BRITISH STANDARD

BS ISO 5985:2002 +A1:2015

Incorporating Corrigendum No. 1

Animal feeding stuffs — Determination of ash insoluble in hydrochloric acid

ICS 65.120

BSI British Standards

National foreword

This British Standard is the UK implementation of ISO 5985:2002+A1:2015, incorporating corrigendum March 2006. It supersedes BS ISO 5985:2002 which is withdrawn.

The start and finish of text introduced or altered by amendment is indicated in the text by tags. Tags indicating changes to ISO text carry the number of the ISO amendment. For example, text altered by ISO amendment 1 is indicated by A1 A1.

The UK participation in its preparation was entrusted to Technical Committee AW/10, Animal feeding stuffs.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Amd. No.	Date	Comments
15702 Corrigendum No. 1	March 2006	Table 1 and Table A.1 replaced
	31 December 2015	Implementation of ISO amendment 1:2015

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INTERNATIONAL STANDARD

ISO 5985

Second edition
2002-11-15

Animal feeding stuffs — Determination of ash insoluble in hydrochloric acid

Aliments des animaux — Détermination des cendres insolubles dans l'acide chlorhydrique

Reference number
ISO 5985:2002(E)

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 identiying any or all such patent rights.

ISO 5985 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 5985:1978), 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 ash insoluble in hydrochloric acid

1 Scope

This International Standard specifies two procedures for animal feeding stuffs for the determination of the ash which is insoluble in hydrochloric acid.

The applicable procedure depends on the nature of the sample.

a) Procedure A is applicable to simple organic animal feeding stuffs and to compound feeding stuffs (except those mentioned under procedure B).

b) Procedure B is applicable to minerals, mineral mixtures and compound feeding stuffs of which the ash insoluble in hydrochloric acid exceeds 1 % (mass fraction), as determined by procedure A.

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
ash insoluble in hydrochloric acid
that part of the ash which is insoluble in dilute hydrochloric acid under the conditions specified in this International Standard

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

4 Principle

4.1 Procedure A

The organic matter in a test portion is decomposed by incineration.

The ash obtained is treated with hydrochloric acid. The mixture is filtered, followed by drying, incineration and weighing of the residue.

4.2 Procedure B

A test portion is treated with hydrochloric acid. The mixture is filtered, followed by drying and incineration.

The ash is treated as in 4.1.

5 Reagents

Use only reagents of analytical quality, and distilled or demineralized water or water of at least equivalent purity.

5.1 Dilute hydrochloric acid, 3 mol/l.

5.2 Trichloroacetic acid solution, 200 g/l.

5.3 Trichloroacetic acid solution, 10 g/l.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

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

6.2 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.

6.3 Drying oven, capable of being controlled at (103 ± 2) °C.

6.4 Hot-plate or **gas burner**

6.5 Boiling water bath

6.6 Incineration dishes, 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^2 and a height of about 2,5 cm.

For samples which are inclined to swell on carbonization, use dishes with a surface area of about 30 cm^2 and a height of about 3 cm.

6.7 Desiccator, provided with an effective desiccant.

〈A1〉 6.8 Ash-free filter paper.

The percentage of ash cannot exceed 0,01 % of the filter paper. 〈A1〉

7 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.

8 Procedures

8.1 Preparation of test sample

Prepare the test sample in acordance with ISO 6498.

8.2 Procedure A

8.2.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the test sample (8.1) into an incineration dish (6.6).

8.2.2 Determination

8.2.2.1 Place the incineration dish containing the test portion (8.2.1) on a hot plate or over a gas burner (6.4) and heat progressively until the test portion has carbonized. Transfer the dish to the muffle furnace (6.2), previously set at 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 (6.3), set at 103 °C. Then replace the dish in the furnace and heat for another 1 h. Allow the dish to cool to room temperature in the desiccator (6.7).

NOTE The ash obtained at this point corresponds to that obtained by the procedure specified in ISO 5984.

8.2.2.2 Transfer the ash with 75 ml of the dilute hydrochloric acid (5.1) to a 250 ml to 400 ml beaker. Heat carefully to boiling on the hot plate or gas burner (6.4) and boil for 15 min. [A1] Filter the hot solution through an ash-free filter paper (6.8) and wash the filter paper and residue with hot water until the washings are free from acid. [A1] Transfer the filter paper with the residue to an incineration dish (6.6), previously heated for at least 30 min in the muffle furnace (6.2) set at 550 °C, cooled in the desiccator (6.7) and weighed to the nearest 0,001 g. Dry the dish and its contents for 2 h in the oven (6.3), set at 103 °C, then ignite for 30 min in the muffle furnace (6.2), set at 550 °C. Cool the dish to room temperature in the desiccator (6.7) then weigh rapidly to the nearest 0,001 g.

