



# SLOVENSKI STANDARD

## SIST EN 12144:1998

01-junij-1998

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**Sadni in zelenjavni sokovi - Določevanje celotne alkalitete pepela - Titrimetrijska metoda**

Fruit and vegetable juices - Determination of total alkalinity of ash - Titrimetric method

Frucht- und Gemüsesäfte - Bestimmung der Aschen-Gesamtalkalität - Titrimetrisches Verfahren

Jus de fruits et de légumes - Détermination de l'alcalinité totale des cendres - Méthode titrimétrique

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Ta slovenski standard je istoveten z: **EN 12144:1996**

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**ICS:**

67.160.20      Brezalkoholne pijače      Non-alcoholic beverages

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EUROPEAN STANDARD

EN 12144

NORME EUROPÉENNE

EUROPÄISCHE NORM

September 1996

ICS 67.160.20

Descriptors: food products, beverages, fruit and vegetable juices, chemical analysis, ash determination, alkalinity, volumetric analysis

English version

### Fruit and vegetable juices - Determination of total alkalinity of ash - Titrimetric method

Jus de fruits et de légumes - Détermination de l'alcalinité totale des cendres - Méthode titrimétrique  
Frucht- und Gemüsesäfte - Bestimmung der Aschen-Gesamtalkalität - Titrimetrisches Verfahren  
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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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## CEN

European Committee for Standardization  
Comité Européen de Normalisation  
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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## Foreword

This European Standard has been prepared by Technical Committee CEN/TC 174 "Fruit and vegetable juices - Methods of analysis", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 1997, and conflicting national standards shall be withdrawn at the latest by March 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard : Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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## 1 Scope

This European Standard specifies a method for the titrimetric determination of the total alkalinity of ash in fruit and vegetable juices and related products.

## 2 Normative references

This European Standard incorporates by dated or undated references, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN ISO 3696:1995	Water for analytical laboratory use - Specification and test methods
ISO 5725:1986	Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests
EN 1135:1994	Fruit and vegetable juices - Determination of ash

## 3 Definitions and symbols

### 3.1 definitions

For the purposes of this standard the following definition applies:

#### Total alkalinity of ash

The alkalinity, expressed in millimoles of sodium hydroxide per litre, of the ash obtained using the method specified in this standard.

### 3.2 symbols

For the purposes of this standard the following symbol applies :

- c substance concentration.

## 4 Principle

After determination of the ash content of the sample, according to EN 1135:1994, the ash is dissolved in an excess of sulfuric acid solution by heating. After cooling, the excess acid is back titrated with standard volumetric sodium hydroxide solution using a mixed indicator (methyl red/methylene blue).

## 5 Reagents

### 5.1 General

Use only reagents of recognized analytical grade and only water in accordance with at least grade 3 of ISO 3696:1987.

**5.2 Sulfuric acid**, standard volumetric solution,  $c(\text{H}_2\text{SO}_4) = 0,05 \text{ mol/l}$ .

**5.3 Sodium hydroxide**, standard volumetric solution,  $c(\text{NaOH}) = 0,1 \text{ mol/l}$ .

### 5.4 Methyl red/methylene blue indicator solution

Dissolve 100 mg of methyl red and 50 mg of methylene blue in 100 ml of mixture from 50 ml ethanol and 50 ml water.

## 6 Apparatus

Usual laboratory apparatus and, in particular, the following :

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### 6.1 Platinum dish with watch glass.

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### 6.2 Waterbath.

**6.3 Burette**, of capacity 10 ml, graduated in 0,02 ml divisions.

## 7 Procedure

### 7.1 Preparation of the test sample

Prepare ash from 25 ml of juice as described in method EN 1135:1994.

### 7.2 Test procedure

Moisten the ash (7.1) with a little water. Add an exactly measured excess of the standard volumetric sulfuric acid solution (5.2). Cover the platinum dish with the watch glass (6.1) and heat for 15 min on the waterbath (6.2), taking care to avoid evaporation to dryness. Transfer the contents of the dish by repeated careful rinsing of the platinum dish and the watch glass with water into a 150 ml conical flask. Cool the flask and contents, add a few drops of the indicator solution (5.4) and titrate with the standard volumetric sodium hydroxide solution (5.3), previously standardized against the same indicator.

NOTE : The indicator colour change is from violet/red to colourless.

## 8 Calculation

The total alkalinity of the ash,  $c$ , in millimoles of sodium hydroxide per litre is given by the formula :

$$c = 4 \times (V_1 - V_2)$$

where

$V_1$  is the volume, in millilitres, of sulfuric acid standard volumetric solution (5.2) required ;

$V_2$  is the volume, in millilitres, of sodium hydroxide solution (5.3) used.

Report the total alkalinity of the ash in millimoles of sodium hydroxide per litre, to the nearest 0,1 mmol/l.

## 9 Precision

Details of the interlaboratory test on precision of the method are summarized in Annex B. The values derived from the interlaboratory test may not be applicable to analyte concentration ranges and matrices other than given in Annex B.

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### 9.1 Repeatability

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The absolute difference between two single test results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit  $r$  in not more than 5 % of the cases.

The value is  $r = 1,3$  mmol NaOH/l

### 9.2 Reproducibility

The absolute difference between two single test results on identical test material reported by two laboratories will exceed the reproducibility limit  $R$  in not more than 5 % of the cases.

The values are :

samples other than orange juice  $R = 1,8$  mmol NaOH/l

orange juice  $R = 4,2$  mmol NaOH/l



## 10 Test report

The test report shall contain the following data :

- all information necessary for the identification of the sample (kind of sample, origin of sample, designation) ;
- a reference to this European Standard ;
- the date and type of sampling procedure (if known) ;
- the date of receipt ;
- the date of test ;
- the test results and units in which they have been expressed ;
- whether the repeatability of the method has been verified ;
- any particular points observed in the course of the test ;
- any operations not specified in the method or regarded as optional, which might have affected the results.

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