# INTERNATIONAL STANDARD

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION®MEXCHAPODHAR OPFAHM3ALUNR DO CTAHDAPTM3ALUN®ORGANISATION INTERNATIONALE DE NORMALISATION

## Animal feeding stuffs – Determination of ash insoluble in hydrochloric acid

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

iTeh STANDARD PREVIEW First edition – 1978-11-15

## (standards.iteh.ai) ISO 5985:1978

https://standards.iteh.ai/catalog/standards/sist/c521c2b6-a747-437e-b853-02c2f8873a83/iso-5985-1978

UDC 636.085/.087 : 543.868

Ref. No. ISO 5985-1978 (E)

Descriptors : animal nutrition, animal feeding stuffs, chemical analysis, determination of content, ash, insoluble matter, hydrochloric acid.

5985

#### FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5985 was developed by Technical Committee VIEW ISO/TC 34, Agricultural food products, and was circulated to the member bodies in May 1977.

It has been approved by the member bodies of the following countries 978

	https://standards.iteh.ai/catalog/standards/sist/c521c2b6-a747-437e-b853-	
Australia	Iran	02c2f8 South3Africa 8Bep of
Austria	Israel	Spain
Canada	Kenya	Thailand
Chile	Mexico	Turkey
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.S.R.
Ethiopia	Peru	Venezuela
France	Poland	Yugoslavia
Hungary	Portugal	-
India	Romania	

No member body expressed disapproval of the document.

## Animal feeding stuffs – Determination of ash insoluble in hydrochloric acid

#### 1 SCOPE

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

#### 2 FIELD OF APPLICATION

Two procedures are established, depending on the nature of the sample.

2.1 Procedure A : applicable to simple organic animal VIHA KP feeding stuffs and to compound feeding stuffs (except those 6.2 Trichloroacetic acid solution, 200 g/l. mentioned under procedure B). stanuaru ten.ai)

2.2 Procedure B : applicable to minerals, mineral mixtures5:1978 and compound feeding stuffs/of which the ash insoluble inds/sist/c521c2b6-a747-437e-b853hydrochloric acid exceeds 1 % (*m/m*), as determined by 50-5985-1978 **7 APPARATUS** 

#### **3 REFERENCE**

ISO 5984, Animal feeding stuffs - Determination of crude ash.

#### **4 DEFINITION**

ash insoluble in hydrochloric acid : The part of the ash which is insoluble in a hydrochloric acid solution under the conditions described below; expressed as a percentage by mass of the sample.

#### **5 PRINCIPLE**

#### 5.1 Procedure A

5.1.1 Decomposition of organic matter in a test portion by incineration.

5.1.2 Treatment with hydrochloric acid of the ash obtained. Filtration, followed by drying, incineration and weighing of the residue.

#### 5.2 Procedure B

5.2.1 Treatment of a test portion with hydrochloric acid. Filtration, followed by drying and incineration.

5.2.2 Treatment of the ash as in 5.1.2.

#### **6 REAGENTS**

All reagents shall be of analytical quality. The water used shall be distilled water or water of at least equivalent purity.

6.1 Hydrochloric acid solution, 3 N.

6.3 Trichloroacetic acid solution, 10 g/l.

Usual laboratory apparatus and in particular :

7.1 Analytical balance.

7.2 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.

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

7.4 Hot-plate or gas burner.

7.5 Boiling water bath.

7.6 Incineration dishes of platinum or platinum-gold alloy (for example 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.

NOTE - For samples which are inclined to swell on carbonizing, use dishes with a surface area of about  $30 \text{ cm}^2$  and a height of about 3 cm.

7.7 Desiccator, provided with an effective desiccant.

#### 8 SAMPLING<sup>1)</sup>

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

#### 9 PROCEDURES

#### 9.1 Procedure A

#### 9.1.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the test sample<sup>1)</sup> into an incineration dish (7.6).

