INTERNATIONAL STANDARD

ISO 6884

Second edition
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Animal and vegetable fats and oils — Determination of ash

Corps gras d'origines animale et végétale — Détermination du taux de cendres

Reference number
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Foreword

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

ISO 6884 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This second edition cancels and replaces the first edition (ISO 6884:1985), of which it constitutes a minor revision.

Animal and vegetable fats and oils — Determination of ash

1 Scope

This International Standard specifies a method for the determination of ash, applicable to all animal and vegetable fats and oils, including acid oils.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
ash
⟨animal and vegetable fats and oils⟩ inorganic residue left after incineration under the conditions specified in this International Standard

NOTE The ash yield is expressed as a percentage mass fraction of the dry product.

4 Principle

The sample is combusted under mild heating and the residue is incinerated at 550 °C to 600 °C until free from carbon particles. The residue obtained is then weighed.

5 Reagents

WARNING — Comply with any local regulations which specify the handling of hazardous substances. Follow technical, organizational, and personal safety measures.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

5.1 Hydrogen peroxide solution, φ = 10 % volume fraction.

5.2 Ammonium carbonate.

6 Apparatus and materials

Usual laboratory apparatus and, in particular the following.

6.1 **Crucible** (low form) of capacity 50 ml, preferably of silica or platinum.

6.2 **Hotplate** or **flame burner**.

6.3 **Furnace**, capable of being maintained between 500 °C and 600 °C.

6.4 **Filter paper**, ashless.

6.5 **Water-bath, boiling**.

6.6 **Desiccator**.

6.7 **Analytical balance**, capable of being read to the nearest 1 mg.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555 [1].

8 Preparation of the test sample

Prepare the test sample from the laboratory sample in accordance with ISO 661.

9 Procedure

9.1 Crude and refined fats and oils

9.1.1 First heat the crucible (6.1) in the furnace (6.3) maintained at 550 °C to 600 °C, then cool in a desiccator (6.6) and weigh to the nearest 1 mg. Weigh, to the nearest 10 mg, about 10 g of the test sample (Clause 8) into the crucible. Heat carefully on the hotplate or over the flame burner (6.2) in a fume cupboard until the sample ignites.

For low ash yields, a larger test portion may be taken by adding successive portions of 10 g after the initial ashing; for high ash yields, a smaller test portion may be used. With an increased quantity of oil, a wick of ashless filter paper may be inserted and lit whilst heating the oil on a hotplate.

The initial heating may be carried out in the mouth of the furnace, if the furnace is installed in a fume cupboard.

9.1.2 When the burning ceases, transfer the crucible to the furnace (6.3) maintained at 550 °C to 600 °C. Maintain the crucible at this temperature for 4 h or for a shorter time if a carbon-free residue is obtained more rapidly. A carbon-free residue is shown by the appearance of the ash, which becomes red-brown (due to the presence of iron) or white, with no black particles remaining in the residue.

9.1.3 If the residue is not carbon-free after 4 h, add a few drops of hydrogen peroxide (5.1), dry on a boiling water-bath (6.5), and reheat in the furnace (6.3) to assist in the removal of carbon.

It may be necessary to repeat this treatment.

9.1.4 When the residue is carbon-free, allow to cool in a desiccator (6.6), and weigh to the nearest 1 mg.

9.2 Acid oils

9.2.1 Follow the procedure in 9.1.1, but in the first step heat the crucible in the furnace maintained at 500 °C to 550 °C.

9.2.2 When the burning ceases, allow the crucible to cool and take up the residue in water. Filter through an ashless filter paper (6.4), and retain the filtrate in a beaker.

9.2.3 Place the filter paper with the residue in the crucible, then place the crucible in an oven at (103 ± 2) °C until the paper has dried. Transfer the crucible to the hotplate or flame burner, and heat carefully as described in 9.1.1 until burning ceases. Then heat in the furnace (6.3) at a temperature of 500 °C to 550 °C until the carbon particles disappear or until there is no change in appearance of the residue. If the residue is not carbon-free, proceed as described in 9.1.3. Allow the crucible to cool.

9.2.4 Transfer the filtrate retained in 9.2.2 quantitatively to the crucible (9.2.3). Evaporate to dryness on a boiling water-bath (6.5).

Add 0,5 g to 2 g of ammonium carbonate (5.2) in order to recarbonate the ash, then heat the residue in the furnace (6.3) at a temperature of 500 °C to 550 °C. Allow the crucible to cool in a desiccator (6.6) and weigh to the nearest 1 mg.

10 Expression of results

The ash yield, w, expressed as a percentage mass fraction, is given by the equation

$$w = \frac{(m_2 - m_1)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion on the dry basis;

m_1 is the mass, in grams, of the empty crucible;

m_2 is the mass, in grams, of the crucible and ash.

11 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

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

d) the result(s) obtained;

e) if the repeatability has been checked, the final quoted result obtained;

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

For further information on the test report, refer to ISO 17025:2005 [2], 5.10.

Bibliography

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

[2] ISO 17025:2005, *General requirements for the competence of testing and calibration laboratories*