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# International Standard



# 5520

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## Fruits, vegetables and derived products — Determination of alkalinity of total ash and of water-soluble ash

*Fruits, légumes et produits dérivés — Détermination de l'alcalinité des cendres totales et des cendres solubles dans l'eau*

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## Foreword

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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 5520 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in August 1979.

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

Australia	France	Netherlands
Austria	Germany, F. R.	Peru
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Bulgaria	India	Portugal
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Chile	Israel	South Africa, Rep. of
Cyprus	Kenya	Spain
Czechoslovakia	Korea, Rep. of	Thailand
Egypt, Arab Rep. of	Malaysia	USSR
Ethiopia	Mexico	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

New Zealand

# Fruits, vegetables and derived products — Determination of alkalinity of total ash and of water-soluble ash

## 1 Scope and field of application

This International Standard specifies a method for the determination of the alkalinity of the total ash and a method for the determination of the alkalinity of the water-soluble ash from fruits, vegetables and derived products.

## 2 Definitions

**2.1 alkalinity of total ash** : The quantity of acid, expressed in milliequivalents, required to neutralize the ash obtained from 100 g of sample, under the conditions specified in this International Standard.

The alkalinity of the total ash may also be expressed as the alkalinity index (2.2).

**2.2 alkalinity index** : The number of millilitres of 1 N acid solution required to neutralize 1 g of the ash obtained from the sample, under the conditions specified in this International Standard.

**2.3 alkalinity of water-soluble ash** : The quantity of acid, expressed in milliequivalents, required to neutralize the aqueous extract of the ash obtained from 100 g of sample, under the conditions specified in this International Standard.

## 3 Principle

### 3.1 Total ash

Incineration of the product at  $525 \pm 25$  °C, addition of an excess of standard volumetric sulphuric acid solution, and back titration with standard volumetric sodium hydroxide solution, in the presence of an indicator.

### 3.2 Water-soluble ash

Incineration of the product at  $525 \pm 25$  °C, extraction of the ash with hot water, and neutralization of the aqueous extract with standard volumetric sulphuric acid solution, in the presence of an indicator.

## 4 Reagents

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

**4.1 Sulphuric acid**, 0,1 N standard volumetric solution.

**4.2 Sodium hydroxide**, 0,1 N standard volumetric solution (for total ash).

**4.3 Indicator.**

Add 4 ml of 10 g/l methylene blue solution to 100 ml of 1 g/l methyl orange solution.<sup>1)</sup>

## 5 Apparatus

Usual laboratory apparatus, and in particular :

**5.1 Dishes**, of vitreous silica or metal inert under the conditions of the test.

**5.2 Electric muffle furnace**, capable of being controlled at  $525 \pm 25$  °C.

**5.3 Desiccator**, containing an efficient desiccant.

**5.4 Burettes.**

**5.5 Analytical balance.**

## 6 Procedure

### 6.1 Alkalinity of total ash

#### 6.1.1 Preparation of test sample

Mix the laboratory sample well. Allow frozen products to thaw in a closed vessel and add the liquid formed during this process to the sample before mixing.

1) This is known as the Tashiro indicator.

### 6.1.2 Test portion

Weigh, to the nearest 1 mg, 5 to 10 g of the test sample, in a dish (5.1) previously dried and tared to the nearest 0,1 mg.

NOTE — In the case of liquid products, the test portion may equally be taken by volume, taking, by means of a pipette, 5 to 10 ml of the test sample and expressing the result in relation to 100 ml of the sample.

### 6.1.3 Incineration

After pre-drying (or evaporating) the test portion if necessary, incinerate it in the electric muffle furnace (5.2) at  $525 \pm 25$  °C until no carbonaceous residue remains.

Allow to cool in the desiccator (5.3) and weigh to the nearest 0,1 mg.

### 6.1.4 Sulphuric acid treatment

Treat the ash obtained in 6.1.3 with an accurately measured quantity (between 10 and 15 ml) of the standard volumetric sulphuric acid solution (4.1) ( $V$  ml of sulphuric acid solution).

Transfer quantitatively to a 200 ml conical flask, rinsing the dish with a little hot water.

