INTERNATIONAL STANDARD

ISO 936

Second edition
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Meat and meat products — Determination of total ash

Viande et produits à base de viande — Dosage des cendres totales

Foreword

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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 936 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 6, *Meat and meat products*.

This second edition cancels and replaces the first edition (ISO 936:1978), which has been technically revised.

Annex A of this International Standard is for information only.

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Meat and meat products — Determination of total ash

1 Scope

This International Standard specifies a method for the determination of the total ash from all kinds of meat and meat products, including poultry.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

3 Definition

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

3.1
total ash from meat and meat products
mass of the residue obtained after incineration at a temperature of (550 ± 25) °C under the operating conditions specified in this International Standard, divided by the mass of the test portion

NOTE The mass fraction of ash is usually expressed as a percentage.

4 Principle

A test portion is dried, carbonized and then incinerated at (550 ± 25) °C. After cooling, the mass of the residue is determined.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Water, complying with at least grade 3 in accordance with ISO 3696.

5.2 Hydrogen peroxide, 30 %.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Mechanical or electrical equipment capable of homogenizing the laboratory sample.

This includes a high-speed rotational cutter, or a mincer fitted with a plate with apertures not exceeding 4,0 mm in diameter.

6.2 Dish, flat-bottomed, made of porcelain, quartz or metal (e.g. nickel, platinum, stainless steel) or of other material unaffected by the conditions of the test, with a diameter of at least 60 mm, and inclined walls of height at least 25 mm.

6.3 Muffle furnace, electrically heated and equipped with a programmable time-temperature controller, and capable of being maintained at (550 ± 25) °C.

6.4 Desiccator, containing an efficient desiccant.

6.5 Analytical balance, capable of weighing to the nearest 0,1 mg.

6.6 Drying oven, capable of being maintained at (103 ± 2) °C (if the muffle furnace has no time-temperature controller).

6.7 Electric hot-plate or a gas flame (if the muffle furnace has no time-temperature controller).

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 3100-1 [1].

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

Start from a representative sample of at least 200 g. Store the sample in such a way that deterioration and change in composition are prevented.

8 Preparation of test sample

Homogenize the laboratory sample with the appropriate equipment (6.1). Take care that the temperature of the sample material does not rise above 25 °C. If a mincer is used, pass the sample at least twice through the equipment.

Fill a suitable airtight container with the prepared sample. Close the container and store in such a way that deterioration and change in composition are prevented. Analyse the sample as soon as practicable, but always within 24 h after homogenization.

9 Procedure

NOTE If it is required to check whether the repeatability limit (see 11.2) is met, carry out two single determinations in accordance with 9.1 to 9.3.

9.1 Test portion

Heat the dish (6.2) for 20 min in the muffle furnace (6.3) set at 550 °C.

Allow the dish to cool in the desiccator (6.4) to room temperature and weigh (m_0) on the analytical balance (6.5) to the nearest 0,1 mg.

Transfer 1,5 g to 2 g of the prepared test sample (see clause 8) to the dish. Spread it out evenly and without delay weigh (m_1) the dish again to the nearest 0,1 mg.

NOTE If deemed appropriate by the analyst, having regard to the nature of the laboratory sample, up to 5 g of the prepared test sample may be taken. This should be noted in the test report.

If the muffle furnace is provided with a time-temperature controller, proceed in accordance with 9.2. If not, proceed in accordance with 9.3.

9.2 Determination applying muffle furnace with time-temperature controller

Place the dish with its contents in the cool muffle furnace (6.3) and gradually raise the temperature of the furnace over 5 h to 6 h to (550 ± 25) °C. Continue the ashing at (550 ± 25) °C until the ash has a grey-white appearance.

Remove the dish from the muffle furnace and allow to cool in the desiccator (6.4) to room temperature.

CAUTION - Avoid loss of ash when transferring the dish with the ash from the furnace to the desiccator and from the desiccator to the analytical balance.

Inspect the ash.

If the ash is still black, treat it with a few drops of hydrogen peroxide (5.2) or water (5.1) and repeat the procedure described in this subclause.

If the ash has a grey-white appearance, weigh on the analytical balance (6.5), to the nearest 0,1 mg, the dish with its contents (m_2). Proceed in accordance with clause 10.

9.3 Determination applying muffle furnace without time-temperature controller

Place the dish with its contents for 1 h in the drying oven (6.6) set at 103 °C.

Remove the dish from the oven and place it on an electric hot-plate or over a gas flame (6.7). Heat progressively until the substance carbonizes with the evolution of smoke. Carefully continue carbonization until smoke evolution ceases. The sample material shall neither ignite nor burn with a flame.

Transfer the dish to the cool muffle furnace (6.3) and raise the temperature to (550 ± 25) °C.

After 4 h, remove the dish with its contents from the muffle furnace and allow to cool in the desiccator (6.4) to room temperature.

CAUTION - Avoid loss of ash when transferring the dish with the ash from the furnace to the desiccator and from the desiccator to the analytical balance.

Inspect the ash.

If the ash is still black, treat it with a few drops of hydrogen peroxide (5.2) or water (5.1) and repeat the procedure described in this subclause.

If the ash has a grey-white appearance, weigh on the analytical balance (6.5), to the nearest 0,1 mg, the dish with its contents (m_2).

10 Calculation

Calculate the mass fraction of ash of the test sample using the equation:

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

where

w_a is the mass fraction of ash, as a percentage, of the test sample;

m_0 is the mass, in grams, of the empty dish;

m_1 is the mass, in grams, of the dish with the test portion;

m_2 is the mass, in grams, of the dish with the ash.

Report the result rounded to the nearest 0,01 %.

11 Precision

11.1 Interlaboratory test

The precision of the method was established by an interlaboratory test carried out in accordance with ISO 5725 [2][1)].

The results of the interlaboratory test have been published (see reference [6]). The values derived from this test may not be applicable to concentration ranges and matrices other than those given.

11.2 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 exceed the repeatability limit r given by the equation:

$$r = 0{,}099\ 0\ \% + 0{,}0093\ 3\ \overline{w}$$

where

r is the repeatability limit, as a percentage;

$\overline{w}$ is the mean of both results, as a percentage.

1) ISO 5725:1986 (now withdrawn) was used to obtain the precision data.

11.3 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 exceed the reproducibility limit R given by the equation:

$$R = 0{,}138\ \% + 0{,}004\ 6\ \overline{w}$$

where

R is the reproducibility limit, as a percentage;

$\overline{w}$ is the mean of both results, as a percentage.

12 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 result(s) obtained; or

— if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Bibliography

[1] ISO 3100-1:1991, *Meat and meat products — Sampling and preparation of test samples — Part 1: Sampling.*

[2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

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

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

[5] Method No 23 (1991*). Moisture and Ash: Gravimetric Determination in Meat and Meat Products.* Nordic Committee on Food Analysis (NMKL), Espoo, Finland (in English and Scandinavian). (UDC 637.5).

[6] Kolar, K. Gravimetric Determination of Moisture and Ash in Meat and Meat Products: NMKL Interlaboratory Study**.** *J. AOAC,* **75**, 1992, pp. 1016-1022.