

INTERNATIONAL STANDARD

ISO
928

Second edition
1997-12-15

Spices and condiments — Determination of total ash

Épices — Détermination des cendres totales

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Reference number
ISO 928:1997(E)

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

This second edition cancels and replaces the first edition (ISO 928:1980), which has been technically revised.

Annex A of this International Standard is for information only.

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Printed in Switzerland

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 total ash

1 Scope

This International Standard specifies a method for the determination of total 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 948:1980, *Spices and condiments — Sampling*

ISO 2825:1981, *Spices and condiments — Preparation of a ground sample for analysis*

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

total ash of spices and condiments

residues obtained after incineration at $(550 \pm 25)^\circ\text{C}$ under the conditions specified in this International Standard, expressed as a percentage by mass

4 Principle

Destruction of organic matter by heating the sample to constant mass at a temperature of $(550 \pm 25)^\circ\text{C}$.

5 Reagents

Use water in accordance with grade 3 of ISO 3696.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Dish, flat-bottomed, of 50 ml to 100 ml capacity, made of platinum, quartz, porcelain or another material unaffected by the conditions of the test.

6.2 Electrical hotplate or surface heater.

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

6.4 **Desiccator**, provided with an efficient desiccant.

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

6.6 **Water bath**.

7 Sampling

Sampling shall be carried out in accordance with the method specified in ISO 948.

8 Procedure

Carry out two determinations.

8.1 Preparation of test sample

Prepare the test sample by the method specified in ISO 2825.

8.2 Preparation of the dishes

Heat the dishes for about 1 h in the muffle furnace (6.3) set at $550 ^\circ\text{C}$. After cooling to room temperature in the desiccator (6.4), weigh to the nearest 0,5 mg (m_1).

8.3 Determination

Weigh, to the nearest 0,0001 g, about 2 g of the laboratory sample into the dish (6.1) prepared as specified in 8.2 (m_2). Heat the dish on the electrical hotplate or under the surface heater (6.2) until the test portion has been carbonized. Heat in the electrical muffle furnace (6.3) set at $550 ^\circ\text{C}$.

After about 2 h, take the dish out. After cooling, wet the ash with water and dry first on the water bath (6.6) and afterwards on the electrical hotplate (6.2). Then again heat in the electrical muffle furnace (6.3) set at $550 ^\circ\text{C}$, to constant mass.

Cool in the desiccator (6.4) and weigh to the nearest 0,0001 g (m_3). Repeat the operations of heating, cooling in the desiccator and weighing until the difference between successive weighing does not exceed 0,0005 g. The total ash may be retained for the determination of acid-insoluble ash.

9 Expression of results

9.1 Calculate the total ash (w_{TA}), expressed as a percentage by mass, using the following equation:

$$w_{\text{TA}} = \frac{m_3 - m_1}{m_2 - m_1} \times 100 \%$$

where

m_1 is the mass, in grams, of the dish found in 8.2;

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 8.3.

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

9.3 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.

10 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.

11 Test report

The test report shall specify the method used and the result obtained. It shall also 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 in accordance with this method was carried out in Germany in 1994 with the participation of 15 laboratories and gave the statistical results shown in table A.1.

Table A.1 — Interlaboratory test for the determination of total ash

Sample	w_{TA} %	Repeatability limit r	Reproducibility limit R	No. of laboratories remaining after elimination of outliers
Pepper, black, ground	4,491	0,188	0,368	13
Oregano, minced	8,963	0,681	1,014	12
Cloves, ground	5,057	0,182	0,241	12