

INTERNATIONAL STANDARD

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Animal and vegetable fats and oils — Flashpoint limit test using Pensky-Martens closed cup flash tester

*Corps gras d'origines animale et végétale — Détermination du point d'éclair
avec la méthode Pensky-Martens en vase clos*



Reference number
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Foreword

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Annex A of this International Standard is for information only.

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Animal and vegetable fats and oils — Flashpoint limit test using Pensky-Martens closed cup flash tester

1 Scope

This International Standard specifies a method to determine whether a sample of oil or fat at a given temperature will flash when a test flame is applied to the sample under specified conditions.

It is applicable to animal, vegetable and marine fats and oils. The fats and oils may or may not contain small amounts of volatile inflammable solvents.

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

ISO 2719:1988, *Petroleum products and lubricants — Determination of flash point — Pensky-Martens closed cup method*.

3 Principle

The sample is heated at a slow constant rate with continual stirring. Once the temperature specified is stabilized, a small flame is directed into the cup. The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample; a halo should be ignored.

4 Apparatus

4.1 Pensky-Martens closed cup flash tester

For details, see ISO 2719.

4.2 Thermometers, having a range from 10 °C to 200 °C.

Alternatively, an IP thermometer 101 C (having a range from 20 °C to 150 °C) can be used.

4.3 Laboratory centrifuge (swing type), of sufficient size to hold stoppered 120 ml centrifuge tubes.

4.4 Centrifuge tubes, of 120 ml capacity, with stoppers.

5 Reagents

Use only reagents of recognized analytical grade, and distilled or demineralized water or water of equivalent purity.

5.1 Sodium sulfate, anhydrous.

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555¹⁾. (See also 8.1.)

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

Samples shall be stored in bottles made of materials which do not allow diffusion of volatile compounds from the sample through the walls of the bottle.

NOTE PET or glass bottles are preferred. Some plastics (e.g. polyethylene or polypropylene) are not suitable for this purpose.

7 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

8 Procedure

Carry out the test in duplicate.

8.1 Preparation of test portion

8.1.1 During preparation of test portion and sampling or sub-sampling for laboratory testing, and any other handling, care should be taken to minimize transfer between containers. It has been found that pouring samples from one container to another can cause evaporation of volatile solvents such as hexane, thus invalidating the result. For related reasons, it is advisable that containers be kept closed.

8.1.2 If the fat is a solid at room temperature, it should be liquefied by warming slowly, in the original container, to a temperature not higher than 5 °C of its melting point. The flashpoint determination should then start at this elevated temperature.

8.1.3 Carefully transfer approximately 90 g of fat or oil from the original container directly into the tube (4.4) and add 5 g of anhydrous sodium sulfate (5.1). Shake the mixture vigorously for 1 min with the stopper secured and allow it to stand for 30 min, if necessary, at an elevated temperature (8.1.2).

8.1.4 Centrifuge the treated oil, as prepared in 8.1.3, at 2 500 r/min for 3 min or until a sufficient amount of clear oil is available to make the flashpoint determination. The maximum time of centrifuging shall be 5 min.

8.2 Determination

8.2.1 Fill the cup with the liquefied oil or fat sample (8.1.4) so that the top of the meniscus is exactly at the filling line marked in the cup. Place the lid on the cup and engage the locating devices.

Insert the thermometer (4.2) and suspend it so that the bottom of the bulb is a minimum of 43 mm and a maximum of 46 mm below the level of the rim of the cup, which corresponds to the level of the lower surface of the portion of the lid inside the rim.

¹⁾ ISO 5555:1991, *Animal and vegetable fats and oils — Sampling*.

8.2.2 Light the test flame and adjust it until it is approximately 4 mm in diameter.

8.2.3 Heat the sample so that the temperature increases not less than 5 °C and not more than 6 °C per minute. During the heating, turn the stirring device at one or two revolutions per second.

8.2.4 At the specified temperature (usually 121 °C), discontinue stirring and apply the test flame by operating the device which controls the shutter and lowers the test flame into the shutter opening.

Lower the test flame within 0,5 s and observe whether this causes a distinct flash in the interior of the cup.

Do not confuse the true flash with the blue halo that sometimes surrounds the test flame.

8.2.5 Do not open the cup more than once during each determination because solvent vapour, although present in too low a concentration to cause a flash, might escape. This would lead to an incorrect result at the measuring temperature.

8.2.6 In order to conserve sample it is permissible to carry out successive flash tests on the same sub-sample to ascertain the temperature range in which the sample will flash. However, any such ranging tests shall not be used to establish or substantiate the reported test result which shall be determined on a fresh sample.

8.2.7 If information is needed about the precise level of the flashpoint, the whole procedure should be repeated at temperatures other than 121 °C. Fresh portions of the test samples should be used for each temperature determination.

9 Precision

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

10 Test report

Report "flash" or "no flash" at the specified temperature and state the method and apparatus used.

If different results are obtained, disregard the "no flash" result.

Annex A

(informative)

Results of interlaboratory test

An interlaboratory test was carried out in 1995, organized by FOSFA International and NOFOTA. Twenty-one laboratories in eight countries participated. The results are summarized in table A.1.

**Table A.1 — Results of international collaborative trial for flashpoint limit
test at 121 °C**

Sample	No. of labs submitting flashpoint results	No. of labs reporting "flash"	No. of labs reporting "no flash"	Labs reporting "flash" %	Labs reporting "no flash" %
1	19 ¹⁾	0	18	0,0	100,0
2	19 ²⁾	12	5	70,6	29,4
3	19 ³⁾	4	13	23,5	76,5
4	19 ⁴⁾	0	18	0,0	100,0
5	19 ⁵⁾	15	3	83,3	16,7
6	19	2	17	10,5	89,5
1) Laboratory 12 stated that oil had leaked from sample bottle and so it was excluded. 2) Laboratories 12 and 21 stated that oil had leaked from sample bottle and so they were excluded. 3) Laboratory 7 submitted "flash" and "no flash" as duplicates and so was not included in the summary. 4) Laboratory 10 submitted "flash" and "no flash" duplicates. 5) Laboratory 21 stated that oil had leaked from sample bottle and so it was excluded.					

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