,18	0,08	0,03	0,18	0,14	0,23
Coefficient of variation of repeatability, %	12,64	3,24	6,22	4,52	2,50	3,47
Repeatability limit, r	0,49	0,23	0,07	0,50	0,40	0,65
Reproducibility standard deviation, s_R	1,10	0,48	0,62	1,15	1,05	0,85
Coefficient of variation of reproducibility, %	79,4	19,3	146	29,0	18,4	12,7
Reproducibility limit, R	3,09	1,34	1,73	3,21	2,95	2,39

Table A.2 — Visible foots (1997/98 study)

	Sample No.			
	1	2	3	4
No. of participating laboratories	9	9	9	9
No. of acceptable results	8	9	9	9
Mean value	0,07	2,46	2,41	4,76
Repeatability standard deviation, s_r	0	0,07	0,02	0,26
Coefficient of variation of repeatability, %	0	3,04	0,98	5,53
Repeatability limit, r	0	0,21	0,07	0,74
Reproducibility standard deviation, s_R	0,05	0,90	0,90	1,90
Coefficient of variation of reproducibility, %	66,6	36,5	37,5	40,3
Reproducibility limit, R	0,30	2,51	2,53	5,38