

# INTERNATIONAL STANDARD

# ISO 5984

Second edition  
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## Animal feeding stuffs — Determination of crude ash

*Aliments des animaux — Détermination des cendres brutes*



Reference number  
ISO 5984:2002(E)

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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 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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5984 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 10, *Animal feeding stuffs*.

This second edition cancels and replaces the first edition (ISO 5984:1978), of which it constitutes a minor revision.

Annex A of this International Standard is for information only.

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# Animal feeding stuffs — Determination of crude ash

## 1 Scope

This International Standard specifies a method for the determination of crude ash of animal feeding stuffs.

## 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 6498, *Animal feeding stuffs — Preparation of test samples*

## 3 Term and definition

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

### 3.1

#### **crude ash**

residue obtained after incineration at 550 °C under the conditions specified in this International Standard

NOTE It is expressed as a mass fraction of the sample in percent.

## 4 Principle

The organic matter in a test portion is decomposed by incineration, then the ash obtained is weighed.

## 5 Apparatus

Usual laboratory apparatus and, in particular, the following.

**5.1 Analytical balance**, capable of weighing to the nearest 0,001 g.

**5.2 Muffle furnace**, electrically heated, thermostatically controlled, and provided with a pyrometer.

The furnace, when set at 550 °C, shall be capable of being controlled in such a way that the temperature in the places where the incineration dishes will be placed will not differ by more than 20 °C from this set temperature.

**5.3 Drying oven**, capable of being controlled at  $(103 \pm 2)$  °C.

**5.4 Hot-plate or gas burner**

**5.5 Incineration dish**, of platinum or platinum-gold alloy (e.g. 10 % Pt, 90 % Au) or of other material unaffected by the conditions of the test, preferably rectangular with a surface area of about 20 cm<sup>2</sup> and a height of about 2,5 cm.

For samples that are inclined to swell on carbonization, use dishes with a surface area of about 30 cm<sup>2</sup> and a height of about 3 cm.

**5.6 Desiccator**, provided with an effective desiccant.

## 6 Sampling

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

Store the sample in such a way that deterioration and change in composition are prevented.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 6497.

## 7 Procedure

### 7.1 Preparation of test sample

Prepare the test sample in accordance with ISO 6498.

### 7.2 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the test sample (7.1) into the incineration dish (5.5), previously heated for at least 30 min in the muffle furnace (5.2) set at 550 °C, cooled in the desiccator (5.6) and weighed to the nearest 0,001 g.

### 7.3 Determination

Place the incineration dish containing the test portion (7.2) on a hot plate or over a gas burner (5.4) and heat progressively until the test portion has carbonized. Transfer the dish to the muffle furnace (5.2), previously heated to 550 °C, and leave it for 3 h. Inspect visually whether the ash is free from carbonaceous particles. If it is not, replace the dish in the furnace and heat for another 1 h. If carbonaceous particles are still visible, or if there is doubt as to whether they are present, allow the ash to cool, moisten with distilled water, evaporate carefully to dryness in the oven (5.3), set at 103 °C. Then replace the dish in the furnace and heat for another 1 h. Allow the dish to cool in the desiccator to room temperature then weigh rapidly to the nearest 0,001 g.

**NOTE** The crude ash obtained by the above procedure may be used subsequently for the determination of ash insoluble in hydrochloric acid (see ISO 5985).

Carry out two determinations on test portions from the same test sample.

## 8 Expression of results

The crude ash,  $w$ , expressed as a mass fraction in percent of the test sample, is equal to:

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

where

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

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

$m_2$  is the mass, in grams, of the dish and the crude ash.

Take as the result the arithmetic mean of the two determinations, provided that the requirement for repeatability (see 9.2) is satisfied. Report the result to the nearest 0,1 % (mass fraction).

## 9 Precision

### 9.1 Interlaboratory tests

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

### 9.2 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, will in not more than 5 % of cases exceed the repeatability limit ( $r$ ) given in Table 1.

**Table 1 — Repeatability limit ( $r$ ) and reproducibility limit ( $R$ )**

Values in grams per kilogram

Sample	Crude ash	$r$	$R$
Fishmeal	179,8	2,7	4,4
Tapioca	59,1	2,4	3,6
Meat meal	175,6	2,4	5,6
Piglet feed	50,2	2,1	3,3
Broiler feed	42,7	0,9	2,2
Barley	20,0	1,0	1,9
Molasses	119,9	3,6	9,1
Palm kernel expellers	35,8	0,7	1,6

### 9.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of cases exceed the reproducibility limit ( $R$ ) give in Table 1.

## 10 Test report

The test report shall specify:

- a) all information required 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) all operating details not specified in this International Standard, or regarded as optional, together with details of any incident which may have influenced the result(s);
- e) the test result(s) obtained;
- f) if the repeatability has been checked, the final quoted results obtained.

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## Annex A (informative)

### Results of interlaboratory tests

The precision of the method was established by interlaboratory tests carried out in accordance with ISO 5725-1 and ISO 5725-2, in which the Dixon test is replaced by the Grubbs test in the case of outliers. In the tests, 40 to 52 laboratories participated and samples were investigated of fishmeal, tapioca, meat meal, piglet feed, broiler feed, barley, molasses, palm kernel expellers. The results of the interlaboratory tests are given in Table A.1.

**Table A.1 — Statistical results of interlaboratory tests**

Parameter	Sample <sup>a</sup>							
	1	2	3	4	5	6	7	8
Number of laboratories	52	48	47	50	48	48	40	49
Number of accepted results	50	47	43	49	44	45	39	46
Mean crude ash, g/kg	179,8	59,1	175,6	50,2	42,7	20,0	119,9	35,8
Repeatability standard deviation ( $s_r$ ), g/kg	1,0	0,9	0,9	0,8	0,3	0,4	1,3	0,2
Repeatability coefficient of variation, %	1,5	4,1	1,4	4,2	2,1	5,0	3,0	2,0
Repeatability limit ( $r$ ), g/kg	2,7	2,4	2,4	2,1	0,9	1,0	3,6	0,7
Reproducibility standard deviation ( $s_R$ ), g/kg	1,4	1,1	1,9	1,1	0,7	0,6	3,1	0,5
Reproducibility coefficient of variation, %	2,5	6,0	3,2	6,6	5,1	9,6	7,6	4,4
Reproducibility limit ( $R$ ), g/kg	4,4	3,6	5,6	3,3	2,2	1,9	9,1	1,6
<sup>a</sup> 1: fishmeal; 2: tapioca; 3: meat meal; 4: piglet feed; 5: broiler feed; 6: barley; 7: molasses; 8: palm kernel expellers.								

## Bibliography

- [1] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*
- [2] ISO 5725-2, *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*
- [3] ISO 5985, *Animal feeding stuffs — Determination of ash insoluble in hydrochloric acid*
- [4] ISO 6497, *Animal feeding stuffs — Sampling*



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