#### 9.1.2 Determination

**9.1.2.1** Place the incineration dish containing the test portion (9.1.1) on a hot-plate or over a gas burner (7.4) and heat progressively until the test portion has carbonized. Transfer the dish into the muffle furnace (7.2), 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 (7.3), controlled at  $103 \pm 2$  °C, replace the dish in the furnace and heat for another 1 h. Allow the dish to cool in the desiccator (7.7) to room temperature.

NOTE – The ash obtained at this point corresponds to that obtained by the procedure specified in 150 5984 indards. itch at/catalog/standards/sist/521 c2b6-a747-437e-b853-02c2f8873a83/so-5985-1978

**9.1.2.2** Transfer the ash with 75 ml of the hydrochloric acid solution (6.1) to a 250 to 400 ml beaker. Heat carefully on the hot-plate or gas burner (7.4) to boiling and boil for 15 min. Filter the hot solution through an ash-free filter paper and wash the filter paper and residue with hot water until the washings are free from acid. Transfer the filter paper with the residue to an incineration dish (7.6), previously heated for at least 30 min in the muffle furnace (7.2) at 550 °C, cooled in the desiccator (7.7) and weighed to the nearest 0,001 g. Dry the dish and its contents for 2 h in the oven (7.3), controlled at  $103 \pm 2$  °C, then ignite in the muffle furnace (7.7) to room temperature and weigh rapidly to the nearest 0,001 g.

#### 9.1.3 Duplicate determination

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

#### 9.2 Procedure B

#### 9.2.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the test sample<sup>1)</sup> into a 250 to 400 ml beaker.

#### 9.2.2 Determination

**9.2.2.1** Add successively to the beaker containing the test portion (9.2.1) 25 ml of water and 25 ml of the hydrochloric acid solution (6.1), mix and allow to stand until foaming has ceased. Add 50 ml of the hydrochloric acid solution and wait again, if necessary, until foaming has practically ceased. Heat the beaker over the boiling water bath (7.5) for 30 min or longer until all starch present is hydrolysed completely.

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

NOTE - If the solution is difficult to filter, repeat the determination using a new test portion but adding 50 ml of the trichloroacetic acid solution (6.2) instead of 50 ml of the hydrochloric acid solution and washing the filter paper and residue with hot trichloroacetic acid solution (6.3) before washing with hot water.

Aransfer the filter paper with the residue to an incineration dish (7.6), dry for 2 h in the oven (7.3), controlled at 103 ± 2°C, then ignite in the muffle furnace (7.2), set at 550°C, for 3 h. Allow the dish to cool in the desiccator (7.7) to room temperature.

#### 9.2.3 Duplicate determination

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

#### **10 EXPRESSION OF RESULTS**

#### 10.1 Method of calculation and formula

The ash insoluble in hydrochloric acid, expressed as a percentage by mass of the test sample, is equal to

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

where

 $m_0$  is the mass, in grams, of the empty dish (9.1.2.2);

1) International Standards on sampling of animal feeding stuffs and on preparation of the test sample are in preparation.

 $m_1$  is the mass, in grams, of the test portion (9.1.1 or 9.2.1);

 $m_2$  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 % (m/m).

#### 10.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed :

0,3 (absolute value) for ash insoluble in hydrochloric acid lower than 3 % (m/m);

10% of the mean value for ash insoluble in hydrochloric acid from 3 to 5% (m/m);

0,5 (absolute value) for ash insoluble in hydrochloric acid from 5 to 20 % (m/m);

2,5 % of the mean value for ash insoluble in hydrochloric acid from 20 to 40 % (m/m);

1 (absolute value) for ash insoluble in hydrochloric acid of 40 % (m/m) or more.

#### **11 TEST REPORT**

The test report shall show the method used (procedure A or B) and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details required for complete identification of the sample,

## iTeh STANDARD PREVIEW (standards.iteh.ai)

## iTeh STANDARD PREVIEW (standards iteh ai) This page intentionally left blank

## iTeh STANDARD PREVIEW (standards iteh ai) This page intentionally left blank

## iTeh STANDARD PREVIEW (This page intentionally left blank)