
**Spices and condiments — Determination of
acid-insoluble ash**

Épices — Détermination des cendres insolubles dans l'acide



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 930 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 7, *Spices and condiments*.

This second edition cancels and replaces the first edition (ISO 930:1980), which has been technically revised.

Annex A of this International Standard is for information only.

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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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Introduction

This International Standard is applicable to most spices and condiments. In view of the number and variety of such products, however, it may be necessary in particular cases to modify the method or even to choose a more suitable method.

Such modifications and other methods will be indicated in the International Standards giving specifications for the spices and condiments in question.

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Spices and condiments — Determination of acid-insoluble ash

1 Scope

This International Standard specifies a method for the determination of acid-insoluble ash from spices and condiments.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were 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 editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 928:1997, *Spices and condiments — Determination of total ash*.

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

acid-insoluble ash of spices and condiments

that part of the total ash remaining after treatment with hydrochloric acid under the conditions specified in this International Standard, expressed as a percentage by mass

4 Principle

Treatment of the total ash, obtained as described in ISO 928, with hydrochloric acid, filtration, incineration and weighing of the residue.

5 Reagents

All reagents shall be of a recognized analytical quality. Use water in accordance with grade 3 of ISO 3696.

5.1 Dilute hydrochloric acid ($\rho_{20} = 1,045$ g/ml to 1,050 g/ml), percentage by mass about 10 %.

5.2 Silver nitrate solution

Dissolve 10 g of silver nitrate in water to a total volume of 100 ml.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Electrical muffle furnace, capable of being maintained at $(550 \pm 25)^\circ\text{C}$.

6.2 Desiccator, provided with an efficient desiccant.

6.3 Filter paper, ashless.

6.4 Analytical balance, capable of weighing to the nearest 0,0001 g.

6.5 Water bath.

7 Procedure

Carry out two determinations.

7.1 Test portion

7.1.1 The test portion may be the total ash retained after determination of the total ash in accordance with ISO 928.

7.1.2 Alternatively, take a new test portion and prepare the total ash by the procedure specified in ISO 928. It is not necessary, in this case, to cool and weigh the total ash.

7.2 Determination

Add to the total ash, in the same dish in which it was prepared as specified in ISO 928, 15 ml of the hydrochloric acid (5.1). Heat the solution for about 10 min in the boiling water bath (6.5) and filter the contents of the dish through the filter paper (6.3). Wash the dish and the filter paper with hot water until the washings are free from hydrochloric acid (about 6 to 8 times). Test for the absence of hydrochloric acid with silver nitrate solution (5.2).

NOTE Lack of turbidity when a portion of silver nitrate solution is added to the filtrate indicates absence of hydrochloric acid.

Return the filter paper with the residue to the dish and ignite it in the muffle furnace (6.1) set at 550°C . Cool the dish in the desiccator (6.2) and weigh to the nearest 0,0001 g. Repeat the operations of igniting, cooling and weighing until the difference between successive weighings does not exceed 0,0005 g (m_3).

8 Expression of results

8.1 Calculate the acid-insoluble ash (w_A), expressed as a percentage by mass, using the following equation:

$$w_A = \frac{m_3 - m_1}{m_2 - m_1} \times 100 \%$$

where

m_1 is the mass, in grams, of the empty dish;

m_2 is the mass, in grams, of the dish and the test portion;

m_3 is the mass, in grams, of the dish and the residue retained from the determination specified in 7.2.

8.2 Calculate the mean of two determinations and express the result to one decimal place.

8.3 For determination on a moisture-free basis, the value should be multiplied by

$$\frac{100 \%}{100 \% - c}$$

where c is the moisture content, expressed as a percentage.

9 Precision

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

10 Test report

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

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

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Annex A (informative)

Results of interlaboratory test

An interlaboratory test, carried out in 1994 with the participation of 15 laboratories in Germany, in accordance with this method gave the statistical results shown in table A.1

Table A.1 — Interlaboratory test for the determination of acid-insoluble ash

Sample	w_A %	Repeatability limit r	Reproducibility limit R	No. of laboratories remaining after elimination of outliers
Pepper, black, ground	0,079	0,058	0,105	12
Oregano, minced	0,987	0,435	0,566	14
Cloves, ground	0,041	0,069	0,136	14