Bring the solution to boiling on a hot-plate and boil until a clear solution is obtained.

### 6.1.5 Titration

Allow to cool, then add 2 drops of the indicator (4.3). Titrate with the standard volumetric sodium hydroxide solution (4.2) until the solution is neutral as indicated by the indicator.

### 6.1.6 Number of determinations

Carry out two determinations on the same test sample (6.1.1).

## 6.2 Alkalinity of water-soluble ash

### 6.2.1 Preparation of test sample

Mix the laboratory sample well. Allow frozen products to thaw in a closed vessel and add the liquid formed during this process to the sample before mixing.

### 6.2.2 Test portion

Weigh, to the nearest 1 mg, 5 to 10 g of the test sample, in a dish (5.1) previously dried and tared to the nearest 0,1 mg.

NOTE — In the case of liquid products, the test portion may equally be taken by volume, taking, by means of a pipette, 5 to 10 ml of the test sample and expressing the result in relation to 100 ml of the sample.

### 6.2.3 Incineration

After pre-drying (or evaporating) the test portion if necessary, incinerate it in the electric muffle furnace (5.2) at  $525 \pm 25$  °C until no carbonaceous residue remains.

Allow to cool in the desiccator (5.3) and weigh to the nearest 0,1 mg.

### 6.2.4 Hot water extraction

Add approximately 20 ml of hot water to the ash and transfer the whole to a filter paper in a funnel.

Wash the residue on the filter paper with a little hot water and add the washings to the filtrate.

### 6.2.5 Titration

Cool the filtrate, add 2 or 3 drops of the indicator (4.3) and titrate with the standard volumetric sulphuric acid solution (4.1) ( $V_1$  ml of sulphuric acid solution).

### 6.2.6 Number of determinations

Carry out two determinations on the same test sample (6.2.1).

## 7 Expression of results

### 7.1 Method of calculation and formulae

#### 7.1.1 Alkalinity of total ash

The alkalinity of the total ash, expressed in milliequivalents per 100 g of sample, is given by the formula

$$\frac{(V - V_1)}{10} \times \frac{100}{m}$$

The alkalinity index of the total ash from the sample, expressed in millilitres of 1 N acid solution per gram of ash, is given by the formula

$$\frac{(V - V_1)}{10} \times \frac{1}{m_1}$$

where

$V$  is the volume, in millilitres, of 0,1 N sulphuric acid solution added in 6.1.4;

$V_1$  is the volume, in millilitres, of 0,1 N sodium hydroxide solution added in 6.1.5;

$m$  is the mass, in grams, of the test portion (6.1.2);

$m_1$  is the mass, in grams, of ash obtained in 6.1.3.

Take as the result the arithmetic mean of the two determinations (6.1.6), provided that the requirement for repeatability (see 7.2) is satisfied.

### 7.1.2 Alkalinity of water-soluble ash

The alkalinity of the water-soluble ash, expressed in milliequivalents per 100 g of sample, is given by the formula

$$\frac{V_1}{10} \times \frac{100}{m'}$$

The alkalinity index of the water-soluble ash from the sample, expressed in millilitres of 1 N acid solution per gram of ash, is given by the formula

$$\frac{V_1}{10} \times \frac{1}{m'_1}$$

where

$V_1$  is the volume, in millilitres, of 0,1 N sulphuric acid solution added in 6.2.5;

$m'$  is the mass, in grams, of the test portion (6.2.2);

$m'_1$  is the mass, in grams, of ash obtained in 6.2.3.

Take as the result the arithmetic mean of the two determinations (6.2.6), provided that the requirement for repeatability (see 7.2) is satisfied.

### 7.2 Repeatability

The difference between the results of two determinations, carried out simultaneously or in rapid succession by the same analyst, on the same sample, shall not exceed 0,1 milliequivalents per 100 g of sample or per 1 g of ash.

### 7.3 Reproducibility

The difference between the results obtained in two different laboratories, on the same sample, shall not exceed 0,2 milliequivalents per 100 g of sample or per 1 g of ash.

## 8 Test report

The test report shall show the method used and the results 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 results.

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

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