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Animal feeding stuffs — Determination of crude ash

Aliments des animaux — Dosage des cendres brutes

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 5984:2002), which has been technically revised. It also incorporates the Technical Corrigendum ISO 5984:2002/Cor 1:2005.

The main changes are as follows:

— an alternative way of incineration (directly in the cold muffle furnace) has been added;

— the possibility to use a thermocouple sensor for the muffle furnace has been added;

— the obligation to perform two determinations has been removed;

— the repeatability and reproducibility ranges have been modified.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Animal feeding stuffs — Determination of crude ash

1 Scope

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 6498, *Animal feeding stuffs — Guidelines for sample preparation*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
crude ash
residue obtained after incineration at (550 ± 25) °C under the conditions specified in this document

Note 1 to entry: It is expressed as a mass fraction of the sample in per cent.

4 Principle

A test portion is carbonized and then incinerated at (550 ± 25) °C. After cooling, 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, provided with a pyrometer or a thermocouple sensor.

The furnace is set at 550 °C. The furnace temperature shall be of (550 ± 25) °C where the incineration dishes will be placed.

It is recommended to use a furnace equipped with a programmable time-temperature controller.

5.3 Drying oven, capable of being controlled at (103 ± 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, such as porcelain or quartz crucibles, preferably with a surface area of about 20 cm^2 and a height of about 2,5 cm.

For samples with a low density and samples that are inclined to swell on carbonization (e.g. molasses), use dishes with a surface area of minimum about 30 cm^2 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 document. A recommended sampling method is given in ISO 6497[4].

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 (see 7.1) into the incineration dish (5.5), previously heated for at least 30 min in the muffle furnace (5.2) set at (550 ± 25) °C, cooled in the desiccator (5.6) and weighed to the nearest 0,001 g. Alternatively, the incineration dish should be washed at the end of the previous test, heated for at least 30 min in the muffle furnace set at (550 ± 25) °C, and then stored in the oven set at least 100 °C.

7.3 Determination

Place the incineration dish containing the test portion (see 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 ± 25) °C, and leave it for at least 3 h. Alternatively, for matrices that do not generate projections, place the incineration dish containing the test portion (see 7.2) directly in the cold muffle furnace and use a gradual heating programme to allow test portion carbonization before temperature reach (550 ± 25) °C.

It is recommended to use a furnace equipped with a programmable time-temperature controller.

EXAMPLE The following temperature gradient can be used:

— add the incinerations dishes in the furnace at room temperature;

— gradually raise the temperature of the furnace up to 250 °C during 2 h;

— keep the temperature at 250 °C for 1 h;

— gradually raise the temperature up to 550 °C during 1 h;

— keep the temperature at 550 °C until the ash has a grey-white appearance (e.g. after 8 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 ± 2) °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.

The crude ash obtained by the above procedure may be used subsequently for the determination of ash insoluble in hydrochloric acid (see ISO 5985[3]).

8 Expression of results

The crude ash, *w*, expressed as a mass fraction in per cent of the test sample, is given as shown in Formula (1):

$$w = \frac{m_2 - m_0}{m_1 - m_0} \times 100 \quad (1)$$

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.

Report the result to the nearest 0,1 % (mass fraction).

If the analyses have been done in replicate, take as the result the arithmetic mean, provided that the requirement for repeatability (see 9.2) is satisfied.

9 Precision

9.1 Interlaboratory tests

Details of interlaboratory tests on the precision of the method are given in Annex A. The values are from 15 g/kg to 200 g/kg. However, it is commonly agreed that the method also applies for values between 0 g/kg and 1 000 g/kg. Therefore, five ranges have been chosen to define the repeatability and three ranges for define the reproducibility (see Table 1).

The method is also applicable to other matrices besides those tested in the interlaboratory tests.

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

Sample	Crude ash g/kg	*r* %	*R* %
Fishmeal	179,8	2,7	4,1
Tapioca	59,1	2,4	3,1
Meat meal	175,5	2,5	5,4
Piglet feed	50,2	2,1	3,0
Broiler feed	42,7	0,9	2,1
Barley	19,6	1,0	1,8
Molasses	119,9	3,6	8,8
Palm kernel expellers	35,8	0,7	1,5

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*) (see Figure 1):

— 2 g/kg (absolute difference) for crude ash values under 40 g/kg;

— 5 % (relative difference) of the mean result for crude ash values between 40 g/kg and 100 g/kg;