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

8.3 Procedure B

8.3.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the test sample (8.1) into a 250 ml to 400 ml beaker.

8.3.2 Determination

8.3.2.1 Add successively to the beaker containing the test portion (8.3.1), 25 ml of water and 25 ml of the dilute hydrochloric acid (5.1). Mix and allow to stand until foaming has ceased. Add 50 ml of the hydrochloric acid and wait again, if necessary, until foaming has practically ceased. Heat the beaker over the boiling water bath (6.5) for 30 min or longer until all starch present has been completely hydrolysed.

Filter the hot solution through an ash-free filter paper and wash the filter paper and residue with 50 ml of hot water.

8.3.2.2 If the solution is difficult to filter, repeat the determination using a new test portion but adding 50 ml of the trichloroacetic acid solution (5.2) instead of 50 ml of the hydrochloric acid, and wash the filter paper and residue with hot trichloroacetic acid solution (5.3) before washing with hot water.

8.3.2.3 Transfer the filter paper with the residue to an incineration dish (6.6). Dry for 2 h in the oven (6.3), set at 103 °C, then ignite in the muffle furnace (6.2) set at 550 °C for 3 h. Allow the dish to cool to room temperature in the desiccator (6.7).

8.3.2.4 Continue as described in 8.2.2.2.

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

9 Expression of results

The ash insoluble in hydrochloric acid, w, expressed as a mass fraction of the test sample in percent, 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 (8.2.2.2);

m_1 is the mass, in grams, of the dish containing the test portion (8.2.1 or 8.3.1);

m_2 is the mass, in grams, of the dish and the ash insoluble in hydrochloric acid.

Take as the result the arithmetic mean of the two determinations, provided that the requirement for repeatability (see 10.2) is satisfied. Report the result to the nearest 0,1 % (mass fraction).

10 Precision

10.1 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.

10.2 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.

〈A1〉 Table 1 — Repeatability limit (r) and reproducibility limit (R)

Values in grams per 100g

Sample	Ash insoluble in HCl, g/100g	r g/100g	R g/100g
Fishmeal	0,82	0,08	0,23
Tapioca	3,46	0,28	0,48
Meat meal	0,86	0,08	0,19
Piglet feed	0,26	0,03	0,10
Broiler feed	0,17	0,03	0,09
Barley	0,26	0,05	0,16
Palm kernel expellers	0,60	0,08	0,11

〈A1〉

10.3 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) given in Table 1.

11 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 (procedure A or B), 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.

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 case of outliers. In the tests, 20 to 30 laboratories participated and samples were investigated of fishmeal, tapioca, meat meal, piglet feed, broiler feed, barley, palm kernel expellers. The results of the interlaboratory tests are given in Table A.1.

[A1] NOTE ISO 5985:2002 contains incorrect figures due to miscalculation in the reproduction of the performance characteristics. The following Table A.1 contains the correct numbers.

Table A.1 — Statistical results of interlaboratory tests

Parameter	Sample[a]						
	1	2	3	4	5	6	7
Mean content of ash insoluble in HCl, g/100g	0,82	3,46	0,86	0,26	0,17	0,26	0,60
Repeatability standard deviation (s_r), g/100g	0,03	0,10	0,03	0,01	0,01	0,02	0,03
Repeatability coefficient of variation, %	3,4	2,8	3,1	4,2	6,4	6,5	4,8
Repeatability limit (r), g/100g	0,08	0,28	0,08	0,03	0,03	0,05	0,08
Reproducibility standard deviation (s_R), g/100g	0,08	0,17	0,07	0,03	0,03	0,06	0,04
Reproducibility coefficient of variation, %	9,8	4,9	7,7	13,6	19,7	22,5	6,6
Reproducibility limit (R), g/100g	0,23	0,48	0,19	0,10	0,09	0,16	0,11

a 1: fishmeal
2: tapioca
3: meat meal
4: piglet feed
5: broiler feed
6: barley
7: palm kernel expellers

[A1]

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 5984, *Animal feeding stuffs — Determination of crude ash*

[4] ISO 6497, *Animal feeding stuffs — Sampling*

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