BRITISH STANDARD

BS EN ISO 19219:2003

(renumbers the BS ISO as BS EN ISO 19219:2003)

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

The European Standard EN ISO 19219:2003 has the status of a British Standard

ICS 67.200.10

BSI British Standards

National foreword

This British Standard is the official English language version of EN ISO 19219:2003. It is identical to ISO 19219:2002..

The UK participation in its preparation was entrusted to Technical Committee AW/11, Animal and vegetable fats and oils, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

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Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, the EN ISO foreword page, the ISO title page, pages ii and iii, a blank page, pages 1 to 7, the Annex ZA page, an inside back cover and a back cover.

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14804	21 January 2004	Renumbers BS ISO 19219:2002 as BS EN ISO 19219:2003, which includes an Annex ZA page

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 19219

November 2003

ICS 67.200.10

English version

Animal and vegetable fats and oils - Determination of visible foots in crude fats and oils (ISO 19219:2002)

Corps gras d'origines animale et végétale - Détermination de la teneur en sédiments visibles dans des graisses et huiles brutes (ISO 19219:2002)

Tierische und pflanzliche Fette und Öle - Bestimmung von sichtbarem Bodensatz in rohen Fetten und Ölen (ISO 19219:2002)

This European Standard was approved by CEN on 25 September 2003.

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Ref. No. EN ISO 19219:2003 E

Foreword

The text of ISO 19219:2002 has been prepared by Technical Committee ISO/TC 34 "Agricultural food products" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 19219:2003 by Technical Committee CEN/TC 307 "Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2004, and conflicting national standards shall be withdrawn at the latest by May 2004.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of ISO 19219:2002 has been approved by CEN as EN ISO 19219:2003 without any modifications.

NOTE Normative references to International Standards are listed in Annex ZA (normative).

EN ISO 19219:2003

INTERNATIONAL STANDARD

ISO 19219

First edition
2002-04-01

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

Reference number
ISO 19219:2002(E)

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

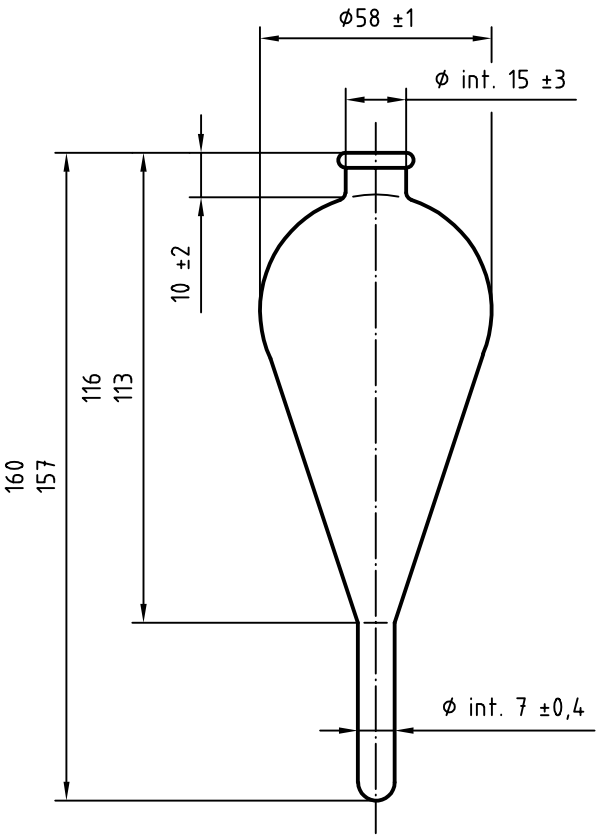

Figure 1 — Pear-shaped sediment tube

Dimensions in millimetres

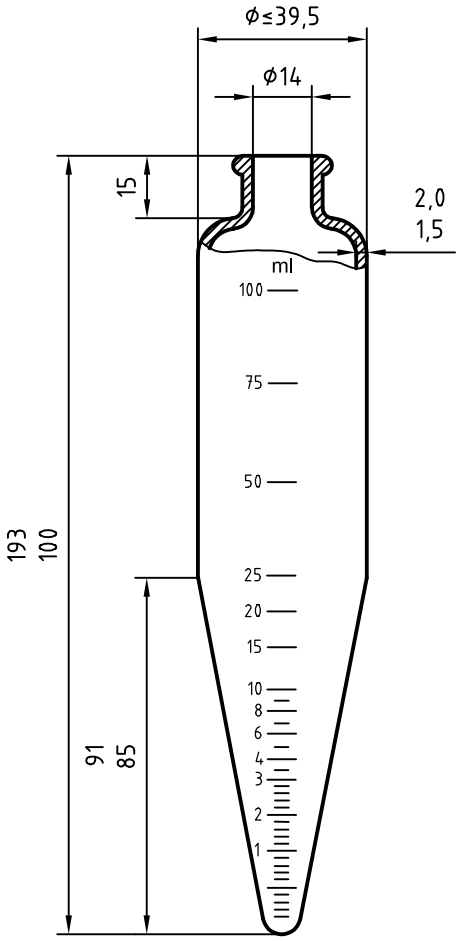

Figure 2 — Cone-shaped sediment tube

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

A.2 Regression analysis for ISO 19219 and ISO 15301 methods

A comparison was made of the results found using the method given in ISO 15301 and the present method. Using the analysis results obtained from both collaborative ring tests, the relationship between the two methods is shown in the following equation (with a correlation coefficient of 0,96):

$$V = 2{,}5C - 0{,}5$$

where

V is the the volume of foots found with the ISO 19219 method (96 h test);

C is the the volume of sediment/foots found with the ISO 15301 method (centrifuge test).

This indicates that the centrifuge method may be considered as a good alternative to the visible foots method, where a rapid control method is required for levels of sediment $>$ 0,5 ml per 100 mg.

Bibliography

[1] ISO 5555, *Animal and vegetable fats and oils — Sampling*

[2] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[3] ISO 5725-2:1994, *Accuracy (trueness and precision) measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[4] ISO 15301:2001, *Animal and vegetable fats and oils — Determination of sediment in crude fats and oils — Centrifuge method*

Annex ZA
(normative)

Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

NOTE Where an International Publication has been modified by common modifications, indicated by (mod.), the relevant EN/HD applies.

Publication	Year	Title	EN	Year
ISO 661	1989	Animal and vegetable fats and oils - Preparation of test sample	EN ISO 661	1995

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