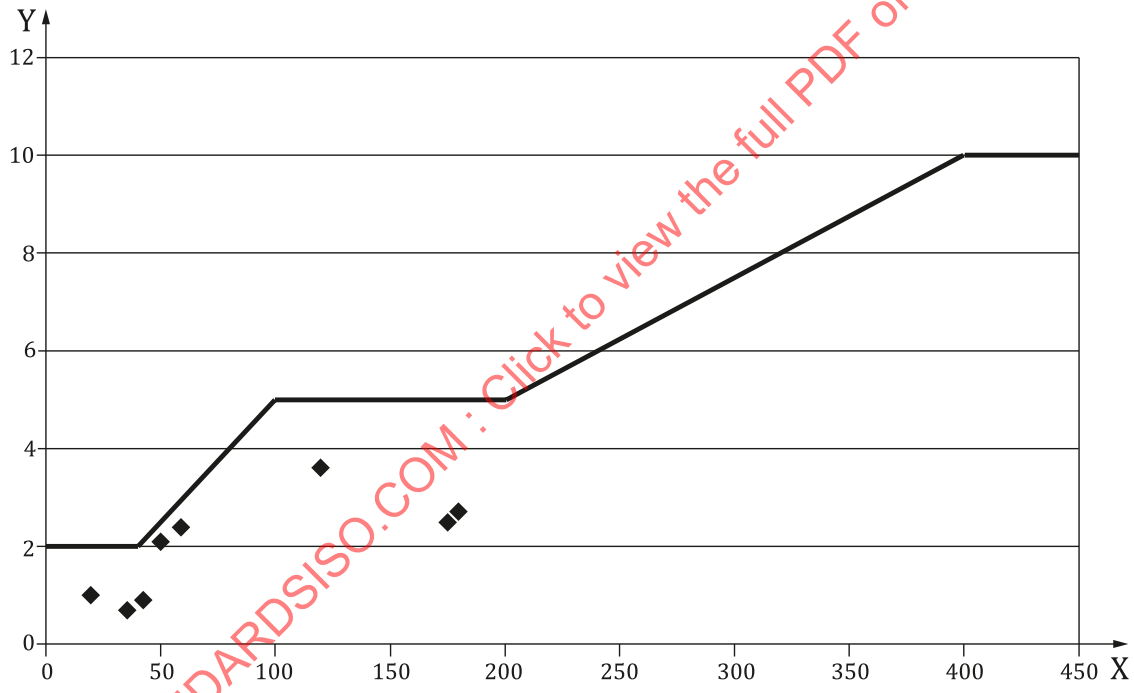
— 5 g/kg (absolute difference) for crude ash values between 100 g/kg and 200 g/kg.

In NF V18-101[5], the repeatability ranges for mean values of crude ash higher than 200 g/kg are defined as:

— 2,5 % (relative difference) of the mean result for crude ash values between 200 g/kg and 400 g/kg

— 10 g/kg (absolute difference) for crude ash values superior to 400 g/kg

These ranges were not tested during the collaborative study for this document but are still valid and can be useful for routine samples.



Key

X crude ash, in g/kg

Y *r*, in g/kg

◆ repeatability limit

— fixed repeatability limit

Figure 1 — Graphical representation of repeatability limit

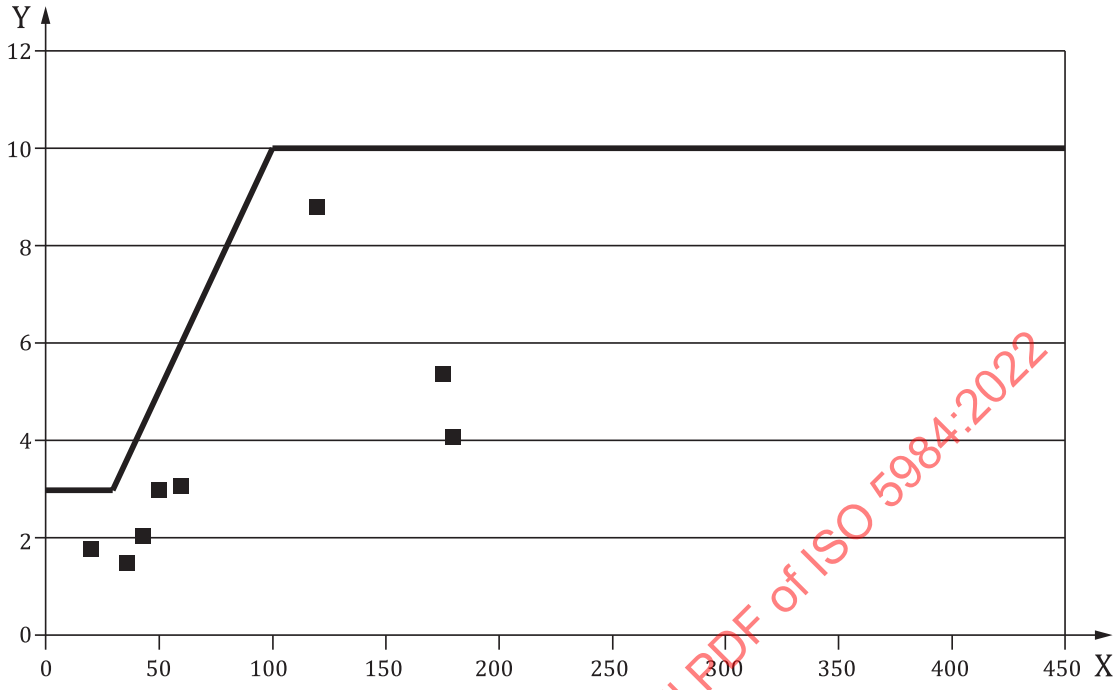
9.3 Reproducibility

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*) (see Figure 2):

— 3 g/kg (absolute difference) for crude ash values under 30 g/kg;

— 10 % (relative difference) of the mean result for crude ash values between 30 g/kg and 100 g/kg;

— 10 g/kg (absolute difference) for crude ash values superior to 100 g/kg.



Key

X crude ash, in g/kg

Y *R*, in g/kg

fixed reproducibility limit

reproducibility limit

Figure 2 — Graphical representation of reproducibility limit

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 document, i.e. ISO 5984;

d) all operating details not specified in this document, or regarded as optional, together with details of any incident which can have influenced the result(s);

e) the test result(s) obtained;

f) if the repeatability has been checked, the final quoted results obtained;

g) the date of the test.