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# DRAFT ZANZIBAR NATIONAL STANDARD

Spices and condiments - Determination of acid - insoluble ash

# **ZANZIBAR BUREAU OF STANDARDS**

#### **Foreword**

This draft Zanzibar national standard has been prepared by the spices and culinary herbs technical committee. In accordance with the Zanzibar Bureau of Standards General Procedures, this draft is here by presented to the public in order to receive any technical comment concerns.

In the preparation of this standard, the reference was made to the following sources:

ISO 930:1997; Spices and condiments — Determination of acid-insoluble ash

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# Spices and condiments - Determination of acid - insoluble ash

### 1 Scope

This draft Zanzibar National Standard specifies a method for the determination of acid-insoluble ash from spices and condiment.

#### 2 Normative references

The following referenced documents are indispensable for the application of this draft Zanzibar National Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies

ZNS 48, Spices and condiments – Determination of total ash.

ISO 3696, Water for analytical laboratory use - Specification and test methods.

#### 3 Terms and definitions

For the purpose of this draft Standard, the following terms and definitions apply:

#### 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 draft Standard, expressed as a percentage by mass

## 4 Principle

Treatment of the total ash, obtained as described in ZNS 48, 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

 $(p_{20} = 1.045 \text{ g/ml to } 1.050 \text{ g/ml})$ , percentage by mass about 10%.

#### 5.2 Silver nitrate solution

dissolve 10g of silver nitrate in water to 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 ± 25)°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/hot plate.

#### 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 ZNS 48.
- **7.1.2** Alternatively, take a new test portion and prepare the total ash by the procedure specified in ZNS 48. 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 ZNS 48, 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  $550^{\circ}$ C. cool the dish in the desiccator (6.2) and weigh to the nearest 0,0001 g. repeat the operation of igniting, cooling and weighing until the difference between successive weighing 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 value 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 specify the method used and the result obtained. It shall also mention all operating conditions not specified in this draft Zanzibar 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.

# 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 acid-insoluble ash

Sample	<b>W</b> <sub>A</sub